ICNF 2023

Nature Inspired
Sustainable Solutions

6th International Conference on Natural Fibers

Book of Abstracts

Edited by R. Fangueiro

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RAUL FANGUEIRO

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FOREWORD

ICNF - International Conference on Natural Fibers has established itself as a premier scientific event focused on all aspects of natural fibers, spanning from their cultivation and extraction to their applications in demanding industries. With each edition, the conference has gained significant recognition on a global scale, becoming a platform that sets and influences the major trends in the field. This is a testament to the exceptional quality of research presented at the conference and the collaborative atmosphere that fosters strong interaction among participants.

Natural fibers have gained significant importance in today's world due to several reasons. First and foremost, their sustainability aspect cannot be overstated. With growing concerns about environmental issues, natural fibers provide a sustainable alternative to synthetic fibers. Derived from renewable sources like plants and animals, natural fibers have a lower carbon footprint compared to their synthetic counterparts, which are typically derived from non-renewable fossil fuels. The biodegradability of natural fibers is another crucial factor. Unlike synthetic fibers that can persist in the environment for hundreds of years, natural fibers are biodegradable. They decompose naturally and do not cause long-term pollution, making them an environmentally friendly choice.

Natural fibers have found promising applications in various technical fields, particularly in the development of natural fiber reinforced composites. These composites, reinforced with fibers such as flax, hemp, or kenaf, offer a sustainable and lightweight alternative to traditional materials like fiberglass or carbon fiber. They exhibit impressive strength-to-weight ratios and can be utilized in the production of automotive interior panels, door trims, dashboards, and even structural components. By incorporating natural fiber composites, vehicles can achieve reduced weight, leading to enhanced fuel efficiency and decreased emissions. Furthermore, these composites provide renewable and biodegradable options for industries that prioritize eco-friendly alternatives.

Research in natural fibers encompasses various scientific trends aimed at enhancing their properties, sustainability, and application potential. Key areas of focus include fiber modification, characterization, sustainable processing and manufacturing, nanocellulose and nanocomposites, multi-scale modeling and simulation, sustainable applications, and biodegradation/recycling.

ICNF2021 is the meeting point for all those interested in these fantastic materials called Natural Fibers.

Guimarães, 19th May 2023

Raul Fangueiro
Conference Chairman

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KEYNOTE LECTURE

NEW HORIZONS FOR 1D MATERIALS: NANOSCALE TO HIERARCHICAL STRUCTURES AND INNOVATIONS

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ABSTRACT

Biological one-dimensional (1D) materials are synthesized by living organisms to give structural integrity and/or serve specific functions and have inspired designs for advanced materials. As the lateral dimensions of fibers are reduced from tens of micrometers to nanometers, the ultra-high specific surface and surface chemistries are multiplied in magnitudes to give significantly greater material potential. Nanocellulose surface chemistries and structures dictate how they behave in liquid phases, self-assemble from drying, and interface with other matter, as well as can be engineered into products. Functionalized nano cellulose carries specific surface chemistries to further expand the design and performance of novel products. This presentation highlights targeted and streamlined approaches in reacting cellulose to be efficiently and directly disintegrated into functionalized nano cellulose to create new sustainable materials from the readily available and underutilized biomass. Advancements in biologically derived 1D nanomaterials offer renewable solutions and versatile opportunities to outcompete fossil fuel-based counterparts and to meet future advanced material needs.

KEYNOTE LECTURE
NEW TECHNOLOGIES TO GENERATE BIOHYBRID MATERIALS WITH ADVANCED FUNCTIONAL PROPERTIES

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ABSTRACT
Biohybrid materials have recently grown in attention due to the enormous range of possibilities presented by a material that has the functionalities of living cells or simply enzymes supported on a scaffold. Among the non-living components, bacterial biopolymers have stood out as their properties can be engineered by microbial biotechnology tools through sustainable bioprocesses. Bacterial cellulose (BC) has aroused particular interest due to the highly porous fiber network formed during the static culture of bacteria, with a tremendous water-holding capacity. The high crystallinity of BC, together with its high purity, renewability, biodegradability, and biocompatibility, make this material unique for certain applications with high-quality requirements. The natural properties of these macromolecules can be tuned by strategies of materials science combined with microbial biotechnology based on synthetic biology and metabolic engineering that make use of microbial cells as factories, enabling the production of next-generation advanced materials with smart functional properties.

KEYNOTE LECTURE
NANOMECHANICS OF NATURAL, BIOINSPIRED, AND BIONIC FIBERS AND RELATED COMPOSITES

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ABSTRACT
Spider silk is one of the best natural fibres in terms of mechanical properties. Although it is spun in a fraction of a second under environmental conditions, it displays a remarkable combination of strength and deformability that are used by spiders to build robust webs and overcome muscles' limitations.

Spider silk also possesses high lustre and biocompatibility that contribute to making it attractive for many technical and medical applications. However, despite being strong and flexible, spider silk is poor in terms of electromagnetic properties, which limits its applicability in soft electronics. To solve this, we have designed a composite made of spider silk and a metallic alloy to be used as a magnetic strain sensor or actuator. Moreover, we exposed spiders to carbon-based nanomaterials solutions to make them directly spin electromagnetic silk, pinpointing as the main challenge the inherent high day-to-day variability of this protein-based fibre.

Unfortunately, the cannibalistic nature of spiders makes it impossible to produce spider silk at the industrial level by harvesting it from animals, meaning that to produce silk composites we need artificial spider silk. A recent technological leap allowed us to produce spider silk proteins in large quantities. These are now currently used to produce composites with magnetic nanoparticles, in order to provide spider silk with extra functionalities, such as magnetic actuation. This in-spinning technique is crucial to avoid common setbacks of post-spinning procedures, such as excessive water plasticization. In summary, this talk will provide the state of the art regarding the creation of composites made with spider silk (artificial and natural), setting and elucidating the challenges and the future possibilities.

INVITED LECTURE
LACTIDE IN IN SITU POLYMERISATION (ISP) DURING MONOMER INFUSION UNDER FLEXIBLE TOOLING (MIFT).

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INTRODUCTION

The ERDF/InterReg 2 Seas Mers Zeeën SeaBioComp project sought to develop durable bio-based composites for use in the marine environment. The long-term ecological impact of plastic litter and microplastics in the marine environment is a growing issue that has gained considerable momentum in public perception and global media. Bio-based polymers, or polymers from renewable resources, could be a viable substitute to conventional oil-based polymers for many applications. The change might significantly reduce greenhouse gas emissions and has potential to ease end-of-life issues if the materials are biodegradable.
One of the polymers of interest is poly(lactic acid), or poly(lactide) produced from the dimer. The SeaBioComp project primarily used compression moulding or fused filament additive manufacture of the polymer to produce demonstrator components.

LARGE MARINE COMPOSITE STRUCTURES

For large composite structures, the process of choice would be Resin Infusion under Flexible Tooling (RIFT), also known as SCRIMP, VARTM or a multitude of other abbreviations [1]. However, molten thermoplastic polymers typically have viscosities far higher than those used for the Liquid Composite Moulding (LCM) processes. Further, the melt temperatures of many thermoplastic systems are higher than the degradation temperature of the lignocellulosic fibres used in biocomposites.

INFUSED THERMOPLASTIC MATRIX COMPOSITES

Van Rijswijk and Bersee [2] reviewed in situ polymerisation for thermoplastics and classified the principal systems of potential use for Monomer Infusion under Flexible Tooling (MIFT). Qing et al [3] further down-selected monomers suitable for biobased composites to be used in the marine environment with natural fibre reinforcement. The parameters considered were (i) monomer viscosity, (ii) processing temperature, (iii) moisture absorption, (iv) mechanical properties, (v) bio-based availability, (vi) process open window, (vii) cost, and (viii) recyclability. Commercially available acrylic resin was the best fit to the above criteria, but is not yet available as a bio-based infusion system.

LACTIDE MONOMER

The in situ polymerisation of lactic acid was deemed inappropriate as the condensation polymerisation would release water that would manifest as voids in the composite. The dimer of lactic acid (lactide) polymerises by ring-opening without releasing water. Lactide is supplied as a white crystalline solid with a melting range of 90-100°C. The product data sheet for lactide says “preferably store below 35°C. On-returning from Covid-19 lockdown, the open package of lactide had gone into solution (deliquescence) in the moist air in the laboratory. A recently delivered package of lactide was labelled “packed under vacuum .. content is moisture sensitive .. use immediately after opening or keep under a nitrogen atmosphere". Our technical team advised that "storing 20 kg under an inert gas is going to be a challenge"! [4]. Louisy et al [5] have reported in situ bulk polymerisation of l-lactide after resin transfer moulding (RTM) preparation of glass fabric composites, but results were limited to degree of polymerisation data and optical microscopy to assess composite quality. The SeaBioComp project established that flow and polymerisation of the lactide is best achieved in the temperature range 120-180°C.

REALISATION OF PREDICTED MECHANICAL PROPERTIES

In comparative tests between test samples, for flexural modulus flax/acrylic samples achieved 53%, while flax/PLA samples achieved 37% of properties predicted by rules-of-mixtures. For flexural strength, using KellyTyson equation and only considering fibres aligned with the stress, flax/ acrylic samples achieved 104%, while flax/PLA samples achieved 62% of the predicted properties.

DEMONSTRATOR COMPONENT

The project sought to deliver a 5G telecommunication dome as a demonstrator component. The intention was to use integrally heated infused composite tooling, but despite placing the order with a well-respected supplier, the mould tool proved to be a challenge due to a combination of complex geometry, with consequent flow paths leaving dry spots and delamination during heating cycles.

CONCLUSION

MIFT for lactide remains at around Technology Readiness Level (TRL) 1. While it may be suitable for just-in-time manufacture, storage of material under dry nitrogen presents challenges. The process temperatures are challenging for integrally-heated composite tooling, so oven-cure or metal mould tools may be appropriate. The composites do not achieve predicted mechanical properties, but the experiments conducted here did not use a coupling agent on the natural fibres.

ACKNOWLEDGMENTS

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INVITED LECTURE
NEXT GENERATION OF SUSTAINABLE NATURAL FIBERS: EXPLORING INNOVATIVE AND MULTIFUNCTIONAL FIBERS AT MACRO, MICRO, AND NANOSCALE

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ABSTRACT
Natural fibers, including cellulosic, protein, and mineral fibers are collected directly from nature and are considered eco-friendly materials. Compared with synthetic fibers, they present numerous advantages, including high abundance, low cost, biodegradability, biocompatibility, low weight, and relatively good mechanical properties.

Nowadays, due to great advances in spinning techniques and experimental procedures, it is also possible to develop innovative natural fibers using natural compounds and residues from natural sources. The latest trend in nanotechnology to produce new fibers is electrospinning. Electrospun Nanofibers (Nfbs) have been extensively investigated for biomedical applications because they exhibit a similar microstructure to that of the extracellular matrix, and they can mimic the biological microenvironment as well. Polymeric Nfbs also present high surface area, and high porosity with interconnectivity which promotes cell adhesion, proliferation, drug delivery, and mass transport properties. Wet spinning is also an excellent technique to achieve fibers at the micro-scale with different functionalities depending on the polymeric formulation under use. One of the main advantages of using techniques such as electrospinning or wet-spinning to produce new fibers is the versatility of the polymeric formulations that can be used. Thereby, both natural and biodegradable commercial polymers can be used as well as those extracted from various sources, including algae and textile industry residues. Alginates and an infinite diversity of bioactive components can be extracted from algae. From textile industry-based residues, mainly cellulose nanocrystals can be extracted in order to be incorporated into the polymeric formulations. Several other examples could be highlighted; however, the main purpose is always to target a combination of (bio)active compounds with natural/biodegradable polymers, as an emerging strategy to produce a range of multifunctional innovative fibrous materials.

INVITED LECTURE
STRATEGIES TO TAILOR THE INTERFACE IN NATURAL FIBRE COMPOSITES

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ABSTRACT
Over the past decades, the widespread use of glass fibres within polymeric matrix composite materials has significantly increased, leading to a strong interest in greater eco-sustainability due to the limited possibilities of disposal of these materials at the end of their life. For this reason, the production of bio-composite materials, using sustainable matrices or natural reinforcing fibres, has recently been a cornerstone of many research fields. Fibres sourced from vegetal and mineral resources feature a poor interfacial adhesion with many polymer matrices, a drawback that still hinders a much wider diffusion in many industrial fields. The aim of this presentation is to share some recent surface modification strategies developed in our group to tailor the interfacial adhesion in natural fibre composites, including plasma-enhanced chemical vapour deposition and the development of hierarchical fibres, in which nanoscale carbon or ZnO nanostructure reinforcements are utilized alongside traditional microscale reinforcing fibres. In particular, these strategies will be presented for the surface modification of flax and basalt fibres.

INVITED LECTURE
A STORY OF PLANT FIBERS FOR COMPOSITE REINFORCEMENT: WHEN CELL WALLS BITE THE DUST

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ABSTRACT
This work proposes a scientific story about different work performed on plant fibres singularities characterization, especially through cutting-edge techniques through a range of Synchrotron radiation beamlines; and also some bibliographic elements about the impact of processing on the intrinsic properties of these sensitive but also strong fibres.

INTRODUCTION
Plant fibres are complex and hierarchical cells with a large range of biochemical compositions and morphologies, depending on their in-planta functions (Bourmaud et al., 2018). This talk will be divided into two main sections: first, we will detail the main plant fibre structural parameters, i.e., biochemical composition, microfibrillar angle and architecture; their impact on the behaviour and mechanical performance of the fibres will be described, thanks in particular to characterisations carried out recently on Synchrotron beam lines(Bourmaud et al., 2022; Richely et al., 2022). In a second step, a link will be made with the manufacturing stage by exploring the impact of the processing parameters, and in particular shear, time and temperature on the ultrastructure of the fibres. Their very specific architecture and structure can be significantly altered by the mechanical stresses involved in conventional composites tools. These structural modifications have a significant impact on the dimensions of the fibres, and therefore on their aspect ratio, degree of crystallinity, but also on inter-polymer bonds and on the overall parietal structure of plant fibres which strongly conditions their reinforcement ability. The importance of the time-temperature relationship but also the choice of fibres will be particularly highlighted.

RESULTS AND CONCLUSIONS
Figure 1 shows two examples of characterisation studies on flax fibres carried out at the Synchrotron Soleil on the ANATOMIX and SWING beamlines; it is possible, by Micro-Tomography, to obtain very original information on the internal structure and the porosities present in the kink-band regions (Fig.1.A). By coupling tests using the X-ray diffractometer of the SWING line and a tensile machine, it is also possible to measure realignments of the cellulose macro fibrils and changes in the associated microfibrillar angle in these same defect areas.
The use of these technologies not only allows access to very short acquisition times but also to a precision and resolution not available on conventional equipment. In the present case, the combination of these two parameters has permitted the achievement of tests on elementary fibres with an unequalled precision in the morphological and ultrastructural characterisation.

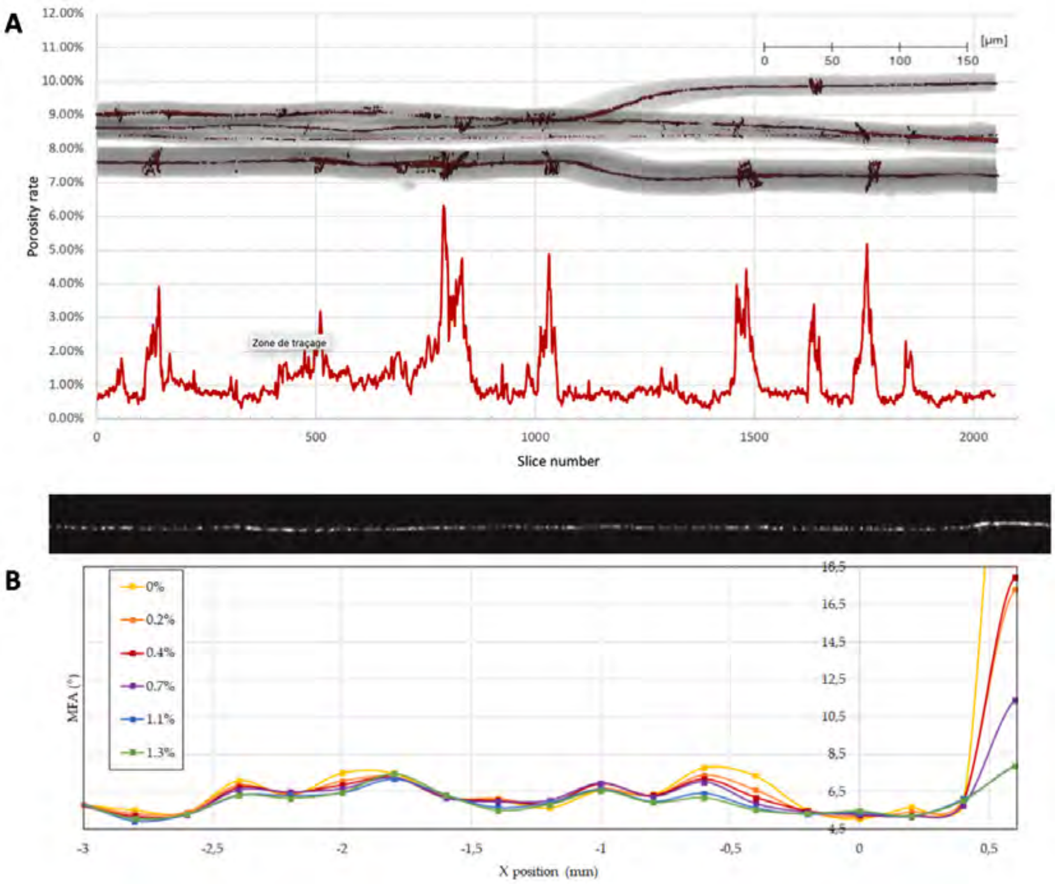

Fig.1 Analysis of flax kink-band regions singularities through Micro-Tomography **(A)** on X-RD-tensile test coupling **(B)**

ACKNOWLEDGMENTS

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ID 1
NATURAL FLAME RETARDANTS FOR NATURAL FIBERS

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ABSTRACT
Textiles are used in a wide variety of applications, including carpets, clothing, upholstery or furnishing fabrics. They are made of synthetic and natural polymers, have a large surface area and an organic origin. Therefore, most of them are flammable and thereby potentially hazardous unless suitable protective coatings are applied (Salmeia, 2016). In order to avoid ignition and spread of the fire, many textile products are finished with flame retardants (FR) (Horrocks, 2011). Due to their high efficiency, brominated FRs have been used for textile coatings for a long time. However, their toxicity and environmental impact together with a potential risk of exposure to humans and the environment, led to legal restriction and prohibition. For that reason, an intense research activity focuses on halogen-free alternatives, and various phosphorus and nitrogen-containing compounds (Laoutid, 2009). Furthermore, the development of bio-based flame-retardant materials made of renewable instead of fossil resources became one of the latest objectives in this field (Sonnier, 2018).
Here, we present our results on the immobilization of phytic acid (PA), as a natural source of phosphorus, on cotton (CO) in a layer-by-layer (LbL) approach with polyvinylamine (PVAm) as the oppositely charged electrolyte to create a partly bio-based flame-retardant finish. PVAm was employed as a synthetic nitrogen source with the highest density of amine groups of all polymers. Increasing the number of bilayers resulted in an increasing phosphorus and nitrogen content. The finishing led to a flame-retardant behavior (no afterflame and afterglow time, no ignition). Cotton finished with 15 bilayers passed the bottom edge ignition flame test according to ISO 15025:2016 (Figure 1).

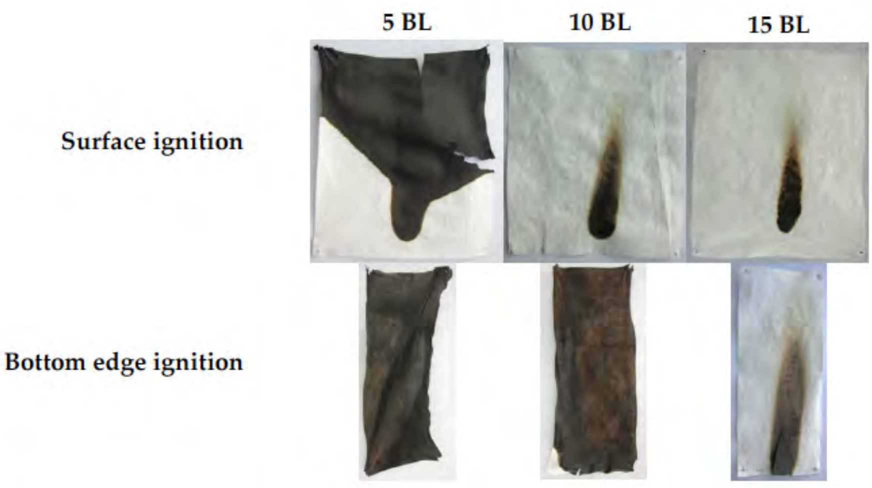

Figure 1: Coated fabrics (5, 10, 15 BL) after the flame tests, according to ISO 15025:2016 Method A and B, of surface and bottom edge ignition. Uncoated cotton fabrics burned completely down.

Microscale combustion calorimetry (MCC) and thermogravimetric analysis (TGA) measurements showed a shift of the decomposition temperatures in combination with high char yields. From Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy analyses, cellulosic dehydration by phytic acid as an acid catalyst and the formation of a protective char layer, as a physical barrier on the surface of the fabric, were proven.

In addition, we present our latest findings on the simplification of the overall process by applying the phytic acid/polyvinylamine mixture in one single step and the substitution of the synthetic polyvinylamine by natural-based chitosan leading to a fully bio-based flame retardant. Moreover, we introduce our research on further potential natural based flame retardants for textiles derived from leather (collagen) and goose quills (keratin).

ACKNOWLEDGMENTS

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ID 4
LIFE CYCLE ASSESSMENT OF A FABRIC BASED ON CONVENTIONAL COTTON VERSUS RECYCLED COTTON

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ABSTRACT
Through the Life Cycle Assessment (LCA), the study compares the environment impacts of two fabrics with different compositions: a 100% conventional cotton fabric versus a fabric with 60% pre-consumer recycled cotton and 40% conventional cotton fibers. The main purpose is to prove how much environmental impacts can be reduced using pre-consumer recycled cotton.

INTRODUCTION
The textile sector is one of the sectors that most harms the environment and has one of the highest levels of industrial waste. Due to the variety and the number of processes, it is causing several negative impacts on the global environment. The leftovers produced by the textile industry are particularly worrying, and become a serious threat to the environment and, consequently, to future generations when incorrectly managed (Refosco, 2012).
Given the growing concern about the environment and considering the textile industry has a prominent place in the pollution of the planet, companies are increasingly focused on finding strategies to minimize waste generation (Ramos, 2009).
To quantify the savings for the environment by reusing pre-consumer waste, it´s calculated through the Life Cycle Assessment (LCA).
For the development of this study, cotton was the selected fiber, cause is one of the most used natural fibers in the textile and garment industries, that is nearly 30% of all textile fiber consumption worldwide (Remy, Speelman, & Swartz, 2016).
According to the standards NP EN ISO 14040:2008 – Environmental management – Life cycle assessment, and NP EN ISO 14044:2008 – Environmental managent – Lyfe cycle assessment, the LCA is a method through which it is possible to “assess the environmental aspects and impacts associated with a product process, throughout its life cycle” (Appel, 2019). The LCA is composed of four phases: 1. definition of the goal and scope, 2. Life Cycle Inventory (LCI), 3. Life Cycle Impact Assessment (LCIA) and 4. Interpretation.

RESULTS AND CONCLUSIONS
The results of LCA are shown in the tables above: Table 1 - Specific consumptions and emissions for the analysed fabrics and Table 2 - Categories of environmental impact for the analysed fabrics. In each table, the values of the last column are the comparation between the production processes of conventional and recycled cotton fabrics.

The results are shown considering the production of 100 kg of finished fabric.

Indicators	Conventional	Recycled	Difference
Primary energy consumption (kWh)	4 791,07	2 947,56	-38,48%
Non-renewable primary energy consumption (kWh)	2 806,97	1 550,15	-44,78%
Renewable primary energy consumption (kWh)	1 984,10	1 397,41	-29,57%
Freshwater usage (kg)	2 593 193,30	1 567 993,86	-39,53%
Freshwater consumption (kg)	531 491,60	211 152,52	-60,27%
Waste (kg)	240,63	286,08	18,89%
Emissions to freshwater (kg)	2 060 037,56	1 354 719,88	-34,24%
Emission to air (kg)	14 589,54	11 768,38	-19,34%

Table 1 - Specific consumptions and emissions for the analysed fabrics

Categories	Conventional	Recycled	Difference
Climate change, default, excl biogenic carbon [kg CO2 eq]	870,20	484,72	-44,30%
Climate change, incl biogenic carbon [kg CO2 eq]	511,57	340,95	-33,35%
Fossil depletion [kg oil eq.]	236,40	130,18	-44,93%
Freshwater ecotoxicity [kg 1,4 DB eq.]	51,14	19,36	-62,14%
Freshwater Eutrophication [kg P eq.]	0,49	0,19	-61,18%
Human toxicity, non-cancer [kg 1,4-DB eq.]	988,32	372,23	-62,34%
Terrestrial Acidification [kg SO2 eq.]	7,32	3,02	-58,79%
Terrestrial ecotoxicity [kg 1,4-DB eq.]	1 144,07	434,63	-62,01%

Table 2 - Categories of environmental impact for the analysed fabrics

According to the study, the recycled fabric has less environmental impacts then the conventional fabric. The results for the indicators relate with resource consumption and emissions generated has shown lower values for recycled fabric, with the lonely exception of waste quantity but with a not significant difference. Concerning to the impact categories, the results are also lower for the recycled fabric.

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ID 5
CHARACTERIZATION OF CIRCULAR COMPOSITES FABRICATED USING RECYCLED TEXTILE WASTE AND POLYPROPYLENE

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ABSTRACT
This research proposes the analysis of mechanical performance of circular composites made from different reinforcements i.e., woven, and non-woven textile waste materials and matrix i.e., polypropylene. The composites contain two types of reinforcements of same areal density with three levels of fiber volume fraction (V_f) levels. The mechanical characterization was performed to observe the difference in the ultimate performance of composite materials w.r.t reinforcement type and V_f.

INTRODUCTION
Denim fabric covers a major portion of the textile industry which have production/ trade of 100 billion USD and is expected 107 billion USD in 2023. And the global jeans market is 84.1 billion USD which is expected to increase to 102.5 billion USD in 2025 (Uncu Akı et al., 2020). Global and environmental sustainability is a hot topic nowadays. There are some problems for a sustainable environment including the greenhouse effect, climate change, acidification, abiotic depletion, toxic contamination, depletion of some species, deforestation, depletion of fossil fuels, land degradation, the release of polluting chemicals, and other hazardous materials to the environment (Esteve-Turrillas & de la Guardia, 2017; Periyasamy et al., 2017). According to the sustainable development goals, recycling, reusing, repurposing, reducing by design, and use of renewable resources according to the international environmental protocols have gained much attention in the current scenario (Geissdoerfer et al., 2018; Joustra et al., 2021; Mohd Nasir et al., 2021; Rebitzer et al., 2004).
In this context, environment-friendly solutions such as circular composites have been developed. These composites can be developed from virgin as well as recycled materials (Mohd Nasir et al., 2021). The circularity of materials e.g., composite materials is a significant challenge in recent years. The observance of the composite circularity improves material diversity and optimizes energy use, resources as well as recycling e-ciencies. The waste related to composites, such as reinforcement waste, resin waste, and post-usage composite waste, like all other materials in the waste stream, needs to be recycled and looped into production to fit into the Circular Economy model (Khalid et al., 2022; Luiken & Bouwhuis, 2015; Such et al., n.d.). The goal of this research is the development of sustainable composites, based on post-industrial, post-consumer textile waste and their comparative analysis.
Results and conclusions
The goal of this study is to create innovative approaches for recycling the global stream of textile waste materials after they have been used up. Converting significant amounts of textile waste into numerous useful applications and composite products is an important step towards circularity. In this research, the

composites made from two types of reinforcements having the same areal density with three V_f levels were analyzed. Initially, the textile waste was shredded and made into non-woven plies. Mechanical properties of circular composites including tensile strength (DIC), bending strength, drop weight impact strength, and SBS were characterized. Results showed that the woven textile waste reinforced composite has better mechanical properties as compared to non-woven textile waste reinforced. The finished product made from these second-generation textile waste composites can be utilized in variety of applications.

ACKNOWLEDGMENTS

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ID 7
CELLULOSE NANOFIBER FROM WHEAT STRAW TO PHOTOCATALYTIC DE-NOX MATERIALS APPLICATION

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ABSTRACT

This work deals with the preparation of aerogels based on nanocellulose obtained from wheat straw, together with TiO2 and their use in the elimination of nitrogen oxide (NOx) species emissions (De-NOx action). For this purpose, an innovative strategy of agricultural waste valorization is employed. The formulation of hybrid structures was successfully carried out following a simple colloidal route based on a heterocoagulation process between delignified cellulose nanofibers (CNF) and TiO2 nanoparticles (TiO2 NPs). Under light irradiation, all samples showed a positive response to NO reduction.

INTRODUCTION

Air pollution caused by rapid industrialization and urbanization currently poses one of the most serious challenges facing modern societies. One of the causes of this pollution is the increased emission of nitrogen oxide species (NOx). The release of these harmful gaseous pollutants into the atmosphere has been identified as the 4th ranked threat to human health (Priya et al.,2021). The manufacturing of cross-linked three-dimensional (3D) structures for example, e.g., hydrogels, sponges, foam, crumpled shapes, especially aerogels, are attracting the attention of researchers as a new category of photocatalyst materials due to their immobilization ability, high specific surface area, structure integrity, mechanical robustness, high porosity and easy separation of the reaction solution from the mixture (Liu et al., 2019, Xu et al., 2016).

Cellulose and nanocellulose fibers were isolated from wheat straw after an environmentally friendly process. The process and operating parameters were optimized for nanocellulose isolation in previous work by the authors (Espinosa et al., 2020). A colloidal suspension of photocatalytic nanoparticles was prepared following the procedure described in the literature (González et al., 2020). The resulting suspensions in each case were casted in plastic molds of 25- and 60-mm diameter, frozen for 24 h and freeze-dried. The nanocellulose-TiO2 interaction and its effect on the aerogels structures were studied by analyzing physicochemical and morphological properties. To evaluate the De-NOx photocatalytic activity of the aerogels samples were light irradiated using a Solarbox 3000e RH solar simulator. The concentration of the simulated atmosphere is similar to levels found in highly populated cities.

RESULTS AND CONCLUSIONS

It has been demonstrated that the design of heterostructures by means of colloidal route provides numerous possibilities that are unattainable using traditional methods, confirming the fundamental hypothesis of the present work. It was shown that CNF presented more suitable characteristics for the preparation of hybrid nanocellulose-TiO2 aerogels by colloidal dispersion. The effect of TiO2 concentration on the physical and mechanical properties and on the photocatalytic activity of the produced hybrid aerogels was also studied. It is observed that with increasing TiO2 concentration in the formulation, the porous radius shifts towards lower sizes, reaching for a concentration of 200% a maximum distribution for 20-30 nm, close to TiO2 P25 nanoparticles. This indicates that the inorganic phase is completely covering the surface of the CNF. An improvement in the mechanical strength of the aerogels was observed is due to the large surface area and structure of TiO2, which when incorporated into the cellulosic matrix produces greater crosslinking and a better interface between the matrix and the nanoparticles, allowing to increase the range of application of this type of photocatalytic lightweight materials to situations requiring higher mechanical strength. All samples showed a positive response to NO reduction under light irradiation. The best selectivity (S=82%) was observed in the CNF100 sample which, together with the highest efficiency of 78% for NO removal, allowed achieving an outstanding NOx efficiency of 64%, values much higher than those exhibited by the commercial TiO2 P25 powdered photocatalyst. It is therefore concluded that the use of CNF as a support for photocatalytic materials not only favors the adequate dispersion and support of the nanoparticles, but also its aerogel conformation and specific cross-linked structure facilitates good contact and transfer of contaminated gas, increasing the efficiency of the photocatalytic activity.

ACKNOWLEDGMENTS

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ID 11
SCREEN-PRINTING OF MICRO/NANO-FIBRILLATED CELLULOSE FOR AN IMPROVED MOISTURE MANAGEMENT AND ABRASION RESISTANT PROPERTIES OF FLAME-RESISTANT FABRIC

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ABSTRACT
Low moisture absorbency of hydrophobically coated flame-resistant (FR) fabrics do not correlate well with the thermophysiological comfort (i.e. moisture transfer to cool the body and reduce the heat by moisture build-up), which is difficult to achieve. In this frame, we were the first to study the effect of screen-printing of micro and nano-fibrilated cellulose (native, carboxylated, phosphorylated and quarternized) on the fabric surface to meet these requirements as wear-related comfortable coating, following also the challenges of sustainability and circularity.

INTRODUCTION
The printability of fibrillated cellulose dispersions, using a screen-printing technology, is challenging compared to traditional printing pastes due to their relatively lower viscosity (20-80 Pas) and yield stress at rather low solids content, typically below 3 wt% (Kokol, 2022). The pattering of micro/nanocellulose as a function of the printing parameters (type of mesh, squeegee pressure and printing speed) and its durability (attachment) after the subsequent printing of hydrophobic polyacrylate on the same (layer-by-layer)) or the other side of the fabric will be presented, with the aim of preserving one side of the fabric (facing towards the wearer) hydrophilic, while keeping the other side (facing outwards) hydrophobic (Kokol, 2021, Kolar 2021 & 2022). The changing of fabrics properties, abrasion resistance, air permeability and water-vapour resistance, as well as surface wetting and absorbency, will be discussed.

MATERIALS AND METHODS
The fibrillated native and surface modified micro/nanocellulose dispersions were purchased from different suppliers as presented on Fig. 1. The acrylate water-based synthetic printing paste / AP was prepared by Tekstina Ltd (Slovenia), according to their self-developed recipe using commercial products of CHT Bezema, Germany, adjusted to the viscosity of 105±5 dPa s. Flame-retardant (FR) fabric of 145±1.4 g/m^2 mass, 0.332±0.02 mm thickness, 36 threads/cm in the warp direction and 51 threads/cm in the weft direction was provided by Tekstina Ltd, Slovenia. The fabric was constructed as two-wefts woven fabrics, made from spun yarns (a mixture of meta aramid and FR Lenzing viscose in the same 34/34% ratio) in both weft (which predominates on the back side of the fabric; i.e. the face that looks outward while wearing) and warp directions, and viscose filaments (32%), which also appear in the weft direction, and predominate on the front side (i.e. the side that looks towards the wearer).

The printing was performed on a Zimmer laboratory printing machine using a nickel-based rotary screen of different perforations (60 and 135 mesh size, 14 and 22% open area, 161 and 88 mm of holes` diameter, respectively) and steel-rod type squeegee of 15 mm in diameter, at different pressures (no. 2 and 6) and a relevant speed (stage 5 ≈ 6 m/min). The prints were performed as two-layer printings applied in two separate steps: i) on the same (back/outer) side of the fabric, where the MFC/NFC dispersion was printed first, followed by printing of AP, or ii) on a different side of the fabric where the MFC/NFC was applied on the face (inside turned) side, followed by AP on the back side, using the same printing sequence. After each printing step, the fabrics were dried at 100°C for 3 min and finally cured at 170°C for 2 min. The mechanical properties and wearing comfort (surface hydrophilicity, air permeability, water vapour resistance, abrasion resistance) of the fabrics were analysed.

RESULTS AND CONCLUSIONS

Fibrillated cellulose	Abbr.	Fibrils size	Surface charge (mV) pH 7	Rotational viscosity 1.5wt% (dPa s)
MFC (Exilva F-01, Borregaard, Norway)	**MFC**	l=22–50 µm d=10-100 nm	**-28.2±2.2**	40±3
NFC (Uni Maine, USA)	**NFC**	l=few µm d=10-50 nm	**-32.6±1.0**	25±2
TEMPO-oxidized NFC (Uni Maine, USA) $-COO^-$, ≈1.5 mmol/g	**Tempo-NFC**	l=few µm d=10-50 nm	**-35.1±3.4**	25±5
Phosphorylated NFC (Xylocel Oy, Finland) $-PO_3^{2-}$, DS≈0.13	**Phosph-NFC**	l=few µm d=10–200 nm	**-39.4±3.6**	45±2
Quaternized-NFC (Xylocel Oy, Finland) $-N^+(CH_3)_3Cl^-$, DS≈0.23	**Quarter-NFC**	l=few µm d=10–200 nm	**+37.6±1.9**	40±2

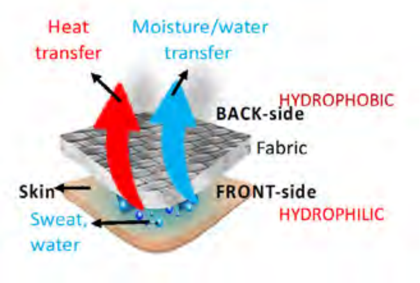

Fig.1

The MFC/NFC pre-printing and its durability (attachment) after a post-printing of hydrophobic polyacrylate on the same/back (LbL) or other/front side of the fabrics was considered to maintain the one/back side of the fabric (facing towards the wearer) hydrophilic, while keeping the other side (facing outward) hydrophobic as presented on Fig.1. Such an anisotropic surface wetting increases moisture absorption rate (from 50 up to 100 %/s), reduces surface wetting time (from 30 up to 7 s), increases water vapour transfer (≤ 5.5 Pa m2/W) and one-way transport capability (up to 540%), when printed with MFC or negatively charged-NFC, thus given significant improvement in fabric moisture management during wearing and exposure to hot environments. TEMPO-ox-NFC had also the lowest effect on air permeability & improved abrasion resistance.

ACKNOWLEDGMENTS

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ID 12
ANTIMICROBIAL NANOCELLULOSE-BASED MICROPOROUS PARTICLES, PROPERTIES AND APPLICATION POTENTIALS

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INTRODUCTION

Research focusing on naturally-occurring biocides with a low-risk of toxicity for humans and the environment is becoming increasingly urgent. The use of nanocellulose (NC) in this context is also increasing due to its low cost and unique properties, which are potentially useful in many applications, such as wound dressings, composites, packaging materials, filters/adsorbents, textiles and paints. However, NC needs to be surface modified to gain an antimicrobial potential. Functionalizations with amino or quarternary ammonium-bearing hydrophobic molecules are of particular interest, as they can, additionally, promote interactions with a negatively charged and hydrophobic bacterial cell membrane, compromising its integrity and leading to leakage of cytoplasmic content and, ultimately, cell lysis (Tavakolian et al. 2020), thereby resulting in a bactericidal effect.
The aim of this study was, thus, to determine the relative influence that NC aspect ratios (fibrillated CNF vs. rod-shaped CNC) and their surface chemical modifications (periodate oxidation to aldehydes and further conjugation with HMDA by the Schiff-base reaction), which alter their morphology and physicochemical properties, have on their antibacterial activity, aquatic (*in vivo*) and human cells (*in vitro*) toxicity, and eco-toxicology (biodegradability in composting soil). By performing an *in vivo* study on rabbits, it was already proven that HMDA-functionalized CNF can be classified as skin non-irritating (Kokol et al. 2021).

MATERIALS AND METHODS

The NC was purchased from the University of Maine, USA, and fully analysed before and after each functionalization step. The content of aldehyde groups was determined by the modified spectrophotometric method using a TTC/KOH solution. Potentiometric titration was performed for quantifying the surface charge contribution using a dual-burette instrument (Mettler Toledo T-70), equipped with a combined glass electrode (Mettler TDG 117). The hydrodynamic size and the zeta-potential of the samples were assessed by Dynamic Light Scattering (Zetasizer, Nano ZS ZEN360, Malvern Instruments Ltd., UK). The high resolution images of the sample were performed using a Sirion 400NC (FEI, USA) Scanning Electron Microscope. The standard dilution antibiogram, using the Gram-negative (G-) bacterium *E. coli* (EXB-V127) and Gram-positive (G+) bacterium *S. aureus* (EXB-V54), obtained from the Microbial Culture Collection Ex, was performed to evaluate the antibacterial properties. The acute toxicity tests were carried out *in vivo* according to modified standard ISO 16197:2014 with *Daphnia magna* ephippia purchased from Microbiotests Inc., Belgium. The *in vitro* cytotoxicity of samples with A549 human lung adenocarcinoma cells was evaluated by three different cytotoxicity assays (Resazurin, Coomassie Blue, and Neutral Red Uptake). The biodegradation of approx. 0.1 g of each sample, prepared by solvent-casting as films or by freeze-drying as sponges, was assessed in a composting soil, according to ISO 11721-1: 2001.

RESULTS AND CONCLUSIONS

Such a two-step surface modification resulted to a porous and micro-sized (0.5-10 μm) particles (CNF/CNC-ox-HMDA) that can catch bacteria by multi-targeted (ionic and hydrophobic) non-specific modes of action, and prevent them from growing at minimum nontoxic (both *in vitro* to human lung cells and *in vivo* to *Daphnia magna*) concentrations (< 0.2 wt%). The hydrodynamically smaller (0.5-1 μm) CNC-ox-HMDA decorated with aldehyde, anionic, amino and hydrophobic surface groups was found to be the most effective, with up to log 9 of bacteria reduction at >0.4 wt.% and a bactericidal effect. While the antimicrobial less-active CNF/CNC-ox were fully biodegradable in composting soil within 24 weeks, this process was inhibited considerably for CNF/CNC-ox-HMDA, indicating their different stability and disposal after use (e.g. composting vs. recycling). As such, however, they can serve as an alternative to nano-biocides (such as metal nanoparticles), which are limited by their toxicity for humans and the environment.

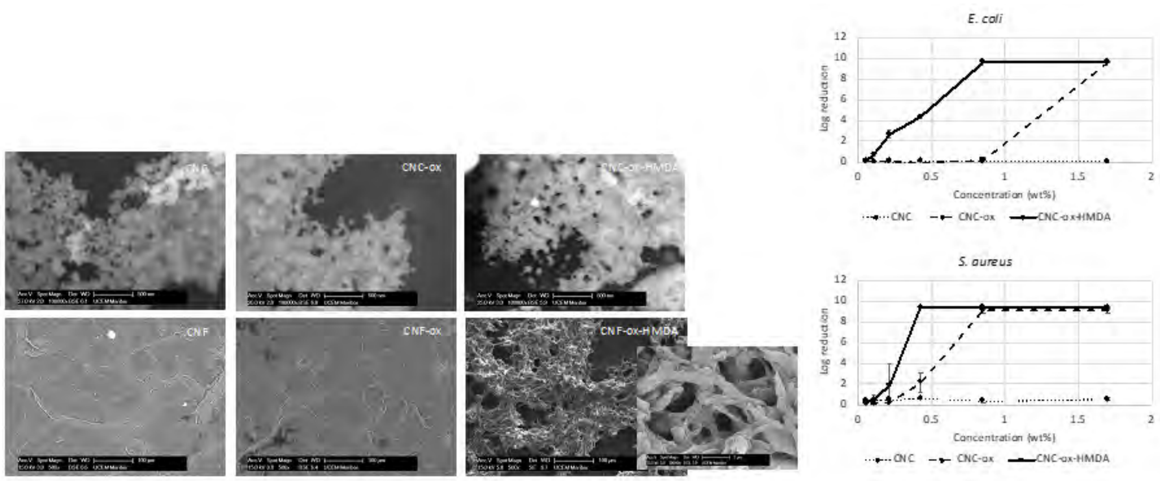

Figure 1: SEM images of native (CNF/CNC), oxidized (CNF/CNC-ox) and HMDA-functionalized (CNF/CNF-ox-HMDA) forms of samples, and the Log reduction efficacy for all CNC forms.

ACKNOWLEDGMENTS

This project has received funding from the Slovenia Research Agency (Grant agreement No. J2-1719 and research program P2-0424).

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ID 13
DIC AND DVC WITH X-RAY COMPUTED TOMOGRAPHY FOR THE MEASUREMENT OF STRAINS AT THE YARN/MATRIX INTERFACE IN SINGLE HEMP YARN COMPOSITES

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ABSTRACT
In this study, single hemp yarn composites are manufactured using epoxy matrix filled with a metal oxide powder as speckle, which allows to perform two and three-dimensional digital image correlations (DIC and DVC). An interrupted fragmentation test with tomographic acquisitions after each unloading is performed. This enables to follow the full-field strain evolution at the yarn/matrix interface throughout the test.

INTRODUCTION
Plant fiber composites compete with glass fiber composites in terms of specific mechanical properties (Manaia, 2019). This is an increasing motivation for industry to consider them in the development of their structural or semi-structural products. However, it is important to analyze the quality of the yarn/matrix interface because debonding can have consequences on the mechanical properties of a plant fiber composite (Perrier, 2017). The aim of this work consists in measuring the strains at the yarn/matrix interface in a single hemp yarn biocomposite by using two and three-dimensional image correlations (DIC and DVC). Holmes *et al.* (2022) performed DVC measurements on micro-CT scans for glass fiber reinforced composite using the fiber sections as markers. In the present work, the speckle pattern was obtained by filling the epoxy matrix with metal oxide powder. Moreover, a specific experimental procedure was developed to enable the reconstruction of the same studied volume after each *ex-situ* tensile test and each tomographic acquisition. The DIC results were obtained by using the OpenDIC software (Vanderesse, 2013) and DVC strain fields were calculated by using an open-source DVC program implemented in *MatLab* (ALDVC v1.0.3). For DIC calculations, the measurement points had a size of 15 pixels and were spaced 9 pixels from each other. For DVC calculations, their size was 20 voxels with a distance between them of 10 voxels.

RESULTS AND CONCLUSIONS
The different steps of the interrupted fragmentation test are described in Table 1, and the associated damage events observed after tomographic acquisitions are detailed. Yarn breaks, internal and surface matrix cracks successively appeared. Location of each detected damage is given in Fig. 1a. The first yarn break occurred during the second step of the fragmentation test, corresponding to a loading up to 410 N. Then, two other yarn breaks appeared during the fourth step. The internal cracks in the matrix appeared during the fifth step and all were initiated from a yarn break zone. Surface cracks were observed after the steps 5 and 6 because of the high displacement levels. Finally, the large amount of damage caused the early failure of the sample in step 7.

Table 1 Charge and displacement levels reached and associated damage events observed by using micro-CT images at each step of the fragmentation test.

Experimental step	Displacement (mm)	Charge (N)	Yarn Breaks (YB) ; internal Matrix Cracks (MC) ; Surface Cracks (SC)
1	0.190	313	-
2	0.281	410	YB1
3	0.320	410	-
4	0.348	400	YB2, YB3
5	0.450	344	MC1, MC2, MC3, MC4, SC1, SC2
6	0.437	248	SC3, SC4
7 (Break)	0.193	219	-

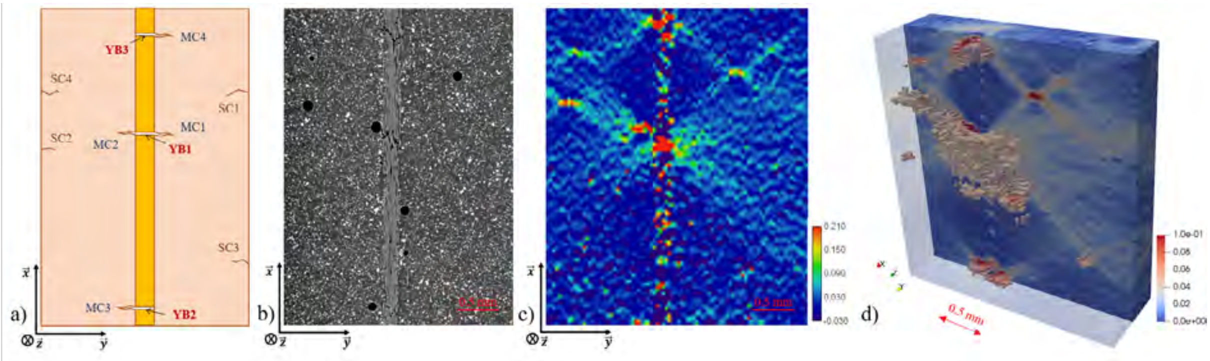

Fig. 1. a) Scheme of the observed damage events in the single hemp yarn composite. For step 5: b) micro-CT slice chosen for DIC calculation, c) longitudinal strain field ε_{xx} obtained by DIC for the chosen slice, d) 3D ε_{xx} field calculated by DVC with zones where $|\varepsilon_{xx}| > 4.5\%$ visible in the volume by transparency.

Some circular pores are visible in Fig. 1b, due to the manufacturing process. The comparison of Fig. 1c and 1d allows to validate the DVC calculation method, showing the same in-plane strain field as the DIC results. The obtained strain fields show that yarn breaks and, to a lesser degree, the pores, play the role of strain concentrators. High strain values can be seen in +45 and -45 directions from each damage, demonstrating the complex stress distribution developing around each yarn break during the fragmentation test.

ACKNOWLEDGMENTS

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ID 14
POLY (LACTIC ACID) (PLA) BIOCOMPOSITES WITH HAZELNUTS SHELL POWDER (HSP) EXTRUSION COMPOUNDING SCALE-UP AND POSSIBLE APPLICATIONS

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ABSTRACT
Two different hazelnuts shell powders (HSPs) were melt-compounded into a poly(lactic acid) (PLA) matrix. Different HSPs contents (from 20 up to 40 wt.%) were investigated, firstly at laboratory scale. The best compositions were then investigated for the up-scale in a semi-industrial twin-screw extruder. The up-scaling production of composites was successful, analytical models were adopted to predict the trend of the mechanical properties considering the interfacial adhesion between the fibers and the matrix. Based on the obtained results the best performant biocomposites compositions and HSPs granulometries were selected for injection molding and 3D printing applications.

INTRODUCTION
In the European Union about 700 million tons of biomass waste is generated by agro-industry. Interesting is the exploitation of walnut and hazelnuts shells to obtain new sustainable biocomposites. Walnut and hazelnut shells have great potential due to large-scale production of these fruits. About 67% of the total product weight consists of the shell, so that 646,818 tons of walnut shells, and 353,807 tons of hazelnut shells are produced each year [1]. Shell grinding results in hazelnut shell powder (HSP) of different sizes and morphologies, while the shell extracts can be exploited as natural antioxidants in polymeric matrices [2]. The addition of HSPs into a poly (lactic acid) PLA matrix must be deeply investigated and few works has been done regarding the scaling-up of these biocomposites into semi-industrial extrusion compounding process.
The materials used in this work are: PLA3251D from Natureworks [density: 1.24 g/cm^3; MFR (210 °C, 2.16 kg): 80 g/10 min]. Two different KERN hazelnut shell powders (HSPs) with different granulometry were provided by Arianna Fibers. HSP with coarser grain size are named H0210, while those with finer grain size are named HM200 [density = 0.954 to 1.08 g/cm^3 with HR 5 to 30%]. PLA based composites with different HSP amounts (from 20 up to 40 wt.%) were extruded at laboratory scale with a Haake Minilab II (HAAKE, Vreden, Germany) twin-screw mini-compounder and the strand coming out from the microcompounder was cooled and pelletized to obtain granules that were injection molded for thermo-mechanical characterizations. The best composition of both HSP typologies was then scaled-up on a semi-industrial Comac EBC 25HT (L/D = 44) twin screw-extruder. An overview of the biocomposites compositions and main results is reported in Table 1.

RESULTS AND CONCLUSIONS

The powders' addition makes the material more brittle with a decrement of both stress and elongation at break. However, the HSPs addition, increases the elastic modulus. The better mechanical response achieved with H0210 can be attributed to several factors: H0210 has a greater diameter distribution that represents a more efficient obstacle towards the crack that advances in Charpy tests. From the application of Pukanszky B parameter, a decrement of the B value from 1.41 for H0210 to 0.56 emerged for HM200 explaining the better mechanical properties obtained with H0210.

Table 1 Mechanical and MFR results of lab-scaled composites with different amounts of H0210 and HM200 HSP.

Blend name	HSP wt.%	Elastic modulus (GPa)	Stress at break (MPa)	Elongation at break (%)	Charpy Impact Resistance C.I.S. (kJ/m²)	MFR (g/10 min)
PLA	0	3.6	58.9	2.3	2.2	3.8
PLA_20_H0210	20	4.0	40.8	2.6	2.6	22.7
PLA_30_H0210	30	4.2	33.8	2.7	2.7	13.8
PLA_40_H0210	40	4.3	27.4	2.9	2.9	8.5
PLA_20_HM200	20	3.9	30.3	2.4	2.4	32.9
PLA_30_HM200	30	4.1	26.8	1.7	1.7	34.1
PLA_40_HM200	40	4.4	16.8	1.7	1.7	33.4
PLA*		3.6	64.6	2.7	2.5	3.2
PLA_30_H0210*		4.3	37.4	1.1	2.6	6.2
PLA_30_HM200*		4.4	38.4	1.4	2.3	4

*Blends extruded with a semi-industrial COMAC twin-screw extruder (scaled-up).

The MFR values of all biocomposites are higher than pure PLA probably due to a slight PLA hydrolysis caused by the not efficient moisture removal from HSP. The lab-scale data with 30 wt.% of HSPs seem promising granting a high fiber content and acceptable mechanical properties and thus were scaled-up. The MFR decrement for the scaled-up biocomposites is attributed to the coupling of a lower extruder residence time and the presence of the extrusion venting system that guarantees a highly efficient humidity stripping during the melt extrusion that avoids or limits any eventual PLA degradation. The mechanical results are noteworthy. The scaled-up composites show higher values of tensile strength and elastic modulus confirming the efficiency of the venting system in removing the HSPs moisture.

ACKNOWLEDGMENTS

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ID 15
OIL/WATER SEPARATION PROPERTIES OF SUPERHYDROPHOBIC COTTON FABRICS

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ABSTRACT
The presented study proposed a simple and low-cost approach for improvement of superhydrophobic properties of cotton fabrics by coating mechanically activated fly ash particles and low surface energy chemicals. The water contact angle was increased to 140°, 144° and 151° for fly ash concentration of 0.5, 1 and 2 wt.% respectively after subsequent treatment of Trimethoxy(octadecyl)silane (OTMS) on fly ash coated fabrics. Furthermore, the coated fabrics showed great potential for separation of floating oil layer, underwater oil droplet or oil/water mixture. The separation efficiency of 97%, 95%, 96% and 94% was obtained for selected model oils toluene, n-hexane, chloroform and petro ether, respectively.

INTRODUCTION
The superhydrophobicity of solid surfaces has been investigated with considerable attention over the past few years due to the increased demands of impermeable textiles, self-cleaning coatings, non-adhesive coatings, microfluidic devices, biosensors, outdoor antennas, etc. [1,2]. Based on superhydrophobic species existing in nature (e.g. lotus leaves, butterfly wings, and water strider legs), surface roughness and low surface energy of materials can be considered as the two important factors to obtain superhydrophobic surfaces [3]. The different methods such as electrospinning deposition, layer-by-layer assembly, chemical etching, phase separation, chemical vapor deposition, colloid assembly, etc. have been employed to obtain superhydrophobic surfaces [4,5]. However, there are still many challenges on large scale fabrication of superhydrophobic surfaces by inexpensive, simple, and eco-friendly techniques. In this work, the surface of fly ash was mechanically activated using a ball milling process to create effective and durable coating on cotton fiber surface. The milled fly ash particles were decorated on cotton fiber surfaces at 0.5, 1 and 2 wt.% concentration. Then, non-fluorinated silane (i.e., OTMS) was applied on fly ash coated fabrics to impart low-surface energy characteristics. The contact angle measurements were performed to determine their superhydrophobic properties, and potential applications for oil in water separation performance were studied. To the best of the authors' knowledge, this is the first study on oil in water separation properties of fly ash/OTMS coated superhydrophobic cotton fabrics derived from cotton fibrous wastes.

RESULTS AND CONCLUSIONS
The effect of fly ash particles on surface wettability of fly ash-OTMS coated cotton fabrics was investigated from measurements of static water contact angle, and roll-off angle. When fly ash particles were applied on cotton fabric with subsequent treatment of OTMS, the contact angle was enhanced and roll off angle was further decreased. The static contact angle of 140°, 144° and 151° and roll off angle of 16°, 11° and 8° were measured for fly ash concentration of 0.5, 1 and 2 wt.% respectively. The enhancement in water repellency can be attributed to the formation of unique two-tier structural surface combined with the micros-

caled cotton fibers (10 µm) and the nanoscaled fly ash particles (1000 nm). Furthermore, the oil separation efficiency of fly ash-OTMS coated cotton fabrics was quantitatively estimated in separate experiments using four model oils of different densities (i.e., toluene, n-hexane, chloroform and petro ether). When the mixture of oil (dyed with red orange) and water was poured onto the superhydrophobic textile, the oil was quickly passed through the coated fabric into the small beaker (see Figure 1). On the other hand, water was accumulated on the cotton surface, and then decanted into the big beaker from the top of the coated fabric. The separation efficiency of 97%, 95%, 96% and 94% was obtained for toluene/water, n-hexane/water, chloroform/water and petro ether/water, respectively.

Figure 1. Oil/water separation performance of fly ash/OTMS coated cotton fabrics

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ID 16
FIBROUS COMPOSITES PREPARED BY AIRLAYING

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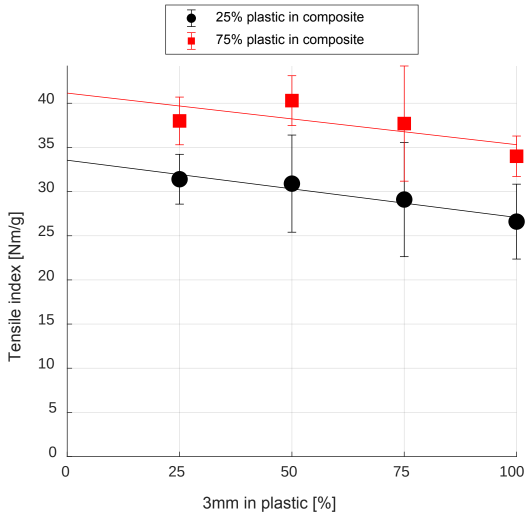
ABSTRACT
This work describes the use of the airlaying process as a manufacturing method for fibrous composites from cellulose. Relatively dense composite sheets were prepared with the short fiber airlaying technique from fluff pulp (75 wt% and 25 wt%) and conventional thermoplastic PP/PE bicomponent fibers (25 wt% and 75 wt%) as a mixture of 3mm and 12 mm long fibers. The effect of the fiber mixture ratios on the tensile and impact strength properties of composites was investigated. The results show that these properties were proportional to weight ratios of components.

INTRODUCTION
Short-fiber (< 6mm) airlaying (or airlaid or airforming) is a commonly known dry web formation process. The fibers are suspended into an air stream and deposited onto a moving permeable wire and bonded with heat, chemicals or mechanical action. Airlaid presents a potential alternative to traditional composite making processes such as injection molding that involves degradative high temperatures and shortens the fibers. Airlaid is a dry and delicate processing method. It saves water, reduces energy use via lack of drying, and preserves raw material properties. In addition, various lignocellulosic and thermoplastic fibers can be processed.
In composite making, airlaid acts as a dry blending and sheet formation process, and is followed by thermal bonding by heat pressing. Depending on the ratio between thermoplastic polymer and fluff pulp, the sheet may resemble either a wood-plastic composite (appr. 70% plastic) or a regular thermally bonded airlaid (15-20% plastic). The nonwoven airlaid process is not a common technique to manufacture composites, especially without additional processes such as needling. However, there are research reports on various air-assisted forming techniques for making, e.g., PLA/flax biocomposites (Alimuzzaman et al 2013) or novel recycled materials (Dieckmann et al. 2019)
Composite sheets (400 gsm, approximately 540 kg/m³) were prepared with fluffed softwood kraft pulp (fluff) and PP/PE (bico) fibers (1.7 dtex) as the bonding fraction (dosage 25 wt% or 75 wt%). The bonding fraction was a mixture of 3 mm and 12 mm long fibers (0 wt% to 100 wt%). The fibers were pre-mixed in a kitchen blender, airlaid to A4 size sheets (with a Walkisoft drum former) and thermally bonded at 145°C and 6.7 bar (pressure on sheet) for 90s. Tensile and impact testing were done. The aim was to study the effect of plastic dosage and mixed fiber lengths in the thermoplastic fraction.

RESULTS AND CONCLUSIONS
The results from the tensile tests are shown in Fig. 1 and from the impact test in Fig. 2. The share of 3mm bico in plastic fraction is shown on the x-axis, the rest is 12 mm bico.
The increasing plastic content increased both the tensile index and the impact strength of the airlaid composites. The increasing fraction of 3mm bico fibers reduced these properties. The other tensile properties, with the exception of E-modulus, followed the same behavior. This suggests that the studied properties

were linearly dependent on the weight fractions of the components. This concept is known as the rule-of-mixtures. It is widely applied to predict composite properties (Kim et al. 2001).



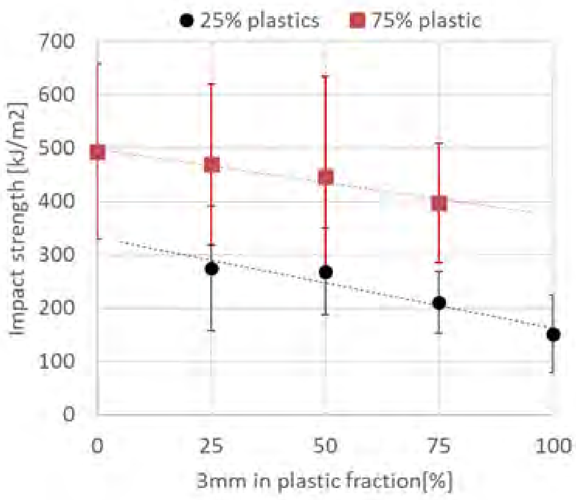


Fig.1 Tensile index of the composite. Mean (N=10), 1std, and linear regression trend lines.

Fig.2 Impact strength. Mean (N=10), 1std, and linear regression trend lines.

In this study, composites were successfully prepared with the airlaying nonwoven process. The results showed that the tensile and impact properties of airlaid composites followed the rule-of-mixtures concept. This applied to a situation when the bonding fiber fraction contained two fiber lengths. PP/PE fibers can be replaced by biobased or biodegradable materials, such as PLA-bico.

ACKNOWLEDGMENTS

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ID 17
CELLULOSE NANOFIBER-BASED AEROGELS FROM WHEAT STRAW: INFLUENCE OF SURFACE LOAD AND LIGNIN CONTENT ON THEIR PROPERTIES AND DYE REMOVAL CAPACITY

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ABSTRACT
In this work, (ligno)cellulose nanofibers ((L)CNF) from wheat straw residues were obtained using two types of pre-treatments: mechanical (Mec) and TEMPO-mediated oxidation (TO), to obtain different consistency bioaerogels (0.2, 0.4, 0.6 and 0.8 wt%), physical and mechanical properties, and their adsorption capacities as dye removers were further studied. An inversely proportional relationship was observed between the consistencies of the aerogels and their achieved densities. Despite the increase in density, all samples showed porosities above 99%. In terms of mechanical properties, the best results were obtained for the 0.8% consistency LCNF-Mec and CNF-Mec aerogels, reaching 67.87 kPa and 64.6 kPa for tensile strength and Young's modulus, respectively. In contrast, the adsorption capacity of the aerogels was better for TEMPO-oxidized aerogels, reaching removal rates of almost 100% for the CNF-TO5 samples. Furthermore, the residual lignin content in LCNF-Mec aerogels showed a great improvement in the removal capacity, reaching rates higher than 80%, further improving the cost efficiency of the samples due to the reduction in chemical treatments.

INTRODUCTION
Water pollution is one of the most serious problems worldwide that not only disrupts water supplies but also endangers public health. Dyes are an important source of contamination due to its large-scale use in different industries (for textile, leather, paper printing, etc.). In recent years, there has been an interest in the study of new methods for the removal of dyes from water (Carpenter et al., 2015). Different treatments have been used, being adsorption among the most effective treatment. Bioaerogels made from renewable resources are a great alternative for water decontamination. In this sense, the cellulose content present in some crop residues has the potential to produce lignocellulose-based materials. Wheat straw (WS) is the principal harvest by-product that presents a high source of cellulose that can be valorized to produce cellulose nanofibers (CNF). The high aspect ratio, specific surface area and anionic charge of nanocellulose makes it ideal for water purification, especially for cationic dyes such as methylene blue (MB).
The different cellulosic fractions (bleached pulp (WS-BP) and unbleached cellulosic pulp (WS-UP)) were delaminated to produce cellulose nanofibers (LCNF for WS-UP and CNF for WS-BP). Two different pre-treat-

ments (Mechanical (Mec) and TEMPO-mediated oxidation (TO)) were used to facilitate the delamination of the fibers in the nanofibrillation treatment. Different consistencies of CNF suspensions were prepared and freeze dried obtaining the bioaerogels. These were characterized in terms of porosity, density, mechanical properties (evaluated by compression tests with a strain limit of 80%, based on the initial aerogel's height, at a speed of 2 mm/min), and adsorption capacity using MB as the pollutant dye (immersion of 100 mg of aerogel in 50 mL of MB solution at MB concentrations of 5 and 20 mg/L under constant magnetic stirring at ambient temperature for 24 h).

RESULTS AND CONCLUSIONS

The effect of the applied pre-treatment (Mec and TO) on the properties of the nanofibers showed that both the efficiency of the different pre-treatments together with the mechanical and adsorption properties of the different bioaerogels were influenced by the initial composition of the fiber. Furthermore, the physical and mechanical properties of the aerogels showed a direct relationship with the initial solid concentration employed: both density and mechanical strength values increased from 4 to 16 g/cm3 and from 16 to 67 KPa when the consistency was incremented from 0.2 to 0.8%. Despite the increase in density, all aerogels showed porosities above 99%, showing a slight reduction with increasing density. Focusing on the adsorption performance, the dye removal was slower for aerogels prepared from nanofibers obtained by mechanical treatment compared to TO cellulose nanofiber aerogels. Regarding the pollutant concentration, an increase in the initial concentration of MB resulted in a faster adsorption, needing 3 h for 80% dye removal and 2 h for the same dye removal performance for concentrations of 5 mg/L and 20 mg/L, respectively. Furthermore, the lignin content had a significant effect on the adsorption properties of aerogels derived from nanofibers obtained by mechanical pre-treatment, reaching removal values of 80–90% for LCNF-Mec, similar to the values obtained by TEMPO-oxidized cellulose nanofiber aerogels, and decreasing to 50–60% for CNF-Mec.

This study shows that nanocellulose-based aerogels show an excellent adsorption capacity which making them ideal for water purification. The high concentration of lignin functional groups that can interact with contaminants, together with the high adsorption capacity of cellulose nanofibers, demonstrated a synergistic effect in their use as materials with a high adsorption capacity. This work provides information that could be used to create useful, environmentally friendly, cost-effective, and efficient adsorbents for the removal of cationic dyes in different wastewater industries. However, further research needs to be performed in order to establish and implement processes for the reuse of these materials, which is relevant to ensure a fully sustainable use of these materials.

ACKNOWLEDGMENTS

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HYDROXYL GROUP ACCESSIBILITY IN CELLULOSE FIBER WITH DYNAMIC VAPOR SORPTION TECHNIQUE

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ABSTRACT

The mechanism of water adsorption in cellulose can be studied by evaluating the quantity and state of the adsorption sites, and hydroxyl groups [1,2]. Novel cellulosic products, such as biofuels and nanofibrils, require chemical modification. Cellulose accessibility can be considered a decisive factor during these chemical or biochemical treatments. Alkali improves the swelling of the aqueous cellulose suspensions due to the ionization of the hydroxyl groups and cleavage of inter- and intra-molecular hydrogen bonds [3]. The Dynamic Vapor Sorption (DVS) can be used to quantify the amount of accessible hydroxyl groups in cellulosic materials. In the current study, the amount of accessible OH groups, is defined by those OH groups that can be exchanged to OD groups by deuterium exchange. Using gravimetric techniques such as DVS, the amount of deuterium exchange can be quantified by an increase of 1g per mol exchanged hydrogen [4].

INTRODUCTION

The Dynamic Vapor Sorption (DVS) technique uses a saturated carrier gas flow over a sample resulting in a change in mass. The amount of accessible hydroxyl groups was quantified by measuring its mass increase during deuterium exchange in DVS.

Material: water-based nano-cellulose (Sample 1), water-based cellulose microfibrils (Sample 2) Microcrystalline Cellulose (PH-101)

Experimental details: Multiple adsorption-desorption cycle experiments were conducted with water between 0 and 90% RH at 25°C, until all the OH group sites were saturated resulting in a stable mass using dm/dt of 0.0005 % min^{-1}. The solvent was then changed to D_2O for a repeat experiment.

The accessible OH groups were then quantified according to Equation 1 shown below:

Equation 1:

$$= \frac{-}{(\quad - \quad)} \; 1000 \; (\qquad ^{-1})$$

Where ***A*** is the accessible OH group content in the dry mass of the sample, m_i is the dry mass of the sample before exposing it to D_2O vapor (g), m_f is the dry mass of the sample after the D_2O exposure (g), M_D is the molar mass of deuterium (2.014 g mol^{-1}) and M_H is the molar mass of hydrogen (1.008 g mol^{-1}).

RESULTS AND CONCLUSIONS

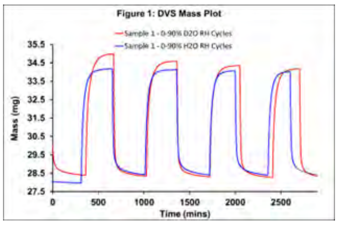

Figure 1. H_2O, D_2O sorption kinetics sample 1

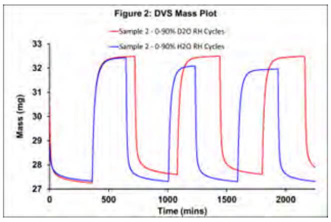

Figure 2. H_2O, D_2O sorption kinetics sample 2

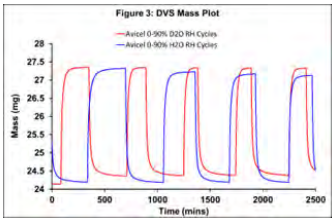

Figure 3. H_2O, D_2O sorption kinetics Sample (PH-101)

The accessible OH groups were measured using Equation 1. The repeatability and reproducibility of the measurements are established by different values of m_f from multiple cycles. The water and D_2O moisture sorption and desorption kinetics for the samples during the multi-cycle experiments are shown in Figures 1-3. As the water vapor cycles continued, the dry mass of the sample slowly decreased, suggesting structural changes within the cellulosic material resulting in lower porosity and solvent accessibility. The average accessibility measurements for all the samples are tabulated in Table 1. The value for Microcrystalline Cellulose PH-101 is in good agreement with the value reported by Väisänen [2].

Table 1: Accessible OH Group content of the samples based on DVS Measurements.

Sample	Drying conditions	Accessible OH group content (mol/kg)
Microcrystalline Cellulose PH-101	Never Dried	9.94
Microcrystalline Cellulose PH-101	Never Dried	9.4 ± 0.4*
Sample 1 - Water based nanocellulose	22 hours under fume hood	18.05
Sample 2 - Water based cellulose microfibrils	22 hours under fume hood	12.32

*Value reported by Väisänen et al.[2]

The current study shows that DVS can be used for direct quantification of the accessible OH groups in cellulosic materials and the robustness of the method is further shown through the Microcrystalline cellulose, PH-101 data , where the results for accessible OH group is in good agreement with literature data published by Väisänen [2].

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ID 19
ACOUSTIC INVESTIGATION ON THE SOUND-ABSORBING PROPERTIES OF POSIDONIA FIBRES

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ABSTRACT
This article illustrates the results of an experimental acoustic characterization carried out on fibers obtained from Posidonia Balls, fibrous aggregates found on Mediterranean beaches made up of residues of Posidonia Oceanica, a marine plant endemic to the Mediterranean which forms vast meadows on sandy sea beads. The aim is to explore the potential of using these fibers as an eco-sustainable sound-absorbing material.

INTRODUCTION
Posidonia Oceanica is a marine plant, endemic to the Mediterranean Sea with ribbon-shaped leaves that can be up to one meter long. Once dead, due to the action of the sea waves and atmospheric agents, the fibrous foliar residues of the Posidonia thickly intertwine to form the characteristic brown balls; essentially the production process of these fibers is carried out entirely by the sea, thanks to the action of the water and the wave motion which frees the fibers from the vegetable material, compacts them into ellipsoidal aggregates and deposits them on beaches [1].
From the acoustic point of view, these materials have been studied in [2] with an in-depth analysis on the physical and acoustic characteristics and on the modeling of regular packets of these spheres as metamaterials.
This research aims to explore the possible acoustic application of loose fibers obtained with a manual process from Posidonia Balls; experimental tests were conducted by compacting the fibers, obtained from Posidonia Balls collected on a beach in Sardinia, with different densities, in order to obtain an analytical model capable of calculating the sound absorption coefficient of this material for any combination of thickness and density.

RESULTS AND CONCLUSIONS
Samples of fibers with densities ranging between 48 and 210 kg/m^3 and thicknesses between 30 and 130 mm were tested experimentally to determine the airflow resistivity, the porosity and the acoustic absorption coefficient. From these measurements, through interpolations and analytical models, the parameters of the JCA model [3,4] were obtained (Fig. 1) as a function of density, in order to model the propagation of sound in the fibrous material considered as an equivalent dissipative fluid. Figure 2 shows a comparison of the normal incidence sound absorption coefficient obtained from the experimental measurements and with the JCA model.
As can be seen, Posidonia fibers have excellent sound-absorbing properties, comparable to many industrial materials such as mineral fibers, polyester fibers or expanded polyurethanes. The proposed model allows to calculate the sound-absorbing properties of different combinations of thickness and density with

excellent precision, and represents a design tool for materials based on Posidonia fibers.

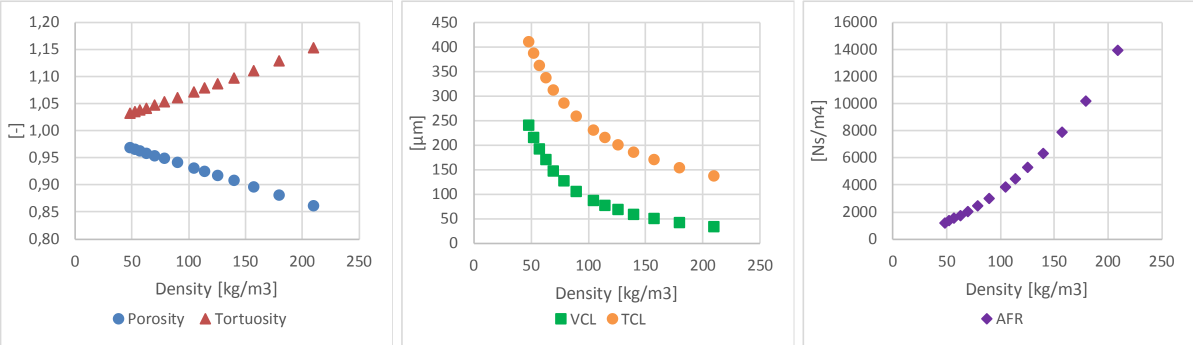

Fig.1 Physical parameters of the JCA model as a function of the density of the loose fibers (VCL=Viscous Characteristic Length, TCL=Thermal Characteristic Length, AFR=Airflow resistivity)

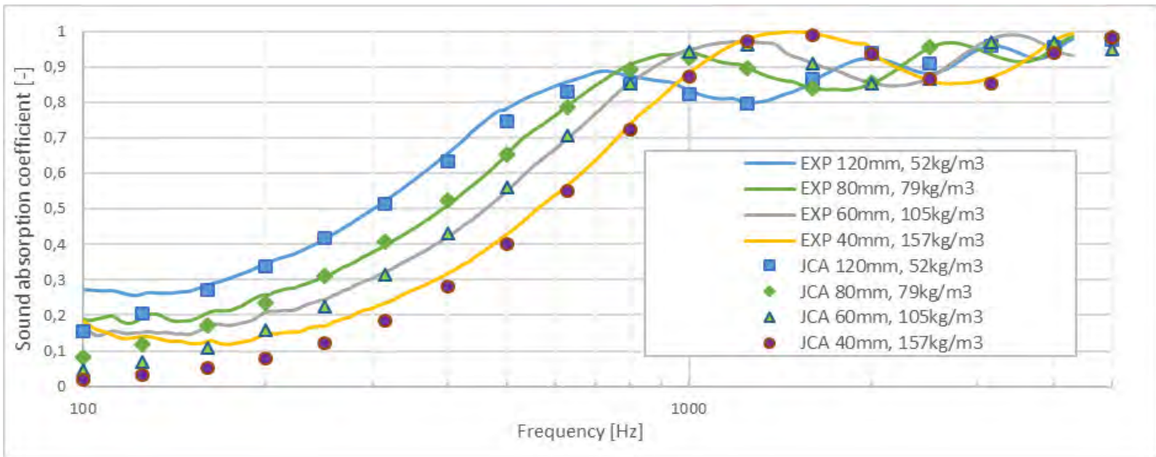

Fig.2 Comparison between experimental measurements (EXP) and results obtained from the JCA model with the physical parameters of figure 1 (JCA). For each sample the thickness and density are indicated.

ACKNOWLEDGMENTS

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ID 20
PHYSICAL AND MECHANICAL PROPERTIES OF BABASSU COCONUT EPICARP FIBERS

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ABSTRACT
This work presents the physical and mechanical properties of babassu coconut epicarp: density, absorption of water, degradation, tensile strength, tensile modulus and elongation at break. The tests were made to identify and compare the results with others fiber that can integrate a biocomposite.

INTRODUCTION
Natural fibers have been used since ancient times, in an isolated or composite form, in objects such as storage containers, defense tools, locker rooms, construction purposes, among others. The most used fibers are sisal, coconut, palm, hemp, jute. In the 20th century, the use of natural fibers quickly emerged due to their low density, good strength and high stiffness, in addition to being a renewable and biodegradable material (Guven et al, 2016). A biocomposite is a material composed of two or more distinct constituent materials (one being naturally derived) which are combined to yield a new material with improved performance over individual constituent materials.
In part of the north and northeast region of Brazil, there are babassu coconut palm trees, a fruit considered the largest native oil resource in the world and its extraction is an important socioeconomic source for the local population (EMPBRAPA, 1984). The fruit is composed of epicarp (11%), mesocarp (23%), endocarp (59%) and almond (7%). For oil extraction, only almonds are used, discarding the shell, but this can be used mainly as charcoal, flour, fertilizer, among others (TEIXEIRA, 2008). The main components of the babassu coconut epicarp are: lignin (14.73%), cellulose (38.65%) and hemicellulose (22.21%), and ash content of 14.61% (LEMOS at al., 2016).

RESULTS AND CONCLUSIONS
The density and water absorption tests were carried out according to the methodology of Oliveira, 2011. The fiber degradation test was adapted from Mariani, 2016 and the tensile strength test adapted from ASTM D2256-1. Table 1 shows the test results.

Table 1. Results of density, water absorption, degradation and tensile strength tests of the babassu coconut shell.

Density (g/cm^3)	Water absorption (%)	Degradation (%)	Tensile strength (Mpa)	Modulus of elasticity (Gpa)	Elongation (%)
1.038	75	8.75	8.90	1.14	7.78

The water absorption test was carried out for 4 days and it was identified that the fiber absorbs water mainly in the first hours, until the first 120 minutes (approximately 90%) and after 2 days the fiber practically does not absorb more water, presented maximum value of 105% (Fig. 1).

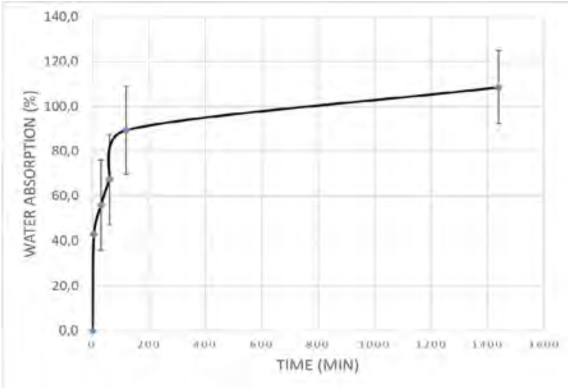

Fig.1 Water absorption of epicarp fibers

This study showed that compared to other natural fibers that are already used in biocomposites - such as sisal, jute, coconut and ramie - the epicarp fibers of the babassu coconut presented similar density, superior water absorption, resistance to traction and modulus of elasticity inferior to all these fibers.

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ID 22
NON-NATIVE PLANT-EXTRACTED FIBROUS NANOCELLULOSE WITH ENHANCED ALPHA-AMYLASE ENZYME INHIBITION ACTIVITY

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ABSTRACT
Arundo donax, commonly known as Giant reed or Cana vieira, is an invasive plant introduced to Europe, especially in southern Europe, and has been widely dispersed as non-native species in Mexico, Pacific islands, the Caribbean, southern Africa, South America, and sub-tropical USA. This plant has affected the growth of native vegetation, which eventually reduces the soil quality and affects the food chain in the region. Until now, there is no usable process to reuse the phytochemicals present in these invasive plants, which will reduce their spread and benefit the desired application. It should be noted that A. donax fibers possess 35% hemicellulose, 31% cellulose, and 18% lignin, making them a better cellulose source than common sources from native plants. Thus, the present work aims to extract cellulose from the Arundo donax leaf via the organosolv fractionation and bleaching method, followed by centrifugal fractionation to produce fibrous nanocellulose. In addition, the alpha-amylase enzyme inhibition activity of the nanocellulose produced was also investigated. These results demonstrate that it is possible to identify new applications for invasive plants, thus contributing to their eradication in the territories where they are planted, potentially replacing in medical applications other plants with food and/or other economic interests.

INTRODUCTION
Arundo donax is a large perennial grass that is native to temperate and tropical regions, especially indigenous to the Mediterranean region. After 1960, this plant was introduced to Europe, and has been widely dispersed as non-native species in Mexico, Pacific islands, the Caribbean, southern Africa, South America, and the sub-tropical USA. A. donax, as an invasive plant, has affected the growth of native vegetation, eventually reduces the soil quality and affecting the food chain in the region. In general, this invasive plant is removed from the environment via mechanical (mowing, tilling), chemical (pesticides), and biological (insect predators or plant diseases) approaches. Each of these approaches also has various limitations, including costs and a longer time to eliminate or reduce non-native plants from the environment [1]. Until now, there is no advantageous process to reuse the phytochemicals present in these non-native plants, which will reduce their spread and be beneficial for the desired application. Further, it is noteworthy that A. donax possesses 35% hemicellulose, 31% cellulose, and 18% lignin, making them a better cellulose source than common non-native plant sources. In addition, the extraction of A. donax will help in reducing their population in the environment and reuse it as a green source of nanocellulose for biomedical applications instead of depending on medicinal or economically significant plants [2]. Recently, several studies have identified that nanocellulose has significant amylase enzyme inhibition activity with anti-diabetic prop-

erties, which is essential to convert carbohydrates and starch to glucose, fructose, or maltose [3]. Thus, the present work aims to extract cellulose from A. donax leaf extract using organosolv fractionation and bleaching methods, followed by centrifugal fractionation to produce fibrous nanocellulose. In addition, the inhibition activity of the alpha-amylase enzyme of the nanocellulose obtained was also investigated.

RESULTS AND CONCLUSIONS

Our results from Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) show (Fig. 1 (A-C)) the formation of cubic nanocellulose in the fibrous matrix. Fig. 1 (D) showed that the nanocellulose possesses dose-related alpha-amylase enzyme inhibition ability, where 80 mg/ml of the sample contains ~60% enzyme inhibition ability.

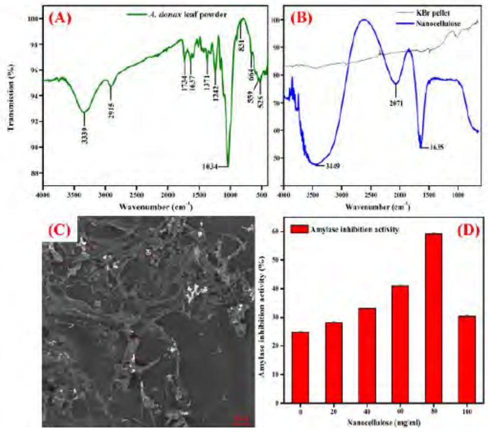

Fig.1 (A) Functional group of analysis of A. donax leaf powder and (B) Nanocellulose; (C) Morphology analysis of nanocellulose and (D) inhibition activity of nanocellulose at 0 h.

This study demonstrates that nanocellulose from a non-native plant source (A. donax) possesses anti-diabetic properties. Further, the produced nanocellulose from A. donax will be formulated in biopolymers for controlled and sustained release. Our results show that it is possible to find new applications for invasive plants, contributing to their reduction in the territories where they are planted, giving them a noble use, and eventually replacing other plants that may have significant applications both from food and other economic applications.

ACKNOWLEDGMENTS

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ID 24
INCREASING THE TOUGHNESS OF BRITTLE BAST FIBRE COMPOSITES MADE OF FIBRE WEBS USING CELLULOSE ACETATE FOILS

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ABSTRACT
The problem with bast fibre-reinforced plastics is their relatively high brittleness compared to many other fibre-reinforced plastics. The present study aims to improve the toughness of kenaf/bio-based epoxy composites manufactured from multilayer fibre webs by compression moulding with cellulose acetate (CA) foils as an impact modifier. Results have shown a significant improvement in toughness by using CA foils placed between the layers of multilayer fibre webs. Other mechanical properties such as tensile strength, flexural strength and modulus of elasticity were not negatively affected.

INTRODUCTION
The low toughness of bast fibre-reinforced plastics often limits their application to materials that are not exposed to higher impact stress (George et al., 2016). Cost-effective bast fibres are usually processed into fibre webs or needle felts. The use of needle felts or fibre webs in composite applications is mostly limited to non-visible cladding parts that are subjected to less mechanical stress. However, in principle, these materials can also be used for higher-quality applications. One approach to improve the mechanical properties is a hybridisation of bast fibres with higher-performance fibres such as carbon or glass (Saleem et al., 2020). In the study presented here, an approach is proposed to process bast fibre webs with CA foils to improve the toughness. In the present study, multilayer fibre webs of kenaf staple fibres are processed with a fibre mass fraction of ~40% in combination with a bio-based thermosetting epoxy matrix (Greenpoxy – GP) using a cold compression moulding process at room temperature using 2 mm thick spacers. To modify the impact strength, different numbers of CA foil layers (1 to 5) were symmetrically distributed between the layers of the bast fibre webs. The foils were previously punched with a needle with small holes (approx. 8-10 holes/cm²) so that the liquid matrix could penetrate the foils and the kenaf layers are better connected to the foils. After the plates had hardened, specimens were prepared for the following experiments. The composites were tested with tensile, bending and unnotched Charpy impact tests. The aim was to improve the toughness of the brittle bast fibre-reinforced plastics without significantly affecting the tensile and flexural properties.

RESULTS AND CONCLUSIONS
The results of the mechanical tests demonstrate that the insertion of CA foils between the multilayer fibre webs does not affect all properties. Fig. 1A shows that the tensile strength of the kenaf/GP composites is not significantly affected by the presence of CA foils in the composites, regardless of whether one or five foils were inserted. Similarly, other properties such as flexural strength, flexural modulus and Young´s modulus (cf. Tab. 1) were not affected. The work at break (Tab. 1) and the impact strength (Fig. 1B), on

the other hand, were significantly influenced by the CA foils. It is evident that the impact strength of the pure kenaf/GP sample is below the impact strength value of the pure matrix. When using three CA foils, the impact strength could be increased significantly above the value of the pure matrix. By using four foils, a further significant increase is achieved. This represents an increase in impact strength by a factor of 2.4 compared to the pure matrix and a factor of 3.8 compared to the kenaf/GP sample. From the use of four foils onwards, no further increase in impact strength was observed.

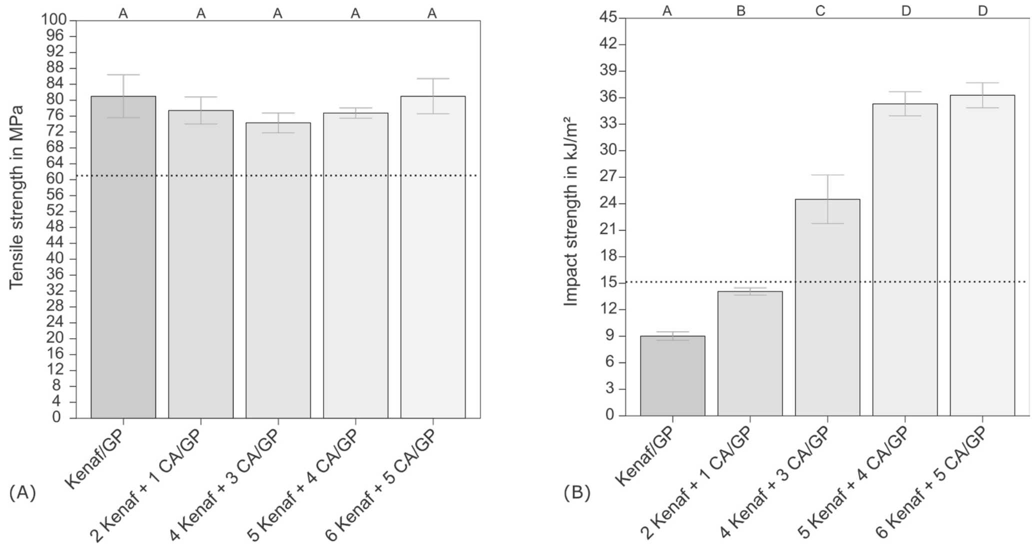

Fig.1 Tensile strength (A) and unnotched Charpy impact strength (B) of kenaf/Greenpoxy (GP) composites with different amounts of CA layers (the dotted line shows the values for the pure GP matrix). Significant differences are labeled with different letters.

The aim of improving the toughness of brittle bast fibre-reinforced composites significantly, without negatively affecting the tensile and flexural properties by introducing CA foils, was achieved. Incorporating or co-needling CA foils in thermosetting composite applications could contribute using needle felts made of bast fibres in applications where a higher toughness is required.

Table 1 Mechanical characteristics of kenaf/Greenpoxy (GP) composites

Sample	Flexural strength in MPa	Flexural modulus in GPa	Young´s modulus in GPa	Work at break in Nmm
Kenaf/GP	116 ± 6	7.3 ± 0.5	9.2 ± 1.1	2897 ± 461
2 Kenaf + 1 CA/GP	110 ± 5	7.9 ± 0.3	9.8 ± 1.1	2325 ± 388
4 Kenaf + 3 CA/GP	117 ± 12	7.8 ± 1.2	9.5 ± 1.6	2618 ± 481
5 Kenaf + 4 CA/GP	114 ± 5	8.1 ± 0.1	10.4 ± 0.5	3033 ± 358
6 Kenaf + 5 CA/GP	124 ± 12	7.3 ± 0.9	10.3 ± 0.8	3449 ± 540

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ID 26
NEW RELIABLE METHODOLOGIES FOR DISTINGUISHING VIRGIN AND RECYCLED CASHMERE FIBERS

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ABSTRACT
Cashmere plays an important role in luxury factory due to its characteristic. Since many companies start to produce and market recycled cashmere textiles, objective and quantitative methods to identify and possibly quantify recycled cashmere are crucial in particular to discourage the adulteration. Available analytical methods for the identification of recycled and virgin fibers are currently confined to subjective assays. For this reason, two different techniques have been studied for having an objective, fast and reliable analysis for fiber discrimination. The first approach involves the use of the NIR spectroscopy coupled with chemometric assessment. The second approach is based on dye adsorption investigated with UV-vis. Finally, both techniques were applied to blends of different proportions of virgin and recycled fibers to evaluate the detection limits.

INTRODUCTION
Cashmere is generally known as a luxury fiber with unique properties of softness, thinness, warmth and rarity. These special characteristics made cashmere widely used for-high value adding by the textile industries. On the other hand, recycled cashmere is made of fibers that are recovered from post-factory scraps and post-consumer textile wastes that are shredded and re-spun through a mechanical process (Niinimäki & Karell, 2020). Nevertheless, recycled fiber quality cannot be compared to virgin fibers since the mechanical operations of recycling cause some extent of damage to their morphological and mechanical properties. Anyway, the quality can sufficiently achieve the requirements for clothing applications since they were often added with virgin fibers to create yarns (Bhatia, Sharma, & Malhotra, 2014). Thus, new reliable methodologies for distinguishing virgin cashmere from recycled are becoming fundamental to avoid counterfeiting in cashmere garments. Nowadays, the most common way to identify recycled fibers is based on morphological studies by microscope; therefore, it can be affected by possible evaluation errors. Consequently, to minimize measurement uncertainty, two novel techniques can be performed. The first procedure involves the use of the NIR spectroscopy coupled with chemometric analysis (Anceschi et al., 2022b). The second method exploits the different adsorption capacities related to diverse kinetic and thermodynamic parameters associated with the dye staining by the virgin and recycled cashmere (Anceschi et al., 2022a).

RESULTS AND CONCLUSIONS

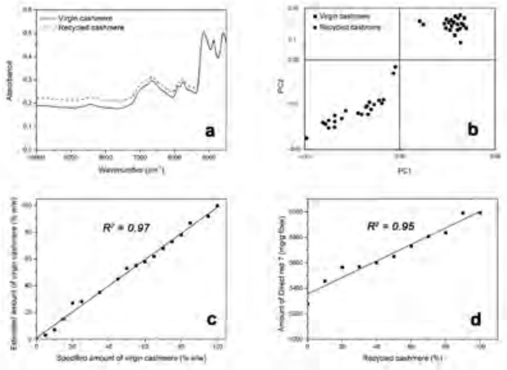

Figure 1: a) NIR spectra; b) P.C. analysis; c) NIR calibration curve; d) UV-vis calibration curve

Since the overall shape of the NIR spectra (Fig. 1a) is very similar for both samples due to the same chemical composition, a significant cluster tendency is revealed by the P.C. analysis (Fig. 1b). Indeed, the first principal component explained 91% of variation between the virgin and recycled cashmere whereas the second component is 8,5% This results indicates that differences in physical and morphological characteristics of the fiber can be stressed by chemometric analysis allowing the discrimination of chemically similar samples. Similarly, the UV-vis spectroscopy proved to be effective in virgin and recycled cashmere discrimination. The fibers were stained with various amounts of C.I. Direct Red 7 in different conditions and the exhaust dye baths were analyzed by UV-visible spectroscopy in order to determine the thermodynamics and kinetics parameters associated with the dye adsorption. Overall, it can be concluded that the ΔG^0 value indicates the process is feasible and spontaneous for both samples. The negative ΔH^0 value suggests the exothermic nature of adsorption and the ΔS^0 can be used to describe the randomness at the cashmere-solution interface. In order to study the mechanism of adsorption processes, pseudo-first-order and pseudo-second-order were applied. It was found out that the pseudo-second-order adsorption mechanism is predominant for both virgin and recycled cashmere.

In the end, in Fig. 1c and 1d, the identification of recycled cashmere in blends with virgin ones has been reported using both the techniques. From these calibration curves, it is possible to deduce that up to 5% of recycled cashmere can be detected using both NIR and UV-vis spectroscopy.

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ID 27
FLUORESCENT CARBON NANOPARTICLES MADE FROM INVASIVE PLANT FIBERS

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ABSTRACT
This work presents and discusses the use of dried fibers from the invasive plant *Ricinus communis L.* to synthesize fluorescent carbon nanoparticles (CNPs) as an alternative to the traditional and natural sources of nanomaterials. The synthesis of CNPs was made using the hydrothermal method by varying, in a systematic way, the reaction time and evaluating its impact on the mass and quantum yields, as well as on the photoluminescent properties and surface composition. The CNPs obtained from *Ricinus communis L.* fibers were characterized using UV-Vis and fluorescence spectroscopies, as well as by Fourier-transform infrared spectroscopy (FT-IR), Dynamic light scattering (DLS), and Transmission Electron Microscope (TEM).

INTRODUCTION
Invasive plants are causing a negative impact on the environment, particularly *Ricinus communis* L. (RC), in the Macaronesia archipelagos. By transforming them into functional nanomaterials like carbon nanoparticles, we can contribute to the reduction of the area affected by these invasive species. Carbon nanoparticles (CNPs) are nanomaterials between 1-100 nm[1,2]. These NPs can be photoluminescent, water-soluble, low cytotoxic, and surface functionalized[3,4] having potential applications in biomedical and forensic sciences. CNPs can be prepared by 'bottom-up' methods, such as hydrothermal synthesis, using small carbon-rich molecules or natural products (like as fruits and plants) as starting materials[5]. Therefore, in this work, we will present and discuss the most recent results on the preparation of fluorescent CNPs using dried fibres from *Ricinus communis L.* and the hydrothermal method. The synthesis was made using a systematic approach to study the influence of the reaction time on the CNPs mass and quantum yields and on the remaining photoluminescent properties. After synthesis, the CNPs were subjected to purification and characterized by UV-Vis, fluorescence, IR spectroscopy, DLS and TEM to evaluate the photoluminescent properties and confirm their composition, size, and structure.

RESULTS AND CONCLUSIONS
The results presented in Figure 1A show an increase in the absorbance intensity, in the UV-Vis spectra, with the increment of the reaction time. This observation means that the production of carbon cores based on RC fibers rises with the reaction time. Fluorescence studies (Figure 1B) revealed a red-shift emission accompanied by reductions in photoluminescence intensity, which is typical of certain carbon nanoparticles[6].
Our best results were obtained for 7 hours of reaction time with the DLS size distribution for the formed CNPs, below 100 nm, and a quantum yield of 0.9% vs. Quinine Sulphate. Through TEM we observed CNPs with an average size of 76 nm.

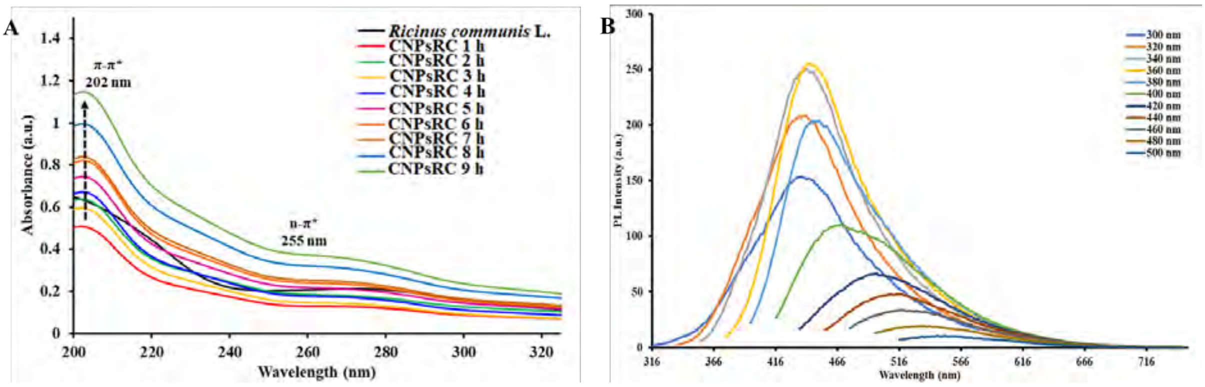

Figure 1. A. UV-Visible spectra of CNPs at different reaction times. B. PL spectra of CNPsRC 7 h.

This study shows that *Ricinus communis L.* fibers can be successfully used as an alternative to the traditional and natural sources of nanomaterials to obtain carbon nanoparticles with controllable fluorescent properties.

ACKNOWLEDGMENTS

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ID 28

EXPERIMENTAL MEASUREMENT OF THE TRANSVERSE YOUNG'S MODULUS OF SINGLE PLANT FIBRES USING MICRO-MECHATRONICS

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ABSTRACT

In order to better understand and model the mechanical behaviour of Plant Fibre Composites (PFCs), it is necessary to accurately characterize the transverse properties of single fibres.

In this work, the transverse compression force/displacement curves were collected for European plant fibres such as flax, hemp and nettle using an innovative and ultra-accurate micro-mechatronic device in controlled hygrothermal conditions. The apparent transverse Young's modulus ranges from 1.5 GPa to 3 GPa. Results showed comparable values to aramid (Kevlar 29).

INTRODUCTION

For synthetic fibres, the transverse properties have been determined so far by transverse compression on single fibres. For plant fibres the only values available in the literature were obtained by indirect method from tests on composite (Baley, 2006). However, those values are uncertain because the rule of mixtures under a usual formulation lacks reliability (Shah, 2016).

Since single fibres are small (a few millimetres in length for a few tens of micrometres in diameter), the force and displacement measured during a transverse compression test are respectively a few milliNewtons and a few micrometres. These metrological specificities require breakthrough and ultra-accurate approaches.

This work proposes to determine the transverse Young's modulus of flax, hemp and nettle fibres from diametrical compression tests using micro-mechatronic systems in controlled hygrothermal conditions. It includes an in-house compliant and high-precision sensor specially developed for the simultaneous measure of axial compression force and the transverse displacement of the fibre in direct contact with it (repeatability in force: 10 µN, in displacement: 30 nm) (Fig.1 (a)) (André, 2022). The proof of concept was already demonstrated in previous works (Placet, 2020). The experimental set-up was further modified to control the parallelism between the two compression platens during the test with an accuracy below 0.1 ° (Govilas, 2022). Recently, a climatic chamber was added to control the relative humidity on a range from 10% to 80%RH with a precision of ± 0.7 % RH.

In the present study, 5 fibres of each type are tested. A progressive repeated loading path is applied on each fibre to study the elastic and inelastic behaviour. The apparent transverse Young's modulus of the fibres is identified with the Jawad's model (Jawad, 1978).

RESULTS AND CONCLUSIONS

This work allows to obtain the transverse force/displacement curve for nettle, hemp and flax fibres (Fig.1 (b)) and to highlight inelastic phenomena during the loading/unloading cycle. For the first time, the transverse Young's modulus of these fibres is identified by direct method; it ranges from 1.5 GPa to 3 GPa (Fig. 1(b)). These values obtained for the plant fibres rival those of Kevlar 29. The influence of relative humidity on the transverse mechanical properties is also quantified and the main results will be presented.

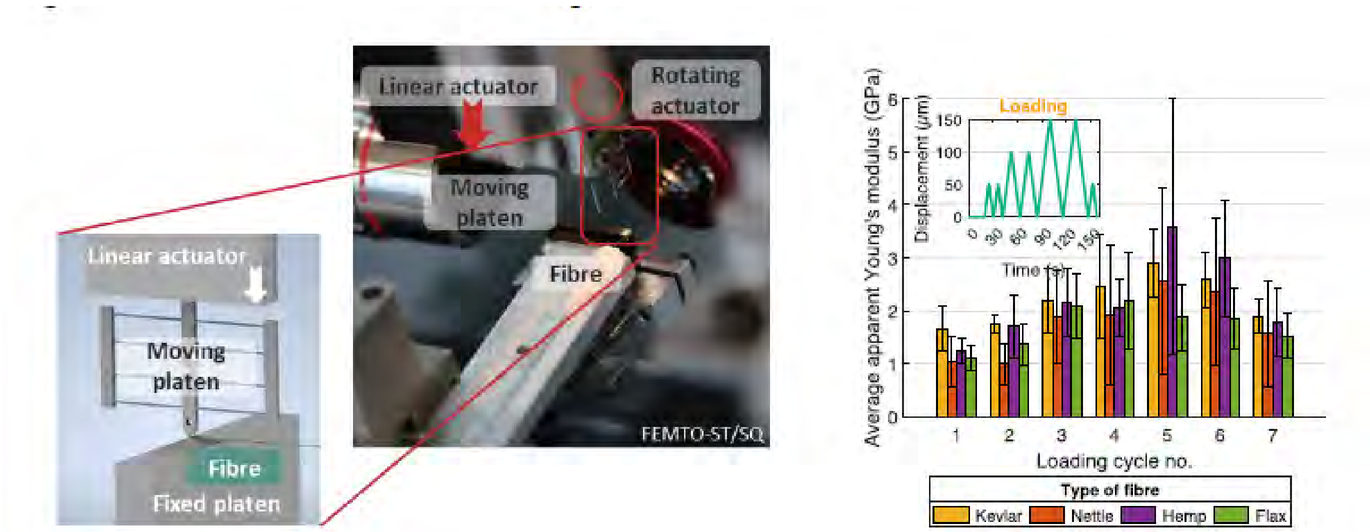

Fig.1 (a) Micro-mechatronic device for diametrical compression with detail of the force/displacement sensor (b) Evolution of the Young's modulus identified at unloading of Kevlar 29, nettle, hemp and flax fibres

ACKNOWLEDGMENTS

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ID 29
HIGH-PERFORMANCE BIOCOMPOSITES - A COMPARISON OF LIGNOCELLULOSE MICROFIBRILS AND WOOD FIBERS

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ABSTRACT
It is challenging to process biocomposites with high reinforcement content. Here, we tackle this problem using a creative in-situ polymerization strategy based on esterifying new polyester oligomers in pre-formed lignocellulosic networks. Microfibrillated lignocellulose and unbleached wood fibers were used as reinforcement. Biocomposites with excellent mechanical properties were created as a result of well-distributed, mechanically undamaged reinforcement. The polymer matrix was revealed to be degradable through hydrolysis. Depending on the type of reinforcement (wood fibers or microfibrillated lignocellulose), the mechanical performance and rate of degradation varied.

INTRODUCTION
Of all plastics globally produced from 1950 to 2015, about 30% of them are in use, about 60% were discarded and 10% are incinerated[1]. Only 1% of them are recycled and reused[1]. Production and incineration of petro-plastics emit 400 million tons of CO_2 annually[2].
Biocomposites based on lignocellulosic materials provide CO_2 sequestration due to plant photosynthesis. However, polymer matrix cellulose biocomposites often are not degradable or their processing is not scalable[3]. Also, the mechanical properties of these biocomposites are often lower than those of commercial petro-plastics. One issue is the processing; for instance, melt-compounding is limited by viscosity (or fiber content) and causes agglomeration and mechanical damage to fibers[4].
In order to create high fiber content biocomposites, in-situ polymerization within a pre-formed fiber network is a practical approach with low energy demand. Degradable polymer matrix synthesis is often carried out through ring-opening polymerization of cyclic monomers[5]. A synthetic challenge is that cellulosic fibers absorb environmental moisture, which initiates polymerization and results in a low molar mass polymer matrix.
In this study, a new double-functional three-arm ε-caprolactone-based oligomer was prepared for impregnation and curing. Oligomers polymerize via esterification to high molar mass, semi-crosslinked polymers within well-dispersed wood fiber or microfibrillated lignocellulose networks. This approach does not require specific conditions or toxic reactants, and the curing is straightforward. This technique allowed us to create degradable, high reinforcement content biocomposites with high mechanical properties. The present approach based on novel aliphatic polyester oligomers and reactive processing shows the potential to replace a fraction of non-degradable plastics and composites with degradable biocomposites.

RESULTS AND CONCLUSIONS

We made a series of biocomposites with different reinforcement contents using microfibrillated cellulose or wood fiber. With the use of this polymerization approach, we were able to create biocomposites with a very high fiber content (>50 wt%) and excellent dispersion, resulting in mechanical properties considerably higher than literature equivalent materials (Fig.1a). We created thermoformable, translucent, and high-performance polycaprolactone-based biocomposites (Fig. 1b). This technique allowed for an accurate assessment of the influence of reinforcement types (fibers and fibrils) on mechanical properties, as reinforcement damage and agglomeration issues were minimized.

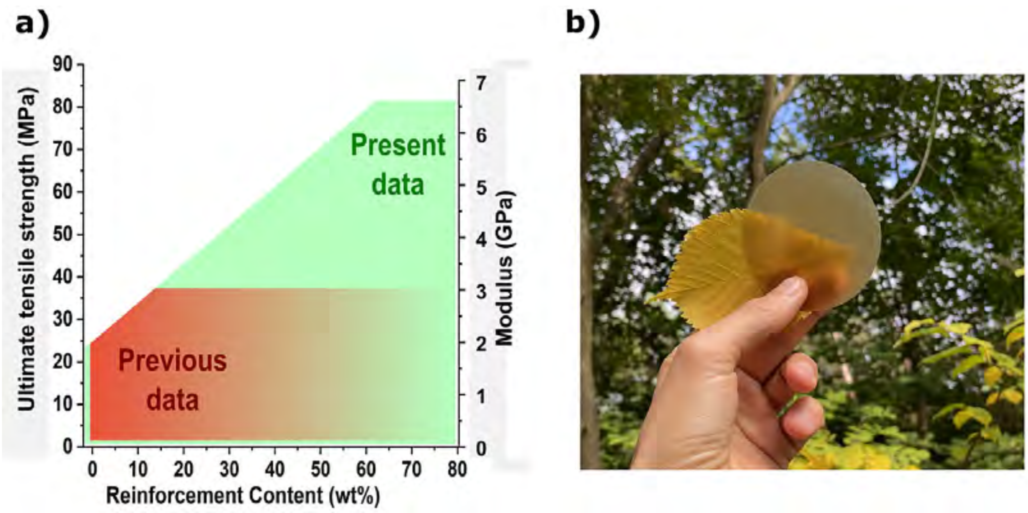

Fig. 1 a) Due to the absence of reinforcement damage and efficient reinforcement dispersion, the high fiber content biocomposites of this study are significantly stronger than literature data. b) An image of a PCL biocomposite containing 25% wood fibers.

It was shown that the polymer matrix could hydrolyze, and the type of reinforcement would affect the rate of degradation. Therefore, depending on the reinforcement, the mechanical properties, ductility, and rate of degradation can be altered. In conclusion, this reactive technique does not damage the reinforcement and enables excellent dispersion, giving rise to biocomposites with much greater mechanical properties than their counterparts. Additionally, this method is scalable and could be done solvent-free.

ACKNOWLEDGMENTS

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ID 30
PERFORMING FLAX COMPOSITES FOR THREE INDUSTRIAL MARKETS: FLOWER PROJECT

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ABSTRACT
This work focuses on the activities carried on by the INTERREG FLOWER research project partners. The partnership spans the entire value-chain from agriculture production of flax fibres to the R&D and commercialisation of end-products to ensure the development of high performance, light weight, biodegradable or recyclable products with reduced environment impacts.

INTRODUCTION
The FLOWER project, for Flax composites, LOW weight, End of Life and Recycling, aimed at developing flax fibre reinforcements produced locally for the composite industry at a lower cost. The outcomes of this project will enable to achieve the aspirations of using sustainable, cost effective and environmentally friendly lightweight composites as a viable alternative for automotive, advertising and sailing sectors. For each sector, one prototype was contemplated: a car headliner, a point of sale (POS) support and a foiling boat.
Eight partners gathered to produce the necessary lightweight preforms and the prototypes: University of South Brittany (leader), INRAE Nantes, University of Cambridge and University of Portsmouth for the academic part and EcoTechnilin, Teillage Vandecandelaere, Howa Tramico and Kairos for the industrial part.
EcoTechnilin manufactured two non-woven preforms: one only made with flax fibres, for the car headliner produced by Howa Tramico and another one made from flax fibres that would be thermocompressed with a biodegradable polymer, PLA, for the POS support designed by Kairos. The Teillage Vandecandelaere produced a woven NCF preform, to be introduced in the foilboat developed by Kairos.
Academic partners on their side were responsible of several types of tests depending on their expertise: ageing, vibration, compostability, biochemical analysis and tensile tests were carried both on the preforms and on the final composites.

RESULTS AND CONCLUSIONS

Very interestingly, one can notice that mechanically speaking, the flax headliner can compete with glass reinforced products, in terms of bending properties, hardness and also adhesion with the foam, as hypotheses based on interfacial properties. The POS support has also proven to be as performing as conventional ones and starts to be commercialized in the sector. Regarding the foiling boat, despite a difficult impregnation, it was a success as a proof of concept and proved that plant fibres can be used in nautical structures.

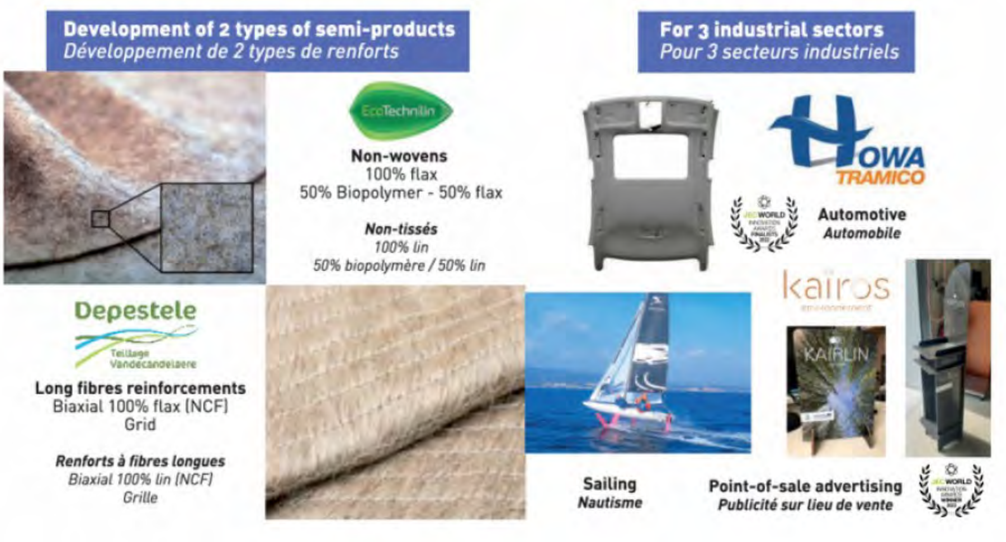

Figure 1. FLOWER industrial developments

Environmentally speaking, the use of flax fibres is consistent. Indeed, Normandy is the world's leading region for the production of flax for technical applications. The use of local resources allows a decrease of transportation and thus a decrease of CO2 emissions. Moreover, the production of the flax fibres needs less energy than other reinforcement and the plant captures CO2 during its growth. During the product development, a Life Cycle Analysis has been carried out to evaluate the environmental impact due to the replacement of glass fibres by flax fibres. The results show a smaller impact on all studied environmental indicators. This LCA confirms the attractive environmental profile of flax.

Following the production of the prototypes, FLOWER partners Howa Tramico and Kairos applied for the JEC Awards, respectively for the car headliner and the POS support. The headliner was finalist in the Automotive & Road Transportation – Surfaces category when the POS support was distinguished in the Design, Furniture and Home category.

The poster will present in detail the industrial development and research work carried out during this project; the latter concerns in particular the understanding of the ageing and performance of flax fibres but also the quality of the composites produced and the development of innovative bio-based composites, associating flax with biodegradable matrices.

ACKNOWLEDGMENTS

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ID 32
DEVELOPMENT OF NEW ALL-CELLULOSE COMPOSITES, THEIR PROTECTION AGAINST ENVIRONMENTAL IMPACTS AND RECYCLING

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ABSTRACT
This work focuses on composites based on the renewable biopolymer cellulose. For an optimal use of the excellent properties of these all-cellulose composites (ACCs) in various fields of application, it is advisable to protect them against environmental influences. By protecting the surface with different coatings, no significant decrease in mechanical properties of the coated ACCs was observed after weathering compared to uncoated and weathered ACCs. With a minimum of additional work to remove the coating from the ACC surface, a successful recycling is feasible similar to the uncoated ACCs.

INTRODUCTION
In recent years, fiber-reinforced plastics (FRPs) have gained importance in the replacing of traditional materials for lightweight construction (Yazdanbakhsh, 2014). In terms of sustainability and complete recycling, the FRPs perform rather poorly because they are mainly made of petroleum-based, polymeric matrices and reinforcement fibers, i.e. glass, carbon or natural fibers. Consequently, the only technically feasible possibility to recycle glass fiber-reinforced plastics waste is the pyrolysis of the polymeric matrix (Yazdanbakhsh, 2014).
Biocomposites have been developed as a result of increasing environmental awareness and to reduce environmental pollution. One type of biocomposite are the so-called all-cellulose composites (ACCs). A special feature of ACCs is that they consist of both a cellulosic reinforcing fibers and cellulosic matrix, thus facilitating a complete recycling of the end-of-life product. The composite material outlined here is such an ACC, made from reinforcement fibers based on regenerated cellulosic filament yarns that are embedded in a cellulosic matrix precursor. The matrix precursor is formed form dissolved cellulose in an ionic liquid, i.e. 1-ethyl-3-methylimidazolium acetate [EMIM][OAc] (Hermanutz, 2019; Hermanutz, 2020).
The ACC surface was coated with three different coatings: an additional cellulose layer, a clear coat and a commercial Roxil wood protection cream (Safeguard Europe). The coated ACCs were weathered by defined parameters. The artificial weathering was executed in a light chamber with xenon lamps (λ = 310 nm) of the device XENOTEST BETA LM (Atlas GmbH) based on DIN EN ISO 4892-2. The weathering time was 595 h at a relative air humidity of 50% and a constant sample temperature of 65 °C. These very extreme weathering conditions were chosen which refer to global radiation in Germany for an aging period of a half year. After the weathering the mechanical properties were measured. For the recycling the coatings were washed by a Soxhlet extraction in acetone. The washed, uncoated ACCs were milled, completely

dissolved in ionic liquid and could thus be used again as a matrix precursor. The characterization of all ACCs was done by analysing SEM images and measuring mechanical properties including tensile and Charpy impact strength.

RESULTS AND CONCLUSIONS

The tensile strength and Young's modulus of both uncoated and coated ACCs are shown in Fig. 1. The results show that coating the ACC surface did not result in a significant decrease in mechanical properties. Furthermore, the results show that the applied coatings protect the ACC surface optimally for environmental influences.

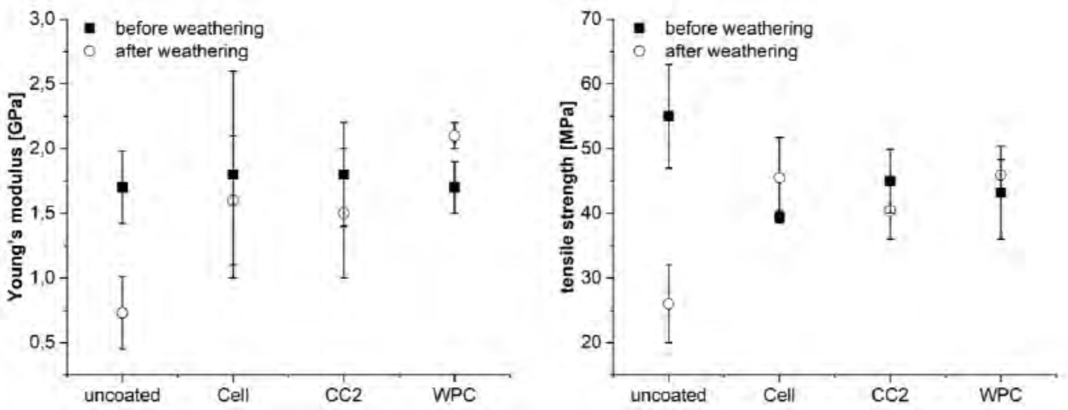

Fig.1 Mechanical properties of (un)coated ACCs; CC: clear coat, WPC: wood protection cream.

The study shows that these ACCs have excellent properties and, thanks to the coatings, they are optimally protected against environmental influences. Thus, they can find application in the field of automotive or light construction.

ACKNOWLEDGMENTS

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ID 33
TRACKING THE CHANGES INTO MECHANICAL PROPERTIES AND ULTRASTRUCTURE OF FLAX CELL WALLS DURING A DYNAMIC HEATING TREATMENT

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ABSTRACT
The impact of temperature on plant fibres is key point when processing plant fibre composite with thermoplastic resin. Structural and biochemical evolution of plant cell walls with temperature may affect their final mechanical properties. These evolutions have been studied in-situ on flax fibres during a dynamic heating treatment, representative of conventional polymer processing temperatures. Originals results were observed regarding the reversibility of the indentation modulus, hardness and cellulose crystalline structure.

INTRODUCTION
Environmental concerns currently raise interest in the use of thermoplastic matrix to replace thermoset material in order to open up recycling and composting as end-of-life routes of plant fibre composites [1], [2]. However, using polymers as matrix involves long cycle processing in temperature, which may negatively affect the structure and the mechanical properties of plant fibres due to their particular composition and ultrastructure [1]. Among plant fibres, flax fibres are elementary cells made up of a primary cell wall and a secondary cell wall divided into three main layers S1, S2-G and S2-Gn. The S2-G layer, responsible of the longitudinal properties of the fibre, is mainly composed of cellulose Iβ but includes a few non-cellulosic polysaccharides and proteins (NCPs,i.e. hemicellulose, pectin and lignin) which are very sensitive to temperature [1]. The cellulose Iβ also present in tension wood is well documented, especially the impact of temperature on structural changes of cellulose [3],[4],[5]. Regarding cellulose Iβ from flax fibres, no data are available.

Therefore, In this present study, the impact of cellulose structural changes and NCPs degradation on mechanical properties of elementary flax fibre was investigated from 25 to 210°C. The evolution of local mechanical properties by nanoindentation and cellulose structure through X-Ray diffraction was monitored during a dynamic heating. The originality of this work is to assess in-situ the mechanical properties and ultrastructural of flax fibre during heating treatment. In addition, biochemical composition of fibre was addressed before and after thermal treatment. Deeper insights are given about the correlation between NCPs thermal degradation, ultrastructure evolution and local mechanical behaviour of flax cell walls.

RESULTS AND CONCLUSIONS

Based on thermogravimetric analysis, an important part of organic compounds is degraded when temperature exceeds 190°C. The correlation with biochemical analysis highlighted that the non-cellulosic monosaccharides content decreases by 16%, 45% and 59% from respectively 190°C to 210°C and 230°C.

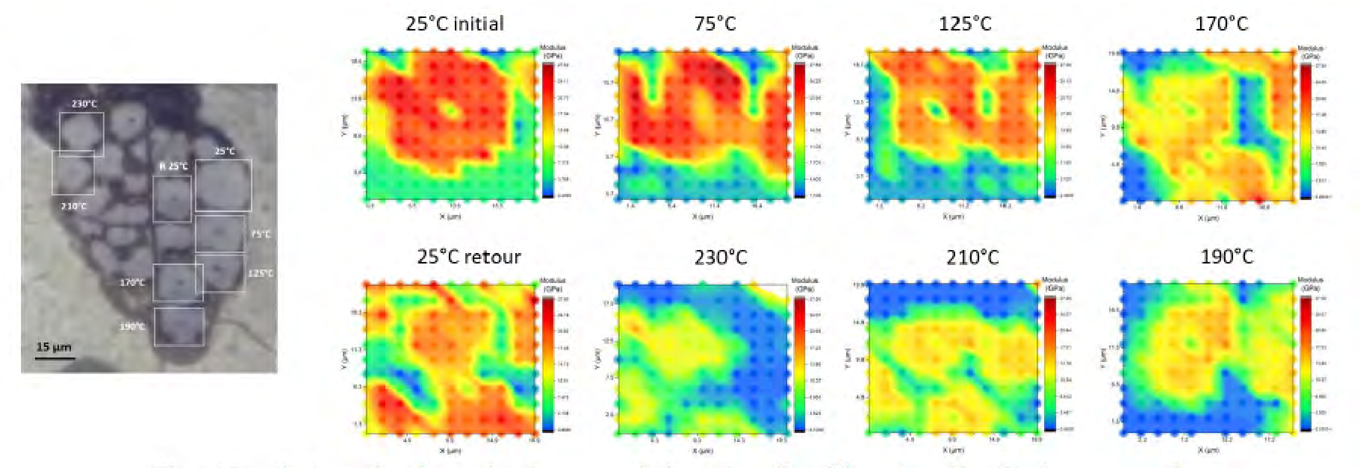

Fig.1 Nanoindentation investigations, carried out in a flax fibres bundle. Each cartography represents the evolution of the indentation modulus, according to the x-y position, for a considered temperature.

A drastic decrease of mechanical properties has been observed between 170°C and 230°C (Figure1). Surprisingly enough, these ones raised from their initial values when temperature get back to 25°C. This partially reversible phenomenon was also noticed regarding the XRD pattern of cellulose crystals during the same heating treatment. Supported by a biochemical analysis, a discussion will be addressed about correlation between the flax cell walls mechanical behavior and ultrastructure evolution during a heating stage.

ACKNOWLEDGMENTS

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ID 34
PLA BASED BIOCOMPOSITES REINFORCED WITH WATER HYACINTH PARTICLES

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ABSTRACT
This work deals with the investigation of the performances of PLA based biocomposites reinforced with water hyacinth particles. In particular, the influence of the particles content and size on the morphology as well as on the mechanical properties of biocomposites was evaluated. In order to improve the particle/matrix adhesion, an eco-friendly treatment based on the use of sodium bicarbonate (Fiore, 2016) was also carried out. Tensile, flexural, impact and dynamical-mechanical tests were carried out to investigate all the parameters.

INTRODUCTION
The rise in environmental consciousness in recent decades has been a driving force behind the use of eco-friendly and more sustainable composite materials, i.e., biocomposites (Faruk, 2012). Indeed, several researchers focused their attention on the use of biobased and/or biodegradable polymers to fully or partially replace conventional plastics derived from oil (La Mantia, 2021) as well as of natural fibers or particles as alternative reinforcement of synthetic fibres. In this context, several plants have been investigated as possible source for natural reinforcement in the last years. Among them, water hyacinth (Eichhornia crassipes) seems promising due to its high strength and availability at low cost (Nugroho, 2022).
In this works, water hyacinth particles and PLA were used as reinforcement and matrix to manufacture biocomposites via extrusion and compression moulding. The influence of particle content (i.e., 10 % and 20 wt.%) and size (i.e., 0-150 μm; 150-300 μm, 300-450 μm) was studied to understand how these parameters affect composite performances. Furthermore, in order to strengthen the interface between particles and matrix, water hyacinth particles were soaked in a sodium bicarbonate solution (10 wt.%) for 5 days at 25°C. Each produced biocomposite was fully characterized through tensile, flexural, Charpy impact and DMTA (dynamical-mechanical-thermal analysis) tests.

RESULTS AND CONCLUSIONS
The mechanical characterization has shown that the particles addition increases the elastic modulus but, at the same time, leads to an overall decrement of the maximum tensile and flexural strength in comparison to the neat matrix. Concerning the tensile and flexural strength, both particles content and size affect the results: i.e., the maximum size and the highest content resulted in the largest strength decreasing (Fig.1a). On the other hand, all the manufactured biocomposites showed higher modulus in comparison to the neat matrix, regardless of both particle size and content (Fig.1b). After the sodium bicarbonate treatment, biocomposites' performances get better due to the strongest particle-matrix interface.

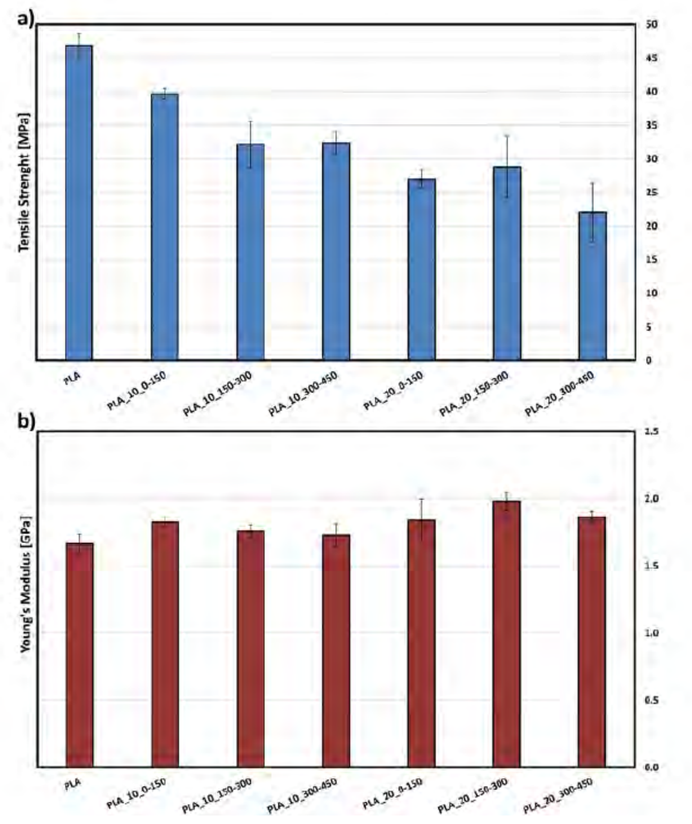

Fig.1: a) Tensile strength and b) elastic modulus of the investigated biocomposites.

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ID 35
DEVELOPMENT AND DEMONSTRATORS OF DURABLE BIOBASED COMPOSITES FOR A MARINE ENVIRONMENT.

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ABSTRACT
The ERDF/InterReg 2 Seas Mers Zeeën SeaBioComp project (2019-2023) sought to develop and deliver demonstrators using innovative bio-based thermoplastic composite materials with the following characteristics:
• a tailored durability according to the specific application (2 to >20 years) for that demonstrator in a marine environment as measured by newly developed analytical methods
• at least equivalent mechanical properties compared to conventional oil-based composites
• reduced CO2 emission (30%)
• reduced ecotoxic impact in the marine environment due to microplastics as measured by newly developed analytical methods
• demonstrated recycling potential of the used materials in the demonstrator
These characteristics should decrease the overall ecologic impact by 50% compared to conventional oil-based counterparts through the entire value chain from production to waste treatment. These proposed bio-composite materials and the developed analytical protocols for long-term durability and ecotoxicity should lead to a shift in mind-set along the value chain realising that bio-based composites may offer a realistic alternative to their oil-based counterparts.

INTRODUCTION
With the ever-increasing world production and consumption of plastic materials and composites comes a growing concern with both the waste treatment and greenhouse gas emissions during production of these predominantly oil-based materials. The long-term ecological impact of plastic litter and microplastics in the marine environment is a growing issue that has gained considerable momentum in public perception and global media. Bio-based polymers, or polymers from renewable resources, can prove a viable substitute to conventional oil-based polymers for many applications with significantly reduced greenhouse gas emissions and potential biodegradability.
The current trend in the field of polymer composites, or plastic materials that are reinforced with e.g. glass or carbon fibre, is to find bio-composite alternatives for both the matrix polymer and the fibre reinforcement. These materials will cater to the current demands in several fields including the maritime industry for substitute materials to replace conventional petrol-based composites in e.g. fish and seaweed farming, energy harvesting, boats, pontoons, anchoring and buoy elements. The bio-composites will not only reduce the depletion of fossil-based resources but should also limit the emission of greenhouse gases and ecotoxic impact of microplastics, while allowing recycling through the use of thermoplastic bio-polymers. At the same time, these materials should meet the highest technical performance standards and

withstand harsh sea conditions, including mechanical forces, aggressive environment, and intense UV light.

WORK PACKAGES

Work Package (WP) 1 sought to realise high-quality bio-composite demonstrators with reduced environmental impact. The three major activities were (i) a self-reinforced biopolymer system composed of low melting temperature (Tm) biopolymer as matrix and high Tm biopolymer as reinforcement, (ii) a hybrid composite system of flax fibres and biopolymer, and (iii) in situ polymerisation (ISP) during monomer infusion under flexible tooling (MIFT) (Qin et al, 2020), to produce bio-based thermoplastic matrix composites for the marine environment.

WP2 sought to optimise industrial additive manufacturing to process biopolymer formulations into biocomposites with large geometrical dimensions.

WP3 sought to develop testing protocols for durability, degradability and ecotoxic impact of bio-based polymers and composites.

The project partners were CenTexBel (Belgium ~ lead), ARMINES: Association for Research and Development of Industrial Processing Methods (France), CETI: Centre Européen des Textiles Innovants (France), Euramaterials (France), IFREMER (France), Marine South East Limited (United Kingdom), Matikem (France), Poly Products (The Netherlands), REWIN (The Netherlands), University of Plymouth (United Kingdom), University of Portsmouth (United Kingdom) and VLIZ - Flanders Marine Institute (Belgium).

CONCLUSION

The project website is at http://www.seabiocomp.eu/. For more information join the SeaBioComp Interest Group at http://www.seabiocomp.eu/interest_group/ to receive regular updates, details of events and project activities.

ACKNOWLEDGMENTS

This work was conducted within the SeaBioComp project, which has received funding from Interreg 2 Seas Mers Zeeën program 2014-2020 co-funded by the European Regional Development Fund under subsidy contract No. 2S06-006.

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ID 40
FROM 2013 TO 2023 : EVOLUTION OF BIOLOGICALLY INSPIRED HYGROMORPH BIOCOMPOSITES

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ABSTRACT
The purpose of the presentation will be to gather the work dedicated to the development of hygromorph biocomposites (HBC) from 2013 to 2023 within the bionics group (Univ of South Brittany France- https://bionics-group.com/). The basic concept is proposed while the chronology and works are presented. Finally, opportunities about future development are discussed.

INTRODUCTION
In 2013, the baseline of HBC has been proposed. HBC are novel generation of architectured smart composites that exhibit both moisture sensing and actuation functions thanks to the large moisture sensitiveness of natural fibres (sorption and hygroexpansion). Using moisture sensitiveness as an opportunity instead of a major drawback was the opposite way of the scientific community. The latter wished to chemically/ physically treat the natural fibres so that their hydrophilicity could be reduced. In the meanwhile, we have applied biomimicry methodology and have inspired from biological structures that exhibit shape-changing functionality triggered by moisture variation (pine cone scale, wheat awn…). The resulting HBC architectured as bilayer/asymmetric laminate has been manually produced by thermocompression. This architecture allows a differential expansion between layers that drives the amplified moisture shape-change displacement with various typologies: curvature, twist, expansion, fold... The work has been published in 2015 [1] and presented in ICNF 2015. Subsequent conceptualization of HBC has been proposed later [2]. In 2016, their performance in term of force generation has been studied and highlighted their original feature by allowing shape changing while keeping relevant stiffness compared to other moisture sensitive smart materials (e.g. hydrogels) [3]. HBC can be classified as semi-structural actuator.
In the same year, 3D and 4D printing of on-the-shelf short fibre biocomposites has been introduced where HBC response and their mesostructure can been tailored. However, Short fibre biocomposite suffer from low hydromechanical performance [4].
In 2017, several experimental parametric studies evidenced the role of the component selection. Fibre type (flax, jute, kenaf…), their microstructure (Cellulose Microfibril Angle) and the biochemical composition influence the expansion of biocomposite and then the shape-changing ability of HBC (reactivity = speed of actuation and responsiveness = amplitude)[5][6]. Recently, matrix stiffness has been evidenced to control the hygroexpansion of natural fibre. A stiff matrix restrains the expansion of natural fibre while softer polymer allows their larger expansion and thus larger actuation [7]. Fibre/matrix interface bond strength is the third key to control the actuation efficiency. Thus, a lower moisture content generates higher actuation owing to better hygroscopic internal stresses transmission across the component [6].
In parallel, amplified shape-changing ability of these 2D planar thermocompressed smart materials has then been investigated by applying Origami and Kirigami principles. This has led to smart hygromorph

macrostructures depending on folding and cutting patterns [5]. Programmation of HBC actuation through a careful control of thermal and hygroscopic internal stress state has been achieved and opened the design space towards novel generation of smart metasurface [8][9].

In 2019, the bending actuation of parallelipedic slender bilayer has been predicted by both using analytical based on modified Timoshenko equations and Finite Element that take into account the differential expansion between each layer, the differential longitudinal stiffness and thickness [10]. The same year, high performance 3D printed biocomposite were produced based on continuous flax yarn and originally modified 3D printer [11]. This reinforced the possibility to apply biomimicry thanks to more complex mesostructure with a sharp control of material distribution. Then, between 2020 and 2022, the first review paper dedicated 3D and 4D printing of natural fibre biocomposites has been published [12] while the relationship between the design parameters, the slicing parameters (inherent to additive manufacturing), the mesostructure and to targeted morphing shape has been investigated [13][7]. Layer height, interfilament distance and filament orientation allow to build local mesostructure that yield to sequential actuation. Geometrical limitation of 3D printing of continuous natural fibre has been studied to identify the design envelop of HBC and lattice structures [14][15]. Last work in 2022-2023 has focused on the design space applying tools dedicated to architecture materials, i.e. evolutionary algorithm and voxel based finite elements [16][17]

ACKNOWLEDGMENT

The author wishes to thanks all the collaborators and PhD students for contributing to the development of HBC since the beginning.

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ID 43
CONTRIBUTION TO THE STUDY OF TENSILE BEHAVIOUR OF FLAX FIBRE BUNDLES

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ABSTRACT
The aim of this work is to develop knowledge on flax fibre bundle mechanical properties in order to set up simple laboratory means for the qualification of the strength. Hence, tensile tests are carried out on flax fibre bundles but a lot of parameters such as the evolution of the cross-section area (CSA) along the length of the bundle, the gauge length or the method for monitoring the strain and the damage progress have a lot of influence in the test results and need to be well defined.

INTRODUCTION
Tensile test conditions on flax fibre bundles are strongly influenced by several parameters including the evaluation of the cross-section area (CSA). It is usually determined by measuring diameters along the bundle length and by making the assumptions that the fibres have a circular cross-section and a constant diameter along the fibre length. However, flax fibers are very irregular, and this method gives an overestimation of the CSA and therefore and underestimation of the mechanical properties. (*Garat et al. 2018*)
The gauge length also influences the results, by decreasing the gauge lengths the number of critical defects is decreased and elementary fibres will be solicited. For higher gauge length, fracture will occur in the middle lamellae (weaker than fibres). (*Bourmaud et al. 2020*)
Another key point to consider for bundles characterization is the strain measurement method. Indeed, it is often measured indirectly via the grip displacement. However, the value is influenced by the system compliance and by the fiber slippage during the test. Therefore, the obtained value must be corrected but the influence of those two phenomena is not easy to see. An alternative could be to use a direct method of measurement as explained by *Depuydt et al. 2017*.

RESULTS AND CONCLUSIONS
Tensile tests were carried out on two batches of flax fiber bundles and the tensile strengths are compared with results from the literature (*Fig. 1*).
The tensile strengths are lower with the two batches that were tested but the trend is the same than literature values. It shouldn't be the case since the mechanical tests on the elementary fibres of those two batches revealed that they have a good quality. This highlights the fact that these experimental points are not sufficient to understand what is going on (lack of information on the tested samples). It is therefore a phenomenon that needs to be investigated further.

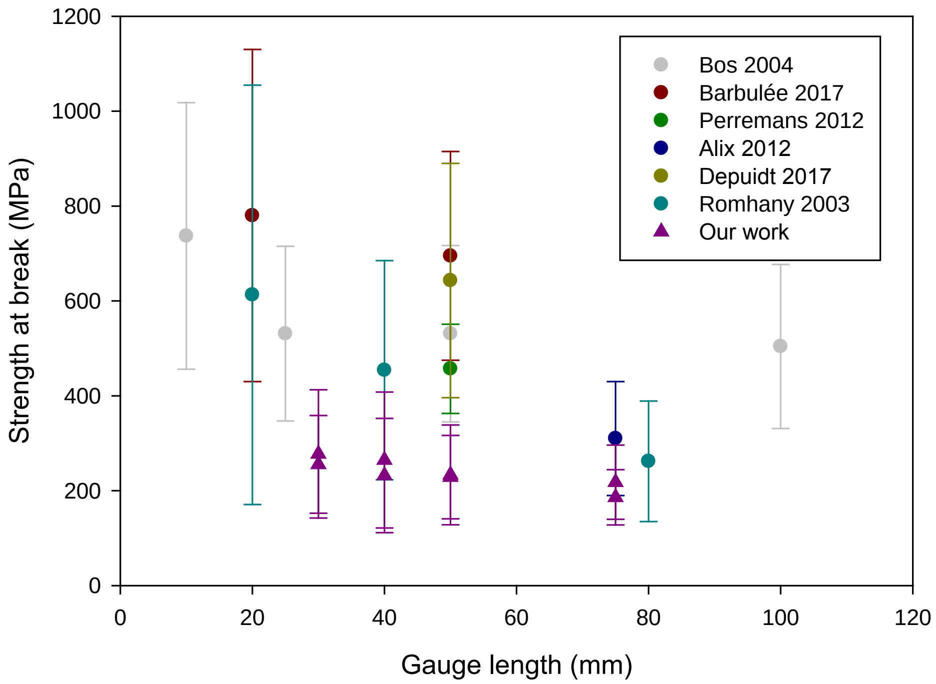

Fig. 1 : Tensile strength of flax bundles as function of gauge length

In order to obtain information on the cohesion between the fibres and the middle lamellae (assessment of the retting quality), acoustic emission devices will be used during the tensile tests. The amplitude ranges will allow the identification of the failure mode that has occurred. (*Romhány et al. 2003*). In situ tensile testing in the SEM and observations under optical microscope will also be carried out to assess the retting quality.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the funding by the Labcom Newflax and the ANR (Agence Nationale de la Recherche).

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ID 47
DEVELOPMENT OF A HIGH DUCTILITY LIME MORTAR USING NATURAL FIBRES

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ABSTRACT
This study examines the use of flax fibres for the development of a high-ductility lime mortar for masonry retrofitting applications. Fibres were incorporated in a matrix formulated using Natural Hydraulic Lime, silica fume, siliceous aggregates and a polymeric admixture. Despite some reduction in compressive strength, fibre addition produced a crack bridging effect which increased flexural ductility and allowed the material to reach bending stresses higher than the first-crack strength.

INTRODUCTION
The aim of this study was to develop a high-ductility cement-free lime mortar that would be compatible with historic masonry substrates and would exhibit mechanical properties suitable for the construction of structural coatings, including textile reinforced overlays. Chopped flax fibres treated with a purposely developed hydrophobic coating were considered. The fibers had a tensile strength ≥ 250 MPa and a nominal length of 16 mm. Natural Hydraulic Lime of class NHL5 was used as the primary binder in mortar synthesis. Silica fume was added at 30% lime replacement ratio to (i) increase strength (Grist et al., 2013), (ii) reduce alkalinity (García Calvo et al., 2010) to prevent fibre degradation and (iii) improve the matrix-to-fibre bond (Wu et al., 2016). Mortar aggregate comprised of siliceous sand with a maximum grain size of 1 mm. The binder-to-aggregate ratio was kept at 1:1.5 by wt. A latex admixture was added at a dosage of 25% by wt. of the binder to reduce water demand and achieve adequate mechanical strength, while being able to attain a target consistency of 175 ± 5 mm. Fibre content was set to 1% by wt. of solid constituents, which corresponds to ~2% by vol. During production the amount of mixing water was adjusted so that the target consistency could be achieved. The mechanical properties of the reference (unreinforced) and fibre-reinforced mortar compositions were comparatively assessed. Elastic modulus was determined via uniaxial compression tests on cylindrical and prismatic specimens. Three-point bending tests under displacement control were conducted to examine flexural behaviour. Compressive strength was determined by load testing half-prisms obtained after the flexural failure of specimens previously subjected to three-point bending. All tests were performed at 28 days.

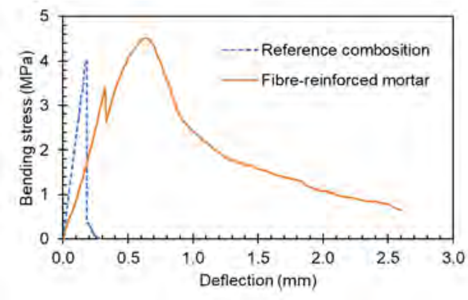
RESULTS AND CONCLUSIONS
The results of the laboratory tests are summarized in Table 1. Fig. 1 compares the flexural behaviour of the reference and fibre-reinforced mortars. It also shows the crack bridging effect imparted by dispersed fibres. Some reduction in the mortar's compressive strength was noted with fibre addition. Nevertheless, the strength achieved is adequate for the intended applications, since structural renders are typically classified as CS IV as per EN 998-1 (f_c > 6 MPa). Both the reference and fibre-reinforced mortars were found have elastic moduli near 8 GPa. In contrast to the reference composition, the fibre-reinforced mortar did not suffer brittle failure under flexure after the occurrence of cracking. Instead, fibre bridging allowed the material to sustain useful load that exceeded the first-crack strength. Notably, the average post-crack

strength of the fibre reinforced mortar is ~40% higher than the bending strength of the reference composition. Although the fibre-reinforced mortar did not exhibit multi-cracking behavior, it can be classified as a deflection hardening composite, in the sense that it has the ability to carry increasing load after the first crack forms ($f_{b,u} > f_{b,cr}$).

Table 1 Average properties (CoV) assessed for the reference and fibre-reinforced mortar compositions.

Property	Reference composition		Fibre-reinforced mortar	
Elastic modulus – E (GPa)	8.1	(9.0%)	7.9	(8.9%)
Compressive strength – f_c (MPa)	15.2	(9.8%)	9.0	(6.0%)
Bending stress at 1st crack – $f_{b,cr}$ (MPa)	4.02	(9.1%)	3.11	(16%)
Peak bending stress – $f_{b,u}$ (MPa)	4.02	(9.1%)	4.39	(14%)

(a)



(b)

Fig.1 (a) Bending stress (calculated with the classical elastic beam theory) vs deflection curves obtained from 3-point bending tests. (b) Bridging of flexural crack by flax fibers.

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ID 48
NATURAL FIBRE BASED SUSTAINABLE FLEXIBLE COMPOSITE: A POTENTIAL ALTERNATIVE OF SYNTHETIC LEATHER

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ABSTRACT
Current Research context elaborates the engineering methods adopted for development of sustainable flexible composite as leather alternative and point by point discussion on scientific difference of the developed flexible composite with marketed synthetic leather and natural leather material. Important Physical properties of the developed product have been examined and mentioned in the paper. Moreover, context was also enlightening with the product developed from the flexible composite material

INTRODUCTION
Natural leather production and processing involved a larger quantity of toxic synthetic chemicals like sulphuric acid, sodium hydroxide, formaldehyde, heavy metal salt, chrome and many more. These chemicals have generated harmful effluent and polluted surrounding environment (CN104172653A, US7347011B2, http://www.leather-dictionary.com) Therefore, development of artificial leather is getting attention for the industries and researchers. Most of the artificial leathers composed of one natural or synthetic fibre (mostly polyester) based base fabric, coated with synthetic polyurethane (PU) or polyvinyl chloride (PVC) or other elastomeric synthetic polymer having long chain linear molecule arrangement. Apart from popular PVC and PU, some other chemicals like butyl rubber (isobutene isoprene copolymers), hypalon (chlorosulfonated polyethylene), neoprene (polychloroprene), nitrile rubber (acrylonitrile butadiene copolymer), styrene butadiene rubber, poly tetra fluoro ethylene also have been used for coating purposes. However, most of the chemicals are petroleum based and synthesized from toxic chemical combinations [Paiva,2019; Toda, 2006; Correia2017; Kohan 2019].

RESULTS AND CONCLUSION
In this regard, ICAR-NINFET taken a step forward for making leather like flexible composite. In the laboratory of ICAR-NINFET, we have developed leather like flexible composite from natural fibre and non-harmful plant latex-based formulations. Developed product is breathable, flexible, resilient, water repellent and biodegradable in nature. Base layer of the flexible composite is made by using natural fibre based non-woven fabric. Coated top layer was developed by using suitable chemical formulation of latex sulphur dispersion, accelerator, activator, stabilizer, protein powder, antioxidant, anti-ozone component. Developed natural fibre made non-woven sheet was coated with the chemical formulation, dried and vulcanised at higher temperature. Top layer of the composite was ornamented by using pigment and other auxiliaries. Developed flexible composite has shown 20% higher permeability and zero shrinkage as compared to the natural leather of same thickness and weight, having 3-4% shrinkage in both directions [9]. It is one of

clear technical advantage of our product as far as the comfort and dimensional stability properties of the product are concerned. Tensile strength of the developed product was 50% less whereas tear strength is almost equal as compared to the natural leather, having same weight and thickness. Abrasion resistance, one of the important parameters (for use as bag, footwear, belt, jacket, upholsteries etc.) of the developed product was in the range of 2-4% after 25000 cycles of abrasion [9-10]. Developed products really have a very good potential to be used as foot wear material, jacket, hand bag, wallet, pencil bag kind of materials.

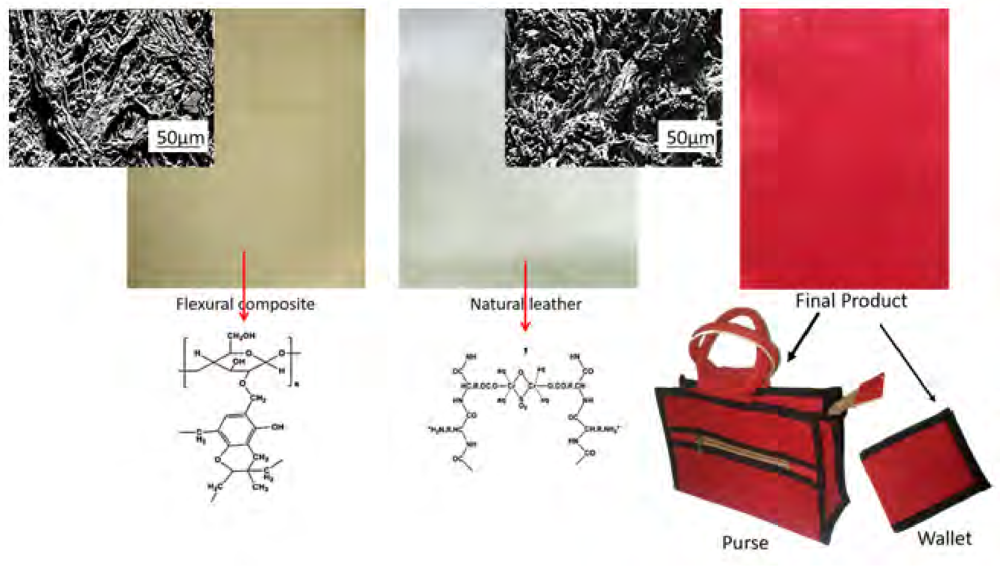

Fig 1. Developed Flexible composite and different product from it [9-10]

Table 1. Comparison of the properties of the flexible composite with natural leather and synthetic leather [9]

Name of the properties	RE (RE-W)*	RE-rub*	Developed Product	Natural leather	Synthetic leather
Thickness (mm)	2.11 (2.32)	1.93	1.87 (after pressing)	1.84	0.97
GSM (g/cm^2)	220 (230)	650	688	720	550
Absorbency (1 mL water)	2sec (5-6sec)	25min	45min	120min	No absorbancy after 24h
Air Permeability (cc/sec/cm2)	44.15 (40.12)	15.12	5.4	0.061	0.04
Abrasion loss (%) after 25000cycles	39 (37)	12	3-4	1-2	1-2
Swelling % (afrter 24h) in weight (wt) and thickness (T)	350 (385) (wt)	79 (wt) 6.5 (T)	103.14 (wt) 3.10 (T)	87.71 (wt) 0.02 (T)	39.73 (wt) 1.28 (T)
Tensile strength (N/ mm2)	1.02 (1.32)	4.10	6.52	13.02	4.12
Elongation (%)	42.11(35.45)	13	25.45	70%	110%
Tear strength (N)	36.45 (40.23)	274.21	235	276	284

RE- Ramie
RE-W- Ramie- wool blend

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ID 50
COMPOSITE NONWOVEN FABRICS MADE OF WASTE MATERIALS

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ABSTRACT
The aim of research was development of a method of managing various types of waste, desirable from the point of view of economy and social effects, and development of a new group of fibrous composite structures based on waste materials.
The scope of research includes the manufacture of composite materials and testing the effect of the addition of waste raw material on their structure and properties.
Achieving the aim of the work was possible due to realization of experimental part including the following tasks:
• Preparation of waste raw materials for processing
• Characteristics of raw materials intended for the nonwovens manufacture
• Optimization of the process of manufacturing composite nonwovens by dry needling
• Characteristics of composite nonwovens produced
• Testing the functional properties of produced composite nonwovens (Agrotextiles, Geotextiles).

INTRODUCTION
Nowadays, environmental problems are of particular importance. Legislative policy forces the industry to search for and use renewable resources that will be able to replace conventional petroleum products (INDA/EDANA, 2005). However, despite the growing demand for environmentally friendly products, there are concerns about the deficiency and availability of natural resources. At the same time, growth population, progressive urbanization and higher living standards generates more and more waste. These wastes also includes materials of biological origin which could be further used. Therefore, representatives of the world of science and industry focused recently on finding new ways to use bio-waste to develop renewable and sustainable products with added value (Das D, Pourdeyhimi B.,2014).
Taking into consideration the most abundant, unique, and inexpensive waste streams that can be used to make value-added products, the poultry industry cannot be missed. Poultry industry wastes include: feathers, internal organs, blood, bones, skins and meat remains. Of these by-products, poultry feathers have the greatest potential for use (Reddy N., 2015).
Another type of post-production wastes, which create a huge problem, are wastes from the textile industry, such as cotton or wool dust, which is generated at all stages of production. These types of post-production wastes are of natural origin, and their fibrous form is suitable to be used in production of composite biodegradable nonwovens (Batra S. K., Pourdeyhimi B., 2012).
There are also post-production wastes from which silicate and aramid fibers (KEVLAR) can be obtained and used for the production of non-biodegradable composite nonwovens for other applications.
The aim of this work is the study of the structure and properties of needled composite nonwovens produced from waste raw materials, which, depending on the type and form of the raw materials used, will be characterized by different functional and strength properties, and therefore, different applications:
• agrotextiles for soil fertilization, protection of plants, vegetables and fruits

• geotextiles to protect embankments and water reservoirs, in the production of roads, drainage and insulation nonwovens

The main premise was the manufacturing of a composite nonwoven fabric with a 3-component structure by dry needling. The base fibers were sheep wool, and the binder of the wool fibers was bi-component thermoplastic fibers. The third component was a waste additive such as:

cotton fibers (JEANS)

newspaper waste paper

poultry feathers

aramid fibers (KEVLAR)

silicate fibers

RESULTS AND CONCLUSIONS

Composite nonwovens, produced by dry needling, ideally suited as a material that absorbs oily substances. Promising results were obtained after carrying out the tests of weight oil absorption..

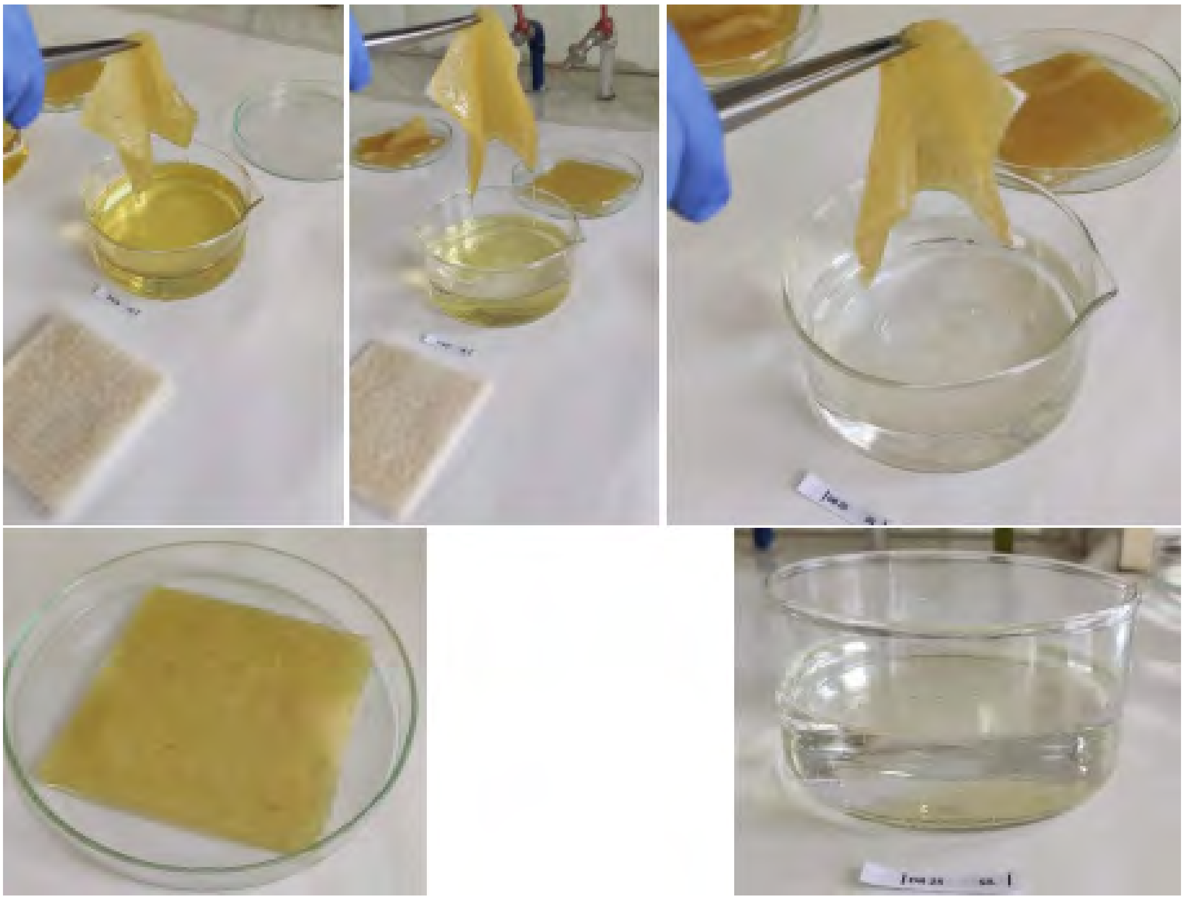

Fig 1. Photodocumentation of oil absorption by weight tests.

Each sample showed more than 20-fold water absorption, i.e. by absorbing oil, its weight increased more than 20-fold. The best result was obtained for composite nonwoven fabrics with the addition of aramid fibers, achieving over 30-fold oil absorption by weight.

Individual waste materials were also subjected to thermal analysis. It was noticed that the smallest weight loss occurred for silicate fibers. At temperature of 900°C, 88% of the raw material remained. These fibers are resistant to high temperatures.

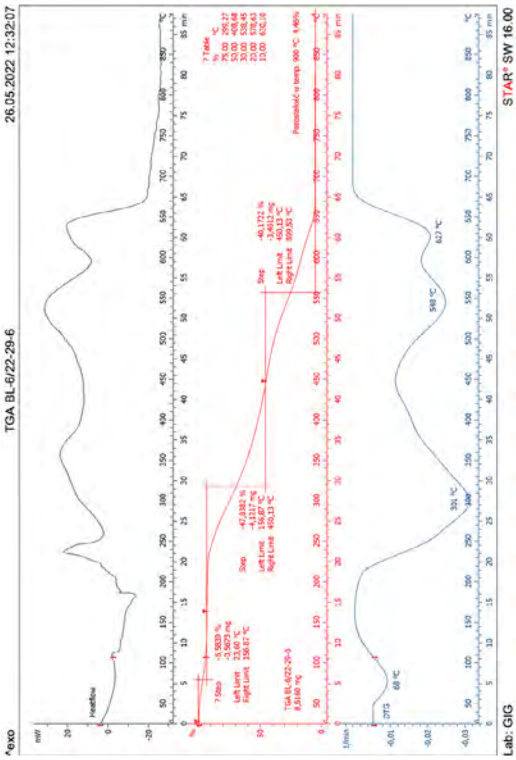

Fig 2. Thermal analysis diagram for a composite nonwoven fabric with the addition of silicate fibers.

The addition of silicate fibers to the composite nonwoven fabric also positively affect the results. This nonwoven recorded the lowest weight loss.

ACKNOWLEDGMENTS

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ID 52
4D PRINTING OF THERMO-HYGRO-SENSITIVE BIOCOMPOSITES: TOWARD MULTI-RESPONSIVE PROGRAMMABLE MATERIALS

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ABSTRACT
This work aims to contribute to the development of new generation of bioinspired smart composites materials, i.e. Thermo-Hygromorph BioComposites (THBC), based on the moisture and temperature sensitiveness of natural fibres. They are able to both support a semi-structural load and give a targeted morphing response to daily humidity and temperature cycle. The development of these smart biocomposites is based on an original approach combining biomimicry and 4D printing. THBC are composed of continuous flax fibres reinforced biocomposite. They are manufactured and mechanically tested over a wide range of relative humidity and temperature conditioning to better understand the design framework of THBC.

INTRODUCTION
Natural fibres are strongly hydrophilic and water sorption leads to swelling of the fibres and reduction of mechanical performances. This behavior can be seen as a weakness for structural applications but it can also be seen as an opportunity for the development of hygro-sensitive biocomposites able to generate actuation for applications such as solar tracking [1]. Based on a biomimetic approach, 4D printed continuous flax fibres (cFF) hygromorph biocomposites have been developed [2]. Their bilayer mesostructure is inspired from hygromorph biological actuators for seed dispersion such as pinecone [3]. The hygroscopic expansion coefficient mismatch between both layers results into bending of the bilayer when the material absorbs moisture. The curvature of the bilayer is predictable thanks to the modified Timoshenko equations and depends on mechanical properties, hygroscopic expansion, and thickness of both layers [3].
In the case of outdoor applications, daily humidity variations are coupled with temperature variations that will influence the sorption mechanism, elastic properties and thus actuation of hygromorph biocomposites. Thus, the development of Thermo-Hygromorph biocomposites for outdoor applications requires an accurate understanding of the complex hygrothermal coupling impact on continuous flax fibre reinforced biocomposites.
Firstly, sorption isotherms of biocomposites have been measured by Dynamic Vapor Sorption (DVS) for different temperature (20°C, 40°C, 60°C, 80°C) to identify the material hygrothermal state for any environmental conditions (Fig.1a). Then, mechanical properties and hygroscopic expansion have been evaluated for various temperature and relative humidity conditions. The considered humidity range (0% to 95%RH) as well as the temperature range (20°C to 80°C) are supposed to cover the RH and temperature variation of various climates, within the limits of experimental constraints.

RESULTS AND CONCLUSIONS

The DVS results show that the rise in temperature accelerates the kinetics of sorption but reduces the moisture content at saturation. The mechanical characterization highlights that the longitudinal elastic modulus of continuous flax fibres reinforced biocomposite is strongly impacted by temperature (-47% from 20 to 80°C for dry material) and moisture content (-60% from 0 to 5.7%MC at 20°C) (Fig.1b).

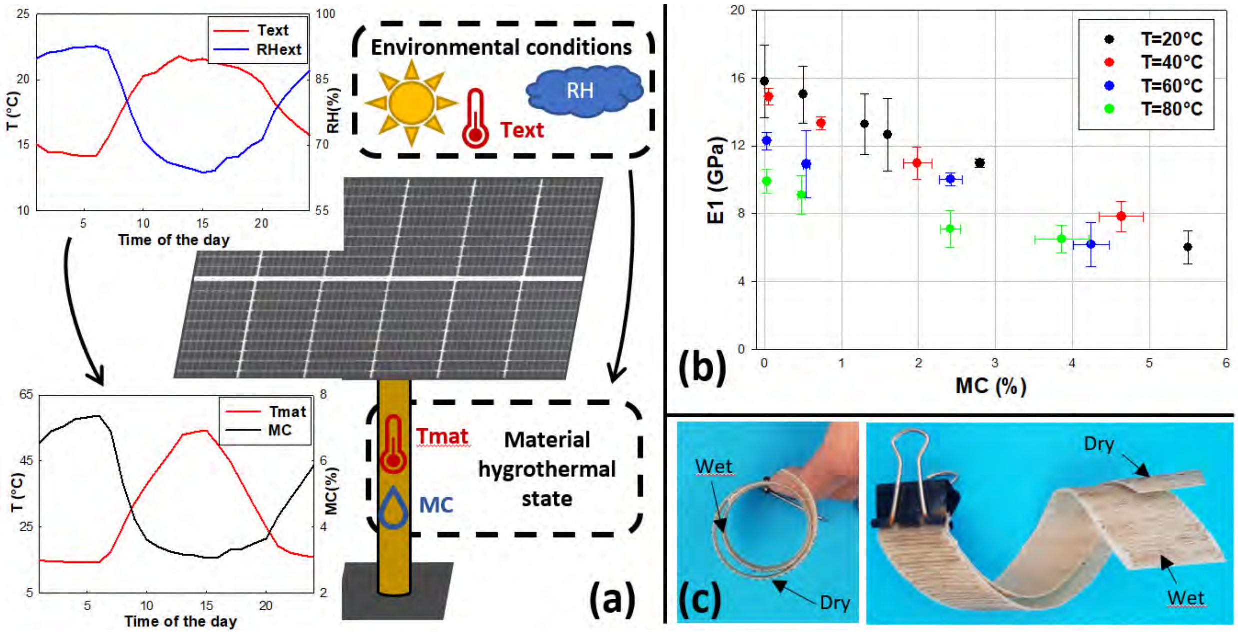

Fig.1 (a) Daily variations of temperature and RH impact the MC and material temperature of biocomposite located outside (b) Influence of temperature and MC on longitudinal elastic modulus of cFF/PLA (c) Helicoidal bilayer of cFF/PLA at dry state and wet state.

More results about mechanical characterization and thermo-hygro-expansion of biocomposites will be discussed. Further tests should be performed on the potential of actuation of helicoidal THBC (Fig.1c).

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ID 53
INFLUENCE OF MOISTURE ON THE ASSESSMENT OF THE DAMAGE ON NATURAL FIBER COMPOSITES INDUCED BY DRILLING

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ABSTRACT
Nowadays, it is essential to find new materials with minimal environmental impact. Natural fibers are being increasingly used in polymer composites due to their eco-friendly nature and sustainability. To use this type of fibers on composites it is important to have knowledge and guarantee of their mechanical properties. Drilling is one of the common methods for connecting structures made of materials reinforced with fibers. Due to the ease of maintenance and repair, the mechanical connection through screws or rivets, which inevitably must create holes in these members. In this work, a jute/polyester laminated composite is produced, characterized, the mechanical properties evaluated, and, at the end, drilled. The hole surrounding region is inspected and the areas of the damaged regions are computed. Then a new set of laminates were produced and after drilling submitted to moisture degradation. The drilled area was analysed and comparisons with the non-degradated laminate were made.

INTRODUCTION
Drilling composite materials is a complex operation due to the heterogeneity, anisotropy, sensitivity to heat and the abrasive characteristics of some reinforcements. Its execution can cause serious defects in parts such as delamination, interlaminar cracks, thermal damage, burrs, and fiber/matrix displacement. Of all the defects that the drilling can originate, delamination can be considered the most critical. [1]
As can be seen in the figure 1, drilling causes delamination both on the top face (entry) and on the bottom (outlet) face of the composite. At the entrance, the drill causes the pulling out of the upper layers, due to the larger cut fillet, from the moment the end of the drill cuts the first layer. At the exit, the drill acts as a punch and the feed force promotes delamination of the lower layers, [2] as seen in figure 1.

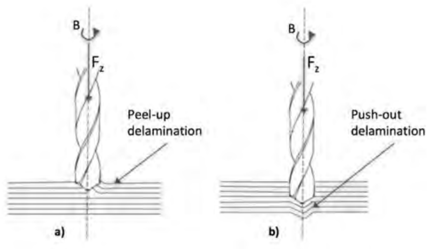

Figure 1 - Delamination during the drilling process: a) at the entrance and b) at the exit [3]

Through image processing techniques it is possible to evaluate the extent of delamination induced by drilling. In the table 1 are presented the most used parameters for delamination quantification. [4]

Table 1 – Quantitative parameters of delamination (Adapted from [4] and [5])

Refs.	Delamination factor	Formula	Drawbacks
Chen	Conventional delimitation factor (F_d)	$F_d = \frac{D_{max}}{D_{nom}}$	Damage area contribution is ignored
Faraz et al.	Two-dimensional delamination factor (Fa)	$F_a = \left(\frac{A_d}{A_{nom}}\right)\%$	Damage area contribution is ignored
Mohan et al.	Delamination factor (F_d)	$F_d = \frac{A_d}{A_{nom}}$	Maximum crack length contribution is ignored
Davim et al.	Adjusted delamination factor (F_{da})	$F_{da} = F_d + \frac{A_d(F_d^2 - F_d)}{A_{max} - A_{nom}}$	Fine crack contribution is ignored
Tsao et al.	Equivalent delamination factor (F_{ed})	$F_{ed} = \frac{1}{D_{nom}}\sqrt{\frac{4(A_d + A_{nom})}{\pi}}$	Maximum crack length contribution is ignored

There are several authors who study the ideal parameters for a drilling of quality. Hocheng and Tsao state that the geometry and material type of the tool are very important factors that influence hole quality [6]. The type, the speed of feed and tool rotation are also factors that have a major impact on drilling of composites according to a study carried out by Ramulu et. al [7]. Hocheng et. al developed a mathematical model demonstrating that pre-drilling reduces delamination [8].

The moisture degradation is one of the most important degradation processes in natural fibers.

Degradation by moisture was made in an Environmental chamber (humidity: 98 ± 0%; temperature: 24 ± 3ºC). Each day, the weight of the drilled specimens was recorded. Once the weight was stabilized, the samples were removed from the chamber and kept under atmospheric conditions. By the moment the weight came back to stabilization some samples were tested to obtain the mechanical behavior and on others were performed bearing tests to assess the induced damage.

RESULTS AND CONCLUSIONS

The mechanical properties of the non-degradated composites and the assessment of the drilling induced damage are expressed in the following tables:

Table 2 – Mechanical Properties

	Max. stress [MPa]	Max. extension [%]	E [MPa]
Tensile Properties (ISO 527 Standard)	59.5 ± 2	2.51 ± 0.2	4796 ± 13
Flexural Properties (ISO 178 Standard)	86.4 ± 5	7.34 ± 0.5	4507 ± 11

Table 3 – Hole Drilled Parameters obtained by image Analysis

	Perimeter [mm]	Hole área [mm²]	Damage area [mm²]	Surrounding circle radius [mm]
Non-degradated composites	26.262 ± 7.69	28.617 ± 0.38	4.413 ± 1.67	4.126 ± 0.5

Table 4 - Results of delamination factors

	Chen [Fd]	Faraz [Fa]	Mohan [Fd]	Davim [Fda]	Tsao [Fed]
Non-degradated composites	1.293 ± 0.05	13.24 ± 1.9%	0.132 ± 0.02	2.479 ± 3.73	1.007 ± 0.01

Table 5 - Bearing test results

	Bearing Stress [MPa]
Non-degradated composites	86.8 ± 9.99

These results drive us to conclude that the damage caused by drilling can be determined through the delamination factor calculated according to the equations of the different authors. Is possible to analyse the geometry of the hole recurring to image analysis techniques such as X-Ray and Digital Image processing. Is also a possibility that the elasticity of the fibers (higher than carbon or glass fibers) combined with the compression state induced on the bearing test allow that the Bearing stress can be higher than the Maximum Stress.

At the end of the work, we want to achieve the comparison between these data and the ones obtained in moisture degradated composites.

ACKNOWLEDGMENTS

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ID 54
HEMP FOR HIGH-VALUE TEXTILE APPLICATIONS: PROGRESS ON UPSCALING CULTIVATION, HARVESTING AND PROCESSING ON THE FLAX SCUTCHING LINE

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ABSTRACT
The main objective of this study is to build a local hemp-for-textiles value chain by scaling up the cultivation, harvesting and processing of hemp. Therefore, during three growing seasons (2020-2022) hemp was grown and harvested on a (semi) practical scale, and processed into long aligned fibers on an industrial flax scutching line. Results show that straw yield can reach 8.6 ton/ha and long fiber yield 15.4%, and most likely crop productivity can further be improved. These findings demonstrate the potential of hemp as a high yielding fiber crop that can help to meet the global demand for sustainable textile fibers.

INTRODUCTION
Hemp offers great potential as a sustainable source of textile fibers within the growing circular, bio-based economy. Yet, to build a viable local hemp-for-textiles industry, agronomic practices and fiber processing need optimization to meet current industrial standards. A straightforward approach for the region of Flanders, which has a long-standing tradition in high-quality linen production (flax), is processing hemp using the existing, modern equipment for flax (Amaducci et al., 2008; Vandepitte et al., 2020).

This study focusses on the upscaling of hemp cultivation, harvesting and primary processing (fiber extraction). During three growing seasons (2020-2022), hemp was grown on a (semi) practical scale on the Bottelare experimental farm or at a farmer (Ghent area, soil type: light sandy loam). Sowing was done in relatively mild and humid weather, preferably in the first half of May. Seeds were drilled into the soil with an automatic seeder, at a sowing depth of 2 cm, a row spacing of 10 cm and a sowing density of 400 seeds/m^2 (70 kg/ha), as recommended by the seed supplier when growing for fiber purposes (HEMP-it). The intended fertilization was 70-100 units of active N, 40 P_2O_5, 110 K_2O and 30 MgO and was applied in the form of liquid N, triple superphosphate and patent potassium in a single dose before sowing. After sowing, bird protection was provided. No further crop protection measures were applied.

RESULTS AND CONCLUSIONS

In 2020, at mature flowering (27/07/2020), 73 days after sowing (15/05/2020), 0.25 ha USO31 was harvested with a prototype harvesting module, developed by the Belgian pre-starter HempInvest. At harvest, mean plant height was 225 cm and plant density was 169 stems/m^2. The fiber-rich, lower stem part (from ~30 to 130 cm above-ground level) was cut and laid down in swaths for field-retting. During retting, swaths were turned once using a one-row flax turning machine (manufacturer: Depoortere). Straw was field-retted for 49 days, after which it was collected using a self-propelled round flax baler (manufacturer: Vlamalin), and subsequently processed on a nearby industrial flax breaking and scutching line (Depoortere/Vanhauwaert at Van de Bilt zaden en vlas, NL). Scutching yielded 15.4% long fiber and 24.2% short fiber.

In 2021, due to abundant, persistent rain in spring, a field of 2.8 ha USO31 was only sown in June (6/06/2021). The further development of a harvesting module by HempInvest was (temporarily) put on hold. However, at the end of summer 2021, the Belgian company Hyler BV announced a new, fully automatic harvesting machine. With this Sativa 200 prototype, approx. 60% of the field could still be harvested (09/09/2021; seed stage, 95 days after sowing). This machine cuts the hemp stem into two parts and lays the upper and lower stem part, each approx. 1 m long, in separate swaths (Fig. 1). At harvest, mean plant height was 182 cm and plant density 175 stems/m^2, but the crop condition was very heterogeneous. During a retting period of 38 days, turning was done with a two-row flax turning machine (manufacturer: Depoortere). Scutching yielded 8.9% long fiber and 31.2% short fiber. Fiber quality of long fibers after scutching showed a fiber fineness and tenacity comparable to flax.

In the dry spring of 2022, 0.82 hectares of hemp was sown (08/05/2022). At the beginning of August (05/08/2022) the crop was harvested at full flowering with Hyler's Sativa 200 module. This time under ideal field conditions. At harvest, the crop reached an average height of 205 cm and a plant density of 195 stems/m^2. The retting period was 38 days and swaths were turned twice using the two-row flax turning machine. The estimated straw yield was 8.6 ton/ha.

Fig. 1. Automated harvesting with Hyler's Sativa 200 module (left), field retting in separate swaths (mid) and scutched long fibers (right) of hemp.

These findings demonstrate the potential of hemp as a high yielding fiber crop that can help to meet the global demand for sustainable textile fibers. Further field tests at farm level should be performed in order to analyze the hemp yield performance in different growing conditions (a.o. weather, soil type, variety, crop rotation, harvest time) and to gather data on the economic feasibility of hemp cultivation for the farmer.

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ID 55

DEEP UNDERSTANDING OF THE RELATIONSHIP BETWEEN THE MECHANICAL PERFORMANCE OF FLAX FIBRES AND THEIR STRUCTURAL DEFECTS

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ABSTRACT

This work investigates the potential relationship between the mechanical properties and both the quantity and the proportion of kink-bands in single flax fibres. For this purpose, a selected batch of textile flax was scutched and combed. The intensity of the processing parameters was varied in order to obtain fibres with four different extraction conditions. The number and size of defects was quantified through polarized light microscopy (PLM) investigations on a large number of single fibres, in order to compare and correlate them with their tensile mechanical properties and behaviour. It was demonstrated that surprisingly an intense combing stage induces a decrease of the severity of defects and interestingly, significantly decreases the tensile strength of elementary flax fibres.

INTRODUCTION

The morphological characteristics of the single flax fibre, (defects, kink-bands, dislocations) are sensitive areas and potential zones of failure. Extensive studies have shown the presence of increased and local porosities in kink-bands by tomography investigations, which probably favours stress concentration in these zones (Bourmaud, 2022). This would result in weakness areas where induced pores would be entry points for micro-organisms, enzymes or water (Hernandez, 2016). Moreover, structural defects are likely to be generated during the mechanical and aggressive industrial extraction and their number increases with the number of processes (Hänninen, 2012). However, it is still difficult to understand the correlation between the defect content and mechanical performance. In some publications, it is shown that modulus decreases with the number of kink-bands (Davies & Bruce, 1998), but this correlation is not clearly demonstrated for other authors (Thygesen, 2007), suggesting that more than the number, other parameters linked to the defect structure itself must be taken in account.

The main objective of this work is therefore to explore the density and morphology of kink-bands by different approaches in order to improve our knowledge on their implications in mechanical properties. Four batches of flax fibres of the Felicie variety (2021) were extracted using different intensity parameters with a laboratory scale scutching/hackling line. The fibres and regions of kink-bands were observed using different experimental techniques, including PLM, second harmonic generation (SHG) microscopy, scanning electron microscopy (SEM), and tomography as well. Then, the number and the size of the defects were correlated with the tensile mechanical properties of the same individual fibres.

RESULTS AND CONCLUSIONS

The four batches were analysed and compared. We found strong differences between the uncombed and the severely combed batches. The first results for the most severely scutched and hackled batch are shown in Fig. 1. The strength at break is negatively correlated (r=-0.68) to the defect content (calculated as the ratio of the total defect area to the total fibre area). However, this correlation does not exist for uncombed batches which have larger defects, showing that defects have an impact on the mechanical properties when the fibres have been subjected to an intense combing.

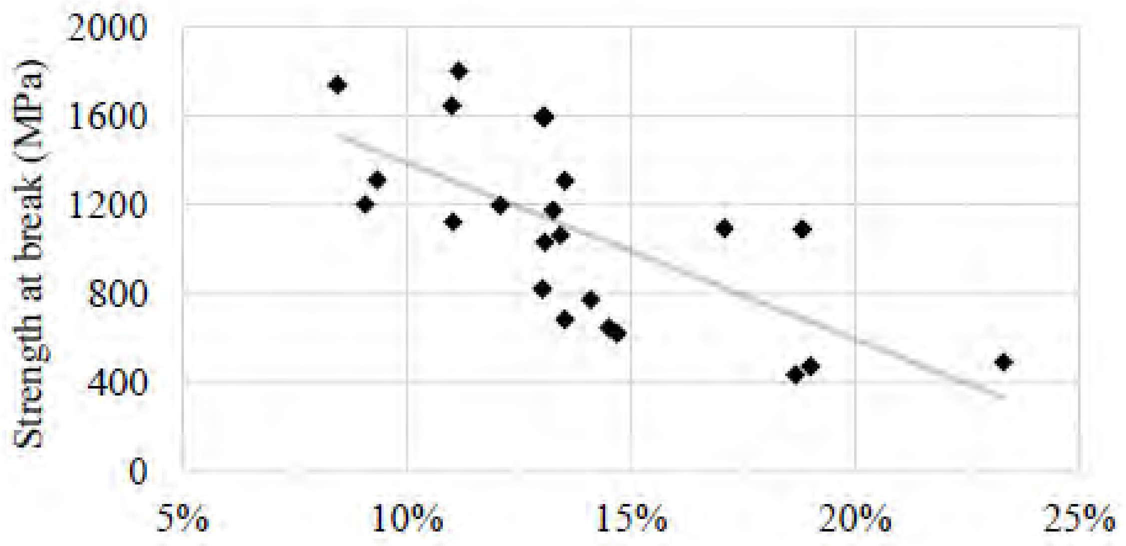

Fig.1 Correlation between fibre tensile strength and proportion of defects for high intensity extraction

Additional points will be discussed in the presentation. Particularly the study of the type of stress-strain curve (type I, II or II) as a function of the mechanical properties and morphological characteristics of flax fibres will be proposed.

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ID 57
POTENTIAL OF CEREAL-BASED AGRICULTURAL WASTE FOR THE PRODUCTION OF TECHNICAL TEXTILES

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ABSTRACT
Major green initiatives of bio-based industries include usage of underexploited feedstocks from crop plants, fungi and algae, trees, agricultural and forestry residues. This research focus on two different residues derived from cereal-based agricultural waste: barley and wheat straw. Applied biomass was used to produce biofibers primarily for technical textiles purposes. Quality of fibers extracted from lignocellulosic waste proved to be in line with convenient cellulose-based products, so the planned path towards production of technical textiles (e.g. biocomposites or biofilters) are achieved. On the other hand, solid waste from the wheat and barley fibers production amounts up to 24.82% (wheat) and 32% (barley), presents valuable resources for biorefineries. Based on the produced fiber quality and energy properties of solid waste from the extraction of fibers the next step of the circular economy processes is performed while all crop parts are utilized.

INTRODUCTION
Although, the concept of using lignocellulosic biomass as a feedstock for biorefineries is already wide accepted its pre-usage for the production of alternative, sustainable fibers is still underexploited. To foster faster transformation to circular economy, new funding possibilities are given through numerous circular and bio-based schemes. The most important principle is *food first* and to satisfy it, a clear distinction should be made between food part (primarily grains) and non-food part (husk, leaves, stalk and straw).

In general, cereal-based agricultural waste contain a large fraction of lignocellulose substances, making them excellent sources for biorefineries, but production of fibers as well. The biomass composition is depending on its origin but in general, it is composed of cellulose (~40−50%), hemicellulose (~25−35%), and lignins (~10−25%) (Isikgor, 2015).

Since pretreatment processes can cost up to 40% of the total cost they represent the drawback of biomass conversion to value added products (Sindhu et al., 2016). Additional challenge is associated with the requirements for green chemicals and methods usage. Therefore, companies are encouraged to use advanced technologies such as: microwaves, ultrasound, high pressure, ionizing radiation (gamma ray, electron beam), pulsed-electric field (Hasan, 2018).

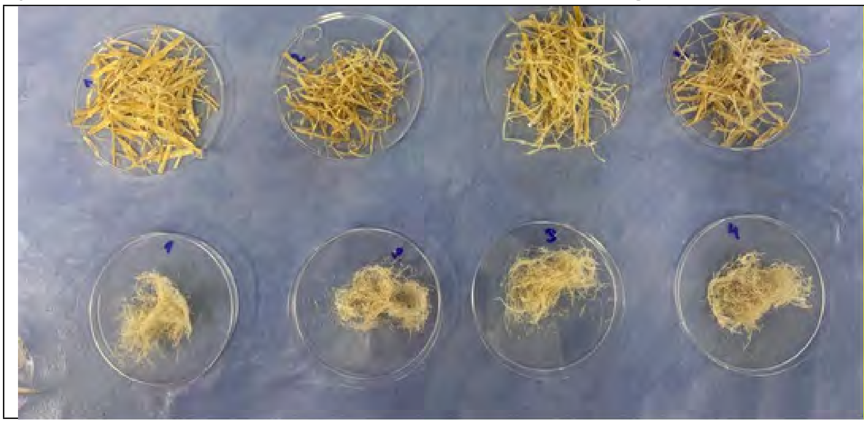
RESULTS AND CONCLUSIONS
The results of fiber yield and fiber properties of different cereal-based biomass pre-treatment are presented in Table 1. Better results of fibre yield were achieved with 3% of NaOH treated for 90 min and the fiber density of this fibers proved to be low. Low density helps to reduce the weight of the final product, which is extremely important, for example, in the automotive industry, taking into the consideration that the reduced weight directly affects the reduction of fuel consumption.

Table 1 Comparison of cereal-based lignocellulosic crops properties

Crop	Chemical agent	Time (min)	Fiber yield (%)	Moisture content (%)	Density (g/cm³)
Barley straw	**3% NaOH**	**60**	8.9	/	/
		90	10.6	10.52 (0.08)	1.48 (0.00)
	5% NaOH	**60**	6.9	/	/
		90	8.2	/	/
Wheat straw	**3% NaOH**	**60**	9.5	/	/
		90	10.2	10.63 (0.03)	1.42 (0.08)
	5% NaOH	**60**	7.2	/	/
		90	8.9	/	/

Data in brackets represent standard deviation

Due to the fact of much higher biomass yield of barley straw (up to 61 tons), this crop is chosen for visual display of pretreatment procedure, as presented at Fig. 1.



a) Biomass after first pretreatment step with NaOH

b) Cellulose fibers after removal of hemicellulose and lignin

Fig.1 Pretreatment of barley straw biomass: 1) 5 % NaOH, t=60 min; 2) 5 % NaOH, t=90 min; 3) 3 % NaOH, t=60 min; 4) 3 % NaOH, t=90 min

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ID 59

NOVEL HETEROGENEOUS CATALYST: $COFE_2O_4$ NANOPARTICLES SUPPORTED ON CELLULOSE BEADS FOR ORGANIC POLLUTANT DEGRADATION VIA PEROXYMONOSULFATE ACTIVATION

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ABSTRACT

Organic dyes are an essential group of industrial chemicals that find widespread usage in various applications such as paints, textiles, cosmetics, leather, pharmaceuticals, and plastics. However, the detrimental impact of these industries on the environment, including water resource degradation and air pollution, has raised significant concerns [1-2]. Organic molecules with hazardous effects including high toxicity, slow biodegradation, and potential carcinogenicity are often found in wastewater and agricultural land. This can harm humans, plants, and living organisms in these areas. Thus, the development of efficient and cost- effective technologies for the removal of organic pollutants from aqueous solutions is a high priority. Advanced oxidation processes (AOPs) are widely used to treat organic pollutants that are difficult to degrade in the environment. Among these, advanced oxidation processes based on peroxymonosulphate (PMS) have shown great potential as a technique for removing a wide range of organic pollutants from water-based solutions [3].

Although hydroxyl radicals (•OH) are known for their high reactivity towards organic compounds and have been extensively studied, their application is limited due to various constraints regardless of how they are generated (e.g., ozone, Fenton/Fenton-like process). These constraints include complex and expensive radical generation processes, pH adjustments, and potential sludge generation. Sulfate radicals (•SO4−) have gained significant research attention as a substitute for hydroxyl radicals, offering a higher standard reduction potential (2.5-3.1V) under neutral conditions, which leads to superior reactivity and selectivity for oxidation processes compared to hydroxyl radicals (1.8-2.7V) [4]. Sulfate radicals can compensate for critical inadequacies of hydroxyl radicals. Additionally, sulfate radicals can be easily released from PMS or persulfate (PS) through catalytic activation by some transition metal ions. Among them, Co2+ coupled with PMS has been found to provide outstanding sulfate radical yield and effectively decontaminate organic pollutants [5].

In this study, new cellulose beads (CBs) were produced from date palm fibers (DPFs) using for the first time a mechanical process patented by our research team. The cellulose beads were then used in the preparation of a magnetic CoFe2O4/CBs hybrid and its catalytic performance in the PMS oxidation of rhodamine

B (RhB) for the first time. In addition, the potential influential factors including catalyst and PMS assays, temperature and pH value were investigated in terms of the catalytic behavior of the CoFe2O4/CBs. The coupling of the spinel ferrite nanoparticles to cellulose beads enhances the stability and reusability of the catalyst, making it a more practical and sustainable option for catalytic processes. The resulting catalyst also shows excellent catalytic activity, which can be attributed to the synergistic effects between the spinel ferrite nanoparticles and the cellulose beads. This new method has the potential to revolutionize the field of heterogeneous catalysis and pave the way for more sustainable and efficient catalytic processes.

EXPERIMENTAL SECTIONS

1. MATERIAL SYNTHESIS

The CBs are produced in three major steps, fibers bleaching, stirring, and recovery. After DPFs extraction, they were initially rinsed in water for surface debris removal and then dried for 24h. Subsequently, the DPFs were ground and treated with an alkaline solution to remove non-cellulosic materials including lignin, hemicellulose, and compounds covering the outer surface of fibers. The fibers were further bleached using bleaching agent under defined conditions of pH, temperature and stirring. The second step consists in stirring the cellulose suspension using a stirrer rod under a well-defined condition of controlled speed and time, which results in different controlled sizes of beads. Finally, the cellulose beads were recovered using sieves. The produced CBs are characterized by high surface area, controlled size, and high chemical, mechanical, and thermal stability. The developed cellulose beads were coupled to spinel ferrite nanoparticles using co-precipitation approach.

2. MATERIAL CHARACTERIZATIONS

The structural, morphological, and thermal properties of CoFe2O4/cellulose beads were characterized using a Fourier-Transform Infrared Spectroscopy (FT-IR), Scanning electron microscopy (SEM), X-ray diffraction (XRD), Thermogravimetric analysis (TGA), and Brunauer–Emmett–Teller (BET). Furthermore, CoFe2O4/cellulose beads were evaluated in terms of the degradation of Rhodamine B (RhB) under different conditions, including Rhodamine B concentration, catalyst dose, PMS dose, temperature, pH, and time. The r cyclability and regeneration of CoFe2O4/cellulose beads were also investigated, Finally, a possible reaction mechanism was hypothesized and elucidated.

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ID 61
INDUSTRIAL HEMP AS A SUSTAINABLE SOURCE OF TEXTILE FIBER: OPTIMIZATION OF HEMP CULTIVATION IN FLANDERS

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ABSTRACT
In the ‘Hemp4Textiles’ project, a continuation of previous research (Vandepitte et al., 2020), we aim to investigate the impact of agronomic factors on crop yield performance and hemp fiber quality. Therefore, the influence of variety, nitrogen fertilization and sowing density were investigated through a 2-year variety and multifactorial field trial. Amongst the five varieties tested (USO31, Bialobrzeskie, Santhica 27, Santhica 70 and Fibror 79), differences in fiber yield were observed. The multifactorial field trials demonstrated that both the fertilization rate (range: 0-100 N) and variety affected bast fiber yield, while the investigated sowing densities (range: 300 – 600 s/m^2) did not. The yield and quality of long hemp fiber after scutching will be determined and both field experiments will be repeated for the next two growing seasons.

INTRODUCTION
The textile industry is one of the most polluting industries in the world (Franco, 2017), therefore the demand for more environmentally friendly and sustainable fibers such as flax has increased. However, due to its long crop rotations (1/7) (Heller et al., 2015), the maximum flax production capacity in Europe has been reached and can no longer satisfy the increasing demand. An alternative might be the cultivation of industrial hemp (*Cannabis sativa* L.) for its long fiber, similar to flax. In flax-producing areas, including Flanders, hemp can be processed using the industrial equipment for flax (Grégoire et al., 2021; Müssig et al., 2020; Vandepitte et al., 2020). However, to establish a viable hemp-for-textiles industry, there is a need for specific optimization of agronomic practices and fiber processing to meet current industrial textile standards.
In this project the influence of variety, nitrogen fertilization and sowing density on long fiber yield and quality (after scutching) are investigated. A 2-year variety and multifactorial field trial were set up. The variety trial consisted of 5 varieties from diverse European origin and maturity. The multifactorial trial evaluated the effect of multiple nitrogen fertilization levels and sowing densities on plant biometrics and yields of 2 varieties (USO31 and Santhica 70). All trials were harvested using a double sickle bar mower at flowering and left in swaths in the field for dew-retting. The ‘green’ bast fiber yield was determined, which gives an estimation of the total potential fiber yield (Tang et al., 2016). After retting, the stems were cut at 1 m length and scutched on an industrial flax processing line (Van de Bilt), rendering long aligned fiber, in addition to short fibers (‘tow’) and shives. At harvest crop parameters, ‘green’ bast yield and long fiber yield were assessed.

RESULTS AND CONCLUSIONS

Variety Trial

The influence of variety on plant biometrics, dry matter yield (Y_{DM}) and 'green' bast yield (Y_{BAST}) is shown in Table 1. Overall, the early and mid-late varieties show a smaller stem diameter, together with a lower plant height and dry matter yield. Differences in bast fiber yield, thus maximum potential fiber yield, are not related to earliness. Bialobrzeskie and Santhica 27 show promising results in Flanders, with the highest bast fiber yield. The dry matter yield for all cultivars was higher in 2022 than in 2021 (data not shown).

Table 1: VARIETY TRIAL OF INDUSTRIAL HEMP IN FLANDERS

Variety	Origin	Flowering	Diameter (mm)	Plant height (cm)	Y_{DM} ($Mg.ha^{-1}$)	Y_{BAST}* ($Mg.ha^{-1}$)
USO 31	Ukraine	Early	8.60 ± 0.69^{ab}	203 ± 4^{a}	9.38 ± 0.72^{a}	2.87 ± 0.33^{a}
Bialobrzeskie	Poland	Mid-late	8.00 ± 0.54^{a}	214 ± 5^{b}	10.32 ± 0.56^{ab}	3.54 ± 0.34^{b}
Santhica 27	France	Mid-late	8.44 ± 0.55^{ab}	212 ± 5^{ab}	10.37 ± 0.57^{ab}	3.45 ± 0.34^{ab}
Santhica 70	France	Late	9.38 ± 0.55^{bc}	217 ± 5^{b}	11.90 ± 0.58^{c}	3.38 ± 0.37^{ab}
Fibror 79	France	Late	9.76 ± 0.54^{c}	221 ± 5^{b}	10.63 ± 0.58^{b}	2.85 ± 0.34^{a}

Variety mean yield performance for the harvest years 2021 and 2022. Letters denote statistically significant pairwise differences ($p<0.05$). *Y_{BAST} is only shown for 2021.

Multifactorial trial (data not shown)

A higher fertilization rate leads to more produced biomass, which increases the bast fiber yield. However, no significant differences are seen in fiber yield of 50 and 100 kg N/ha while 0 kg N/ha showed significantly lower yields in the field trial of 2021, which indicates that the highest fertilization rate might not be necessary for optimal fiber yield. Additionally, lower fertilization rates help to reduce the environmental impact of hemp which ties in with the targets of the European Green Deal. Similar to the variety trial, variety has a significant influence on stem diameter, while the sowing density (400 & 500 s/m²) showed no significant effect on any tested parameter. This implies that a lower seeding rate (400 s/m²), does not significantly affect the yield but reduces the initial seed cost. The long fiber yield and quality will be determined and both field trials will be repeated for the next 2 growing seasons.

ACKNOWLEDGMENTS

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ID 62
FLAX/BIODEGRADABLE POLYMER COMPOSITES: OPTIMIZATION OF PLA/PBAT/PHBV TERNARY BLEND FORMULATION AND EFFECT OF THE NATURAL FIBERS ON THE COMPOSITE PROPERTIES.

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ABSTRACT
In this work a ternary blend of polylactic acid (PLA), poly(butylene adipate-co-terephthalate) (PBAT) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) was used for the manufacture of green polymer composites by melt extrusion and injection molding. The optimization of the blend formulation was conducted by measuring the mechanical (tensile, flexural, impact and hardness) and the thermal properties (thermogravimetry, differential scanning calorimetry and dynamic mechanical analysis) of specimens with different weight ratios of PLA/PBAT/PHBV (16/64/20 Blend_1, 12/48/40 Blend_2 and 8/32/60 Blend_3 (w/w/w)). To further improve the mechanical properties of the best formulation (as a trade-off between strength and elongation), the blend was used for the manufacture of green composites with different amounts of flax fibers as a reinforcing agent.

INTRODUCTION
The transition to bioplastics and biodegradable polymers represents a valid strategy to meet the objective of the Zero Pollution Action Plan as one of the policy areas of the EU Green Deal. However, the complete replacement of conventional plastics cannot be implemented in a short time because of their extensive use (Mohite, 2022). Among the biodegradable polymers PLA, PBAT and PHBV are noteworthy but they have pros and cons. PLA is the most commonly used thanks to its high biodegradability and low cost but its major drawbacks are the high glass transition and brittleness that limit its processability. PBAT is a biodegradable copolymer with high flexibility which allows its use only in specific applications while PHBV has similar properties to polypropylene but it is rigid and brittle. To overcome the limits of such materials, the blend of polymers with opposite properties is a simple and economic technique (Pal, 2020). Despite several works on binary blend formulations are available in the literature that highlight limitations on the improvement of mechanical properties (Qiao, 2022), only a few authors have studied the properties of PLA/PBAT/PHBV ternary blends: PLA-rich mixture for 3D printing filament production (Yang, 2019) and equal mass fraction blends as films for packaging applications (QuilesCarrillo, 2019). In this work, a ternary blend based on a PBAT-dominated commercial PLA/PBAT (20/80 w/w) blend and PHBV was investigated. The best formu-

lation (optimum between strength and flexibility) was used as matrix for the manufacture of biodegradable composites with different amounts of flax as a green reinforcing agent.

RESULTS AND CONCLUSIONS

The mechanical properties evaluated through quasi-static tests are summarized in Table 1 and it is possible to notice that with the increase of PHBV content in the ternary blend the strength and the modulus increased in both tensile and flexural conditions while a decrease of the max elongation up to 2.67% for the Blend 3 was measured. The Table 1 also reports the effect of the addition of flax fibers (10 wt%) in Blend_2. The tensile properties of the composite (Blend_2_Flax) were negatively affected by the presence of the flax fibers because of the poor adhesion between the matrix and the filler which needs to be optimized. On the contrary, a positive effect was observed in flexural properties with an increase around 34% in flexural strength and 79% in flexural modulus with respect to the pure polymer blend. It is known that the properties of short-fiber composites are determined not only by the quality of the fiber/matrix interface, but also by the fiber orientation factor, which in injection molded composites is complex including a skin layer, a shell layer and core layer. Comparing tensile and flexural tests, the active volume that sees the highest stresses, i.e. the sampling surface/volumes, are very different, as well as their interaction with the fibers.

Table 1 Quasi-static mechanical properties

Sample	PLA/PBAT/PHBV/Flax [wt%/wt%/wt%/wt%]	Tensile strength [MPa]	Young's modulus [GPa]	Flexural strength [MPa]	Flexural modulus [GPa]
PLA/PBAT	20/80/0/0	10.42 ± 1.86	0.30 ± 0.06	8.51 ± 0.16	0.19 ± 0.09
Blend_1	16/64/20/0	16.02 ± 0.97	0.94 ± 0.08	14.52 ± 2.03	0.48 ± 0.11
Blend_2	12/48/40/0	20.36 ± 0.58	1.37 ± 0.08	21.44 ± 2.67	0.87 ± 0.19
Blend_3	8/32/60/0	25.65 ± 3.57	2.07 ± 0.20	47.06 ± 1.51	2.25 ± 0.08
PHBV	0/0/100/0	38.39 ± 0.75	4.07 ± 0.05	57.56 ± 2.28	3.78 ± 0.17
Blend_2_Flax	10.8/43.2/36/10	16.03 ± 1.51	1.32 ± 0.21	28.75 ± 3.10	1.56 ± 0.09

As regards the thermal stability, a decrease in the onset temperature of PLA/PBAT blend of about 17% when the concentration of PHBV reaches the 40 wt% of the ternary blend ($T_{5\%}$= 284 °C) was observed. The addition of flax fibers did not further decrease the thermal stability, while keeping constant polymers properties in terms of glass transition and melting temperatures and polymers crystallinity (X_{PBAT}= 2.65% and X_{PHBV}=72.5%).

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ID 64

FACILE AND BIOINSPIRED DEVELOPMENT OF A NOVEL BIO-BASED COATING FROM GALLIC ACID OF NATURAL FIBERS FOR COMPOSITE APPLICATIONS

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ABSTRACT

In this work, natural fibers were coated with a novel, sustainable and bioinspired gallic acid-iron-phenyl-phosphonate (GA-FeP) complex in order to improve fiber-matrix interfacial adhesion and flame retardancy in composite materials. Basalt and flax fibers were chosen as models. Microwave plasma atomic emission spectroscopy (MP-AES) analysis and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS) of the treated fibers revealed a significant increase in iron content, changes in fiber morphology, and presence of phosphorus, respectively.

INTRODUCTION

Over the past decade, natural fibers have emerged as an alternative material for reinforcing polymer matrices. Compared with conventional synthetic fibers (e.g., carbon, glass and aramid fibers), the use of natural fibers in fiber-reinforced polymer (FRP) composites provides recyclability, renewability, low density, low cost, thermal insulation, and CO_2 neutrality. However, natural fiber-reinforced composites (NFRCs) have some drawbacks, such as poor fiber-matrix interfacial compatibility and fire resistance. Therefore, flame retardants (FRs) are needed to improve the flammability resistance of NFRCs and expand their fields of application. Methods of applying FR to composite materials reported in the literature include treatment of reinforcing fiber (Shi, 2022). In this work, we present an innovative bioinspired approach for coating natural fibers with a complex from renewable sources containing iron phenyl phosphonate, that holds promise to behave as FR (Zhang, 2018) and, given its organic component, might improve interfacial fiber-matrix adhesion. Flax fabrics and basalt fabrics, which show different chemical and physical properties, were taken as models to demonstrate the effectiveness of the treatment. The coating of the two natural fibers involves three steps: (I) the fibers are treated with O_3, resulting in free hydroxyl groups on the surface that allow the subsequent (II) covalent immobilization of biosourced gallic acid (GA) units on the fiber through ester linkages. Inspired by the bacterial siderophore enterobactine (Raymond, 2003), (III) the phenolic -OH groups of the gallic acid units are then exploited for the complexation of iron phenyl phosphonate (FeP), thus obtaining the fiber coated with the GA-FeP complex.

RESULTS AND CONCLUSIONS

The schematic representation of fabric treatment is shown in Figure 1a. The amount of Fe on the treated fabrics was measured by MP-AES analysis. The basalt and flax fabrics showed an iron loading of 0.1 wt% and 0.3 wt%, respectively, confirming the Fe^{III} - gallic acid unit complexation on the surface of both samples. SEM analysis of the untreated basalt fand flax fabrics (Figures 1b and 1d) reveals smooth surface fibers, while SEM-EDS analysis of the treated fabrics (Figures 1c and 1e), reveals a globular shaped coating on the surface of both fibers. EDS spectra show a high amount of P and Fe, confirming that the material bound to the two fabrics is iron phosphonate.

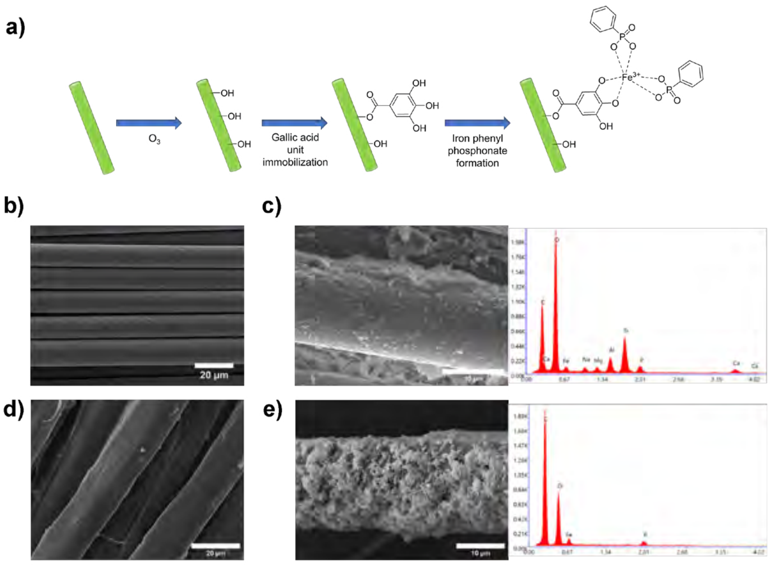

Figure 1. (a) Proposed natural fabric surface modification; (b) SEM micrograph of untreated basalt fabric; (c) EDS analysis of treated basalt fabric; (d) SEM micrograph of untreated flax fabric; (e) EDS analysis of treated flax fabric.

A sustainable, bioinspired coating for natural fibers having different chemical and physical characteristics has been proposed and successfully implemented. The coating was conceived with the aim of improving fire resistance and fiber-matrix compatibility in composite materials.

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ID 65
FROM EPOXIDES TO SUSTAINABLE POLYHYDROXYURETHANES: CAN CO_2 ENHANCE NATURAL FIBER COMPOSITE PROPERTIES?

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ABSTRACT
This work investigates the interest to implement non-isocyanate polyurethane (NIPU) chemistry as sustainable matrices in natural fiber composites. As obtained by aminolysis reactions between CO2-sourced cyclic carbonates and amines, such PHU represents an interesting platform in terms of sustainability and strong interactions with cellulosic fibers via their hydroxyl moieties along the backbone. To our best knowledge, different PHU thermoset formulations were investigated in order to demonstrate its feasibility to design flax fiber composites for the first time. By comparison with those of its epoxy counterpart, the resulting flax/PHU materials exhibited significantly higher mechanical properties and pave the way for more sustainable fiber-based composites with enhanced properties.

INTRODUCTION
The high specific mechanical properties and low environmental footprint of natural fibers make them strong alternatives to glass fibers in composite (Mutel, 2018). However, there is still a lack of satisfying eco-friendly solutions for the polymer matrix. Gathering high compatibility at the fiber/matrix interphase, sustainability of the constituent, and processability while maintaining or enhancing the final properties remain a major lock-in (Saba et al., 2016). In the meantime, non-isocyanate polyurethanes synthesized from cyclic carbonates (obtained by carbonatation of epoxy functions with CO2) and polyamines emerge as safer and greener polymers (Khatoon et al., 2021). These new poly(hydroxyurethane) (PHU) thermosets offer highly tunable properties (Carré et al., 2019). Interestingly, the presence of hydroxyl groups within the crosslinked PHU networks could represent an efficient way to enhance the interfacial adhesion with natural fibers (NF). In this work, we investigate a PHU formulation and its feasibility to fabricate flax fiber composites to be applied in structural applications. The curing rheological behavior of the thermoset formulation and its mechanical, chemical, and physical properties were studied. Finally, flax fibers were impregnated and the resulting materials were characterized. For sake of comparison, a biobased flax/epoxy composite was also manufactured and characterized.

RESULTS AND CONCLUSIONS
The processability of PHUs was assessed through isothermal rheology over cured at 80°C, as displayed in Fig.1 (left). The initial viscosity of the PHUs system is higher than the epoxy counterpart. However, it is worth noting that the pot life (limit of processability, where $\eta < 300$Pa.s) is higher for the PHUs due to the lower reactivity of the cyclic carbonate compared to oxirane during the aminolysis. This indicates that if

the process should be adapted to PHUs, the impregnation of flax fibers remains feasible in a reasonable amount of time with conventional composite manufacturing processes.

The mechanical properties of the unidirectional flax composites were measured by three-point bending tests, and the resulting stress-strain curves and mechanical parameters are shown in Fig.1 (right) and Table 1, respectively. The flexural modulus and strength are 13% and 20% higher for the PHU-based laminates in the longitudinal direction. Moreover, the transverse properties are significantly higher for the PHU-based composite, showing a 120% higher flexural modulus and a 56% greater flexural strength. This is due to the enhanced interfacial adhesion between the flax fibers and the PHU matrix, promoted by the polar hydroxy-urethane functions in the PHU.

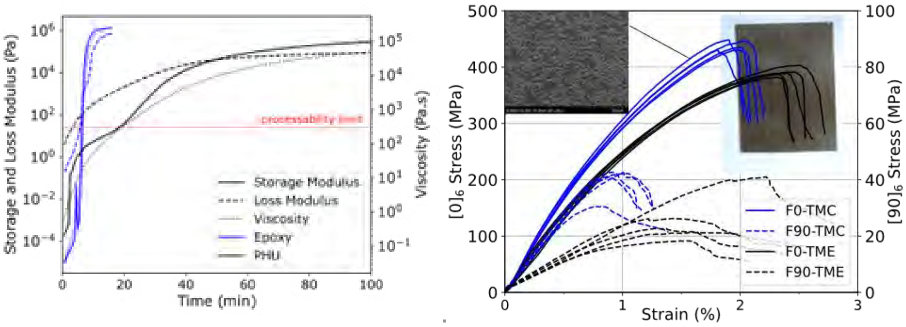

Fig.1 Three-point bending behavior of PHU (TMC) and epoxy (TME) neat matrices (left) and flax $[0]_6$ and $[90]_6$ laminates (right)

Table 1 Three-point bending test results for neat epoxy (TME) and PHU (TMC) and the manufactured flax laminates in the two orthotropic directions

	E (GPa)	σ (MPa)	ε (%)
F0-TME	27.9 ± 0.7	390.9 ± 6.9	2.39 ± 0.09
F0-TMC	35.2 ± 0.6	438.2 ± 8.1	1.94 ± 0.08
F90-TME	2.2 ± 0.2	25.9 ± 8.0	1.62 ± 0.32
F90-TMC	5.4 ± 0.4	39.8 ± 4.7	0.93 ± 0.09

We show that PHU thermoset, as obtained from biomass and CO_2, can be a sustainable alternative to epoxy, leading, in the case of natural fibers composites, to an improvement of the mechanical properties. However, if the interest in such polymers is certain, the scale-up to industrial process methods should be performed and studied to a larger extent.

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ID 66
USE OF GOOSEBERRY SHELL FOR THE BASIC RED 46 REMOVAL THROUGH ADSORPTION

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ABSTRACT
Different unconventional materials have been evaluated as adsorbents for the treatment of colored effluents from the textile industry. This work evaluated the capacity of gooseberry shell, an agro-industrial waste for the removal of basic red 46 (BR46), which is a cationic dye, widely used in different staining processes. For this analysis, a 2^3 factorial design was carried out under a discontinuous system, where the factors analyzed were, adsorbent dosage (D), initial dye concentration (Co) and contact time (t). The final concentration of the dye was quantified by UV-Vis spectrophotometry. In this way, a maximum removal of 89.39% was obtained for BR46. Additionally, Tukey's multiple range test was performed to identify if there was a statistically significant difference between the experiments.

INTRODUCTION
The dyeing process used in the textile industry usually requires an amount of water equivalent to 1 to 10 times the weight of the fibers to be dyed (Chequer et al., 2013), these effluents are generally discharged without prior treatment in the water sources generating a negative impact on the environment due to its great content of remaining dyes, approximately 8-20% (Noroozi, et al., 2008). Different treatments have been developed for the removal of dyes dissolved in water, among them adsorption is one of the most implemented techniques, since it is a low-cost alternative given that its low consumption of reagents and also allows the complete dye molecule to be retained (Díaz et al., 2014) without generating by-products that could be more toxic than the initial molecule (Gupta and Suhas, 2009).
Currently, it is feasible to use agricultural by-products, such as gooseberry shell (GS), as an alternative adsorbent for the synthetic dyes removal like basic red 46 (BR46). Thus, this work aimed to assess the uptake capacity of this agricultural waste under a discontinuous system. The effect of the factors: initial dye concentration (Co), adsorbent dosage (D) and contact time (t), were evaluated with a 2^3 factorial design with central point and two replicates per sample. The experiments were carried out at a pH value of 7.0, a particle size ranging from 0.3 to 0.5 mm, a temperature of 25 °C and a stirring speed of 180 rpm. The values of the factor levels are presented in Table 1.

Table 1. Values at the levels in the design of the 2^3 factorial of BR46

Point	Dosage [g/L]	Concentration[mg/L]	Time [h]
Low	5	30	2
Medium	7.5	45	3
High	10	60	4

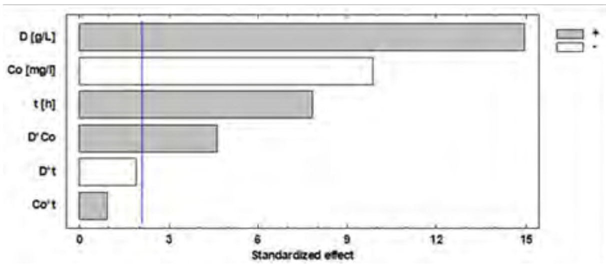
For each experimental unit, the corresponding amount of GS was weighed and added to a 50 mL Erlenmeyer flask with the dye solution at the given concentration. Finally, the concentration in the final time was measured with a Lambda 35 UV-Vis Spectrophotometer at a wavelength of 530 nm, using a calibration curve ranging from 1 to 20 ppm. The response variable corresponds to the removal percentage, defined by equation (1).

$$\% Rem = \frac{C_0 - C_f}{C_0} * 100\%\% Rem = \frac{C_0 - C_f}{C_0} * 100\% \quad (1)$$

RESULTS AND CONCLUSIONS

The results obtained with the 2^3 factorial design for the GS-BR46 system are presented in a Pareto chart (Fig.1-a) where it is shown that the most significant factor is the adsorbent dosage (D) with a positive effect, therefore, as this factor increases, the removal percentage increases proportionally. The second most significant factor is the initial concentration of dye, (Co), with a negative effect, since as said concentration increases, the percentage of removal decreases. The third most significant factor is the time followed by the interaction between the adsorbent dose and dye concentration factors, with a positive effect on the removal dye process. Finally, the interactions D*t and Co*t do not have a significant effect on the system.

(a)



(b)

Group	t	D	C	% Remotion	Tukey's range test
1	2	5	30	76,41	d,e
2	2	5	60	65,23	f
3	2	10	30	85,88	a,b,c
4	2	10	60	80,92	c,d
5	3	7,5	45	77,64	d,e
6	4	5	30	83,40	b,c
7	4	5	60	72,90	e
8	4	10	30	89,39	a
9	4	10	60	86,44	a,b

Fig.1 (a) Standardized Pareto chart for percentage removal. **(b)** Percentages of removal for the factorial design 2^3.

According to the results of the factorial design (Fig.1-b), GS allowed a maximum removal of 89.39 % at a concentration of 30.0 mgL-1, a dosage of 10 gL-1 and a contact time of 4 h (group 8). Even so, it is possible to obtain satisfactory results if we keep (t) and (D) but duplicate (Co), as the conditions described for group 9, indicating that GS has a good capacity to adsorb BR46. In this sense, it is also possible to obtain similar results if operating under the conditions of group 3, with even a shorter contact time. In short, under different operating conditions, is feasible to reach a good BR46 removal using GS as unconventional adsorbent.

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ID 68

THE EFFECT OF POLYURETHANE-GRAPHENE NANOCOMPOSITE COATINGS ON THE PROPERTIES OF NATURAL FIBERS AS REINFORCING ELEMENTS

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ABSTRACT

This work demonstrates the improvement of the mechanical properties of long natural fibers upon impregnation with polyurethane/graphene nanocomposite coating. The coating also resulted in an improved fiber-lime matrix interaction leading to a significant enhancement in the composites bond behavior. The coating was prepared with pristine and functionalized graphene nanoplatelets employing mussel-inspired chemistry. The latter resulted in enhanced graphene-polyurethane interfaces.

INTRODUCTION

Natural fibers form a promising solution to substitute synthetic fibers for composite materials in structural engineering applications. However, reinforcing yarns prepared from natural fibers possess a specific structural behavior presented in inelastic evolution of tensile properties; up to 10-15% of the yarn's tensile strength the yarn shows a low Young´s modulus, then the constituent fibers start to align in the direction of the load promoting an increased Young´s modulus up to rupture [1]. The low initial stiffness as well as the weak mechanical interlocking of natural fibers in brittle matrices, i.e., lime or cement, result in insufficient design of composite materials for structural purposes [2] [3]. Therefore, in this work we present the effect of a pre-engineered nanocomposite coating on the improvement of the tensile properties and the bond behavior of long natural fibers. The coating is dip/dry-based and comprises of graphene nanoplatelets (GNPs) dispersed in water-based polyurethane in the presence of a dispersing agent (Polyethylene glycol) and a plasticizer (Glycerol). The coating was prepared with pristine and polydopamine-functionalized-GNPs. To prepare the coated yarns, first hemp yarns were washed with ethanol/acetone/hexane sequence then they were dipped in the coating suspension and then heat treated for 3hs at 80°C. A total of 10 yarns samples (coated and noncoated) were tested under unidirectional tensile test with displacement control (250 mm/min) according to EN ISO 2062-1995. The pullout mechanisms of 3cm-yarns (before and after coating) cast in lime mortar prisms was also tested using 6 samples each to characterize the bond behavior at yarn-matrix interfaces [4]. The matrix used in natural hydraulic lime with a 10MPa-compressive strength.

RESULTS AND CONCLUSIONS

Tensile tests results are shown in Fig.1a. The coated yarns presented significant improvements in both tensile strength and Young's modulus compared to their noncoated counterparts. The coated yarns scored 120 % higher tensile strength than that of the noncoated ones. Furthermore, unlike the noncoated samples, the stiffness of the coated yarns was unified during the tensile loading due to the restoration of the low initial stiffness (more than 4 times higher than the initial stiffness of the noncoated samples). This issue is significantly essential to guarantee a sufficient design of composite materials for strengthening systems in structural engineering application.

The results from the pullout tests are shown in Fig. 1b). For a bond length of 3 cm, the coated yarns demonstrated a significant enhancement of their bond behavior if compared to the noncoated ones. The pullout load was 6 times higher (170 N) than that of the noncoated yarns (27 N). These results highlight that the mechanical interlocking of the noncoated yarns is poor and the need for a surface treatment of the natural fibers is essential before their incorporation in brittle matrices.

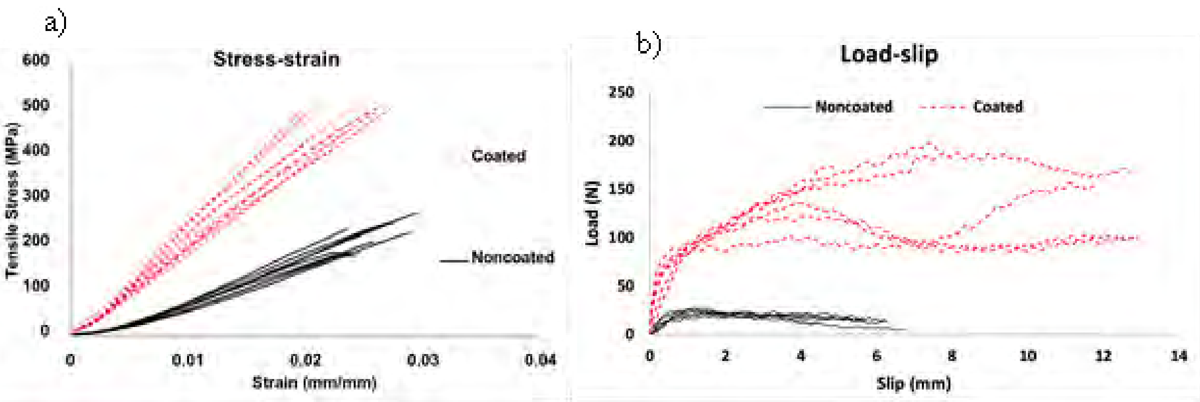

Fig.1. a) stress-strain curves and **b**) load-slip curves of coated and noncoated yarns

ACKNOWLEDGMENTS

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ID 69

A NEW APPROACH FOR SEMI-AUTOMATIC MEASUREMENT AND MORPHOLOGICAL ANALYSIS OF FIBRES THROUGHOUT THERMOPLASTIC COMPOUNDING PROCESS: A CASE STUDY ON GIANT REED FIBRES.

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ABSTRACT

This work describes a systematic method for the analysis of the defibrization of natural fibres during the compounding process by twin-screw extrusion. The variation of morphological features such as length, diameter and aspect ratio of natural fibres is analysed by affordable optical scanning methods and open-source software. The use of this technique for the measurement of giant reed fibres in PE and PLA-based composite materials has proved that there are no significant differences in the output fibre morphology of the compound, regardless the fibre feed sizes and extruder scale, using equivalent screw configuration and extrusion parameters.

INTRODUCTION

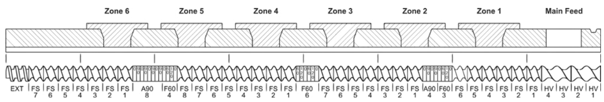
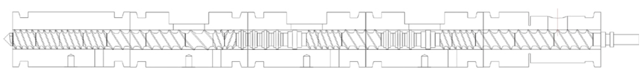
The morphology of the fibres used as reinforcement in the production of polymeric composite materials has a decisive impact on the mechanical properties and performance of such materials [1]. The physical degradation of lignocellulosic fibres during compounding process is affected by parameters such as the feed and shear rate, and the rotation speed and geometric configuration of the screws. Different fibre measurement methods ranging from timeconsuming manual techniques to fully automated systems, with auto-sample preparation, and image acquisition and analysis based on artificial intelligence have been documented in the literature [2]. As an alternative to rough hand measuring methods and to expensive automated equipment, a new approach based on affordable optical scanning methods and the open-source software ImageJ is proposed. This measurement method can provide a statistically substantial number of measurements at each sampling (thousands of data per sample) and has been tested for the study of giant reed (*Arundo donax* L.) fibre during processing.

RESULTS AND CONCLUSIONS

Lignocellulosic fibres obtained from the stems of giant reed plants as described by Suárez et al. [3] were used for compounding with HDPE (HD6081, Total) and PLA (Luminy® L105, Total Corbion) matrices. Multiple batches with fibre content up to 40% w/w were produced using two different extruders with the characteristics shown in Table 1.

Samples to measure the fibres present in the compounds were obtained by compression moulding of small amounts of material (≈1g) at each stage of the process: different extrusion screw sections, extruded filament, pelletisation and subsequent injection moulding. The samples were then scanned using a high-resolution flatbed scanner at 4800 dpi. For image analysis, Photoshop CS4 (Adobe), with its great potential for segmentation of large bitmap graphic files, was combined with the open-source software ImageJ for image processing and feature measurement.

Table 1. Twin-screw extruders used for compounding processing

Extruder model	D (mm)	L/D ratio	Screw configuration	Screw speed (rpm)
ThermoScientific Process 11	11	40:1		100
Collin ZK25	25	30:1		250

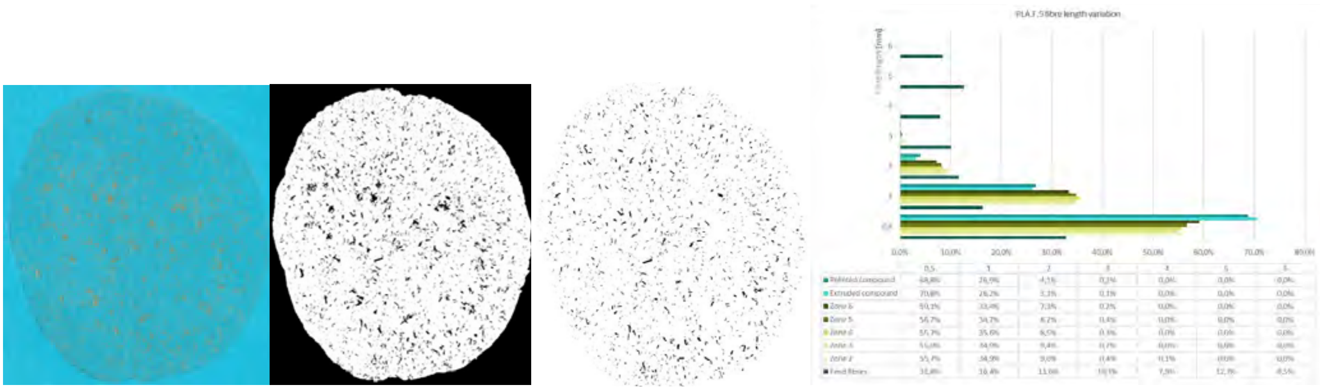


Figure 1. Scanned sample, image segmentation, features detection and fibre length distribution for PLA-5% fibres.

Having used two fibre feed sizes (3 and 5 mm) and two different extruders to produce compound with fibre contents of 5, 10, 20 and 40% by weight, it is observed that:

-The greatest reduction in fibre length occurs from the first kneading zone of the extrusion process, where more than 85% of the fibres for HDPE and 90% for PLA compound become shortened to less than 1 mm in length.

-The fibre size distribution (lengths and diameters) is the same at the end of the compounding process, regardless the size of the input fibres.

-The aspect ratio is reduced to less than 10 for all the fibres present in the final composites. This decrease is only slightly less drastic for the compounds produced with 25 mm diameter extruder screws and for low-load compounds produced with the smaller extrusion machine.

-The injection moulding process does not significantly affect the morphology of the fibres.

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ID 74

EFFECT OF FIQUE YARNS SURFACE MODIFICATION WITH TIO_2 AND REDUCE GRAPHENE OXIDE NANOPARTICLES ON THEIR PHYSICAL, THERMAL, AND MECHANICAL PROPERTIES

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ABSTRACT

This work analyses the effect of TiO_2 and reduce graphene oxide nanoparticles coatings on thermal, mechanical, and physical surface properties of fique fibers. A method of cross-sectional area determination based on optical micrographs and digital image processing is proposed for the tensile mechanical characterization of fique yarns. Six types of industrially manufactured yarns were analyzed. Tests showed changes in the hydrophilic behaviour of the fibers, as well as in their degradation temperature as a result of proposed coating. The mechanical tests, together with the statistical treatment of the obtained data, allowed determining the maximum tensile strength, elastic modulus, and percentage of axial deformation with dispersion values lower than those reported in the literature.

INTRODUCTION

Fique fibers are endemic to the Colombian Andean region and belong to the same specie as sisal and henequen fibers. These fibers are suitable for reinforcement in composite materials of polymeric and cementitious matrix. Like fibers of similar characteristics, their hydrophilicity and irregularity in the shape and size of their cross-section are disadvantages that have delayed their application at the industrial level. Tensile strength values of fique fibers have been reported between 50 and 625 MPa (Contreras, Hormaza, & Marañón, 2009; Delvasto, Toro, Perdomo, & de Gutiérrez, 2010), as well as fiber diameters between 50 and 300 MPa. μm (Andrade Teles et al., 2015; Gañán & Mondragon, 2004).

The present work proposed a coating method with TiO_2 nanoparticles and reduced graphene oxide (rGO) based on immersion cycles in an aqueous dispersion of the selected nanoparticles. A total of 240 individual tensile tests were performed, 40 for each type of yarn, and the data obtained were statistically treated to obtain their probability distribution. Contact angle tests were also performed using the sessile droplet method adapted to 5-micron microdroplets on fique yarns. TGA and SEM were also used to evaluate the results. Metallographic preparation of 16 sections for each type of yarn, encapsulated in epoxy resin, was carried out for optical micrographs. The images were processed, and the values of the section areas were obtained using the software Fiji (Schindelin et al., 2012).

RESULTS AND CONCLUSIONS

The result of the image segmentation appears in Figure 1. Samples analysis showed that the cross-sectional yarn area was between 0.156 and 0.194 mm^2, with coefficients of variation between 6 and 10 %. These values are equivalent to effective yarn diameters between 0.45 and 0.49 mm.

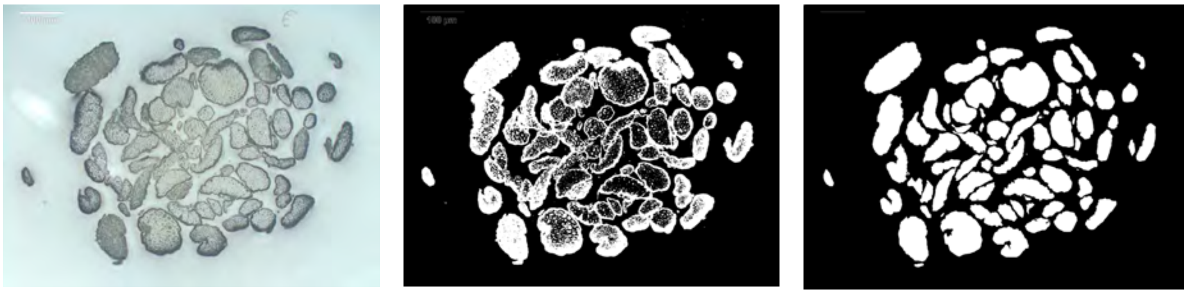

Fig.1 Fique yarns cross-section digital image treatment

Table 1 shows the values obtained for the maximum tensile strength of the fique yarns studied. According to the data obtained, the maximum strength of the fique yarns was between 758 and 997 MPa. On the other hand, the coefficients of variation for the fique yarns studied were between 19 and 30%; these values are lower than those reported in the literature.

Table 1. Tensile strength of fique yarns

Linear density		Twist degree	Maximum resistance	Standard deviation	Variation coefficient
N_m	Tex	Turns per inch	MPa		%
1.14	877.1	1.5	832.45	161.44	19.31
		2.0	781.37	159.42	20.40
		2.5	861.77	213.84	24.81
1.4	714.2	1.5	758.42	227.98	30.06
		2.0	942.35	185.99	19.74
		2.5	997.32	193.55	19.41

N_m: Metric number

It was observed that yarns with nanoparticle coatings (TiO_2 and rGO) showed a decrease in stiffness concerning pretreated yarns but with values similar to those of raw yarns; this behavior was also observed in the maximum resistance (Figure 2-a).

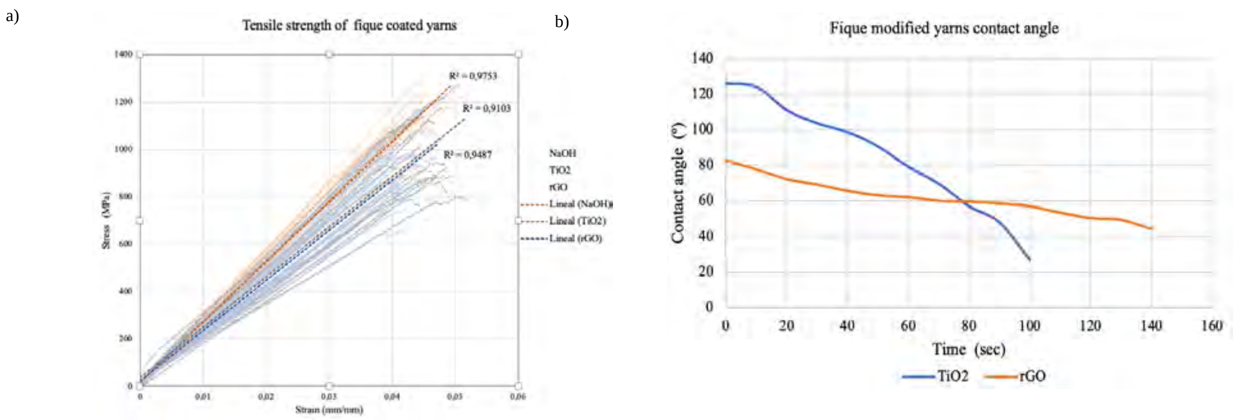

Figure 2. Tensile mechanical properties and contact angle of fique modified yarns

The axial deformation in this type of yarn increased concerning the raw and pretreated yarns. Furthermore, nanoparticle coating promotes a hydrophobic behavior in the yarns (Figure 2-b), as well as an increase in the thermal degradation temperature of the fibers.

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ID 75
EXPERIMENTAL EVALUATION OF BEARING CAPACITY AND SETTLEMENT REDUCTION OF SAND BED REINFORCED WITH SUSTAINABLE MATERIALS

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ABSTRACT
The difficulties faced on infrastructural development in a country due to the scarcity of structurally sound and stable earthen base can be resolved by means of suitable ground improvement methods. The present study explores the benefits of using rattan as an alternative to geosynthetic products in improving the soil strength and settlement characteristics of sand bed. Rattan is an eco-friendly, economical and easily available material that has the potential to enhance the strength and stiffness of soil. Series of plate load tests were conducted to understand the ultimate load bearing capacity of square footing resting on sand bed reinforced using rattan mats with and without apertures after considering its engineering and mechanical properties. The paper presents the effectiveness of closely woven rattan mats in sand after varying the parameters like embedment depth, size and number of reinforcement layers. The effect of apertures of varying size in rattan mats is also considered to analyse the variation of bearing capacity and settlement reduction of reinforced sand bed. The results obtained were compared with load settlement behaviour of unreinforced sand bed. Improvement in strength increased by 7.14 times and reduction in settlement of 88% was observed after placing four layers of reinforcement mats of width 3B at a top layer spacing and consecutive spacing between layers of 0.3 B.(were B is the width of footing)

INTRODUCTION
The rapid growth of infrastructures like buildings, railways, highways and other structures have forced the engineers in most of the countries to facilitate construction on weak soils by initiating the ground improvement methods that ideally suits the field conditions or designed requirements, to improve their bearing strength and settlement characteristics. Although several traditionally practiced ground improvement methods like soil replacement, soil densification through vibration and compaction were effective, scientists are always compelled to find more economical efficient and time saving method that satisfies the design requirements. Thus, among the conventionally practiced methods, soil reinforcement using different intrusions of sufficient tensile strength can be identified as an efficient method to improve the performance of weak soils.(Ouria, Mahmoudi, and Sadeghpour 2020; Wu and Pham 2013) The idea of enhancing the tensile ability of soil by means of human made and natural intrusions cannot be considered as a latest concept as they have been practiced since 1960s(Hegde and Sitharam 2013). During earlier times, steel and aluminum strips were incorporated as reinforcements in soils to enhance its physical and mechanical properties. Later the high cost and corroding properties of metallic strips led to the research and development of new sources of polymer reinforcement materials like geosynthetics that are readily available in the market.

The evolution of Geosynthetics in the field of geotechnical engineering has wholly changed the aspects of ground improvement for solving construction problems on weak soils(Abdelkader et al. 2018; Haeri, Noorzad, and Oskoorouchi 2000). Laboratory studies were conducted to understand the load settlement behaviour of geotextiles(Benmebarek, Djeridi, and Benmebarek 2020; Cicek, Guler, and Yetimoglu 2015; Kazi, Shukla, and Habibi 2016; Ouria, Mahmoudi, and Sadeghpour 2020) , Geogrid(Biswas, Krishna, and Dash 2013; B.; Dixit and Patil 2014; Xu, Liang, and Shen 2019; Mittal and Shukla 2020;Shrigondekar and Ullagaddi 2021) and Geocell (Makkar, Chandrakaran, and Sankar 2017) by varying the parameters like reinforcement spacing, width and number of reinforcement layers and concluded that bearing capacity of sand can further be enhanced with an appropriate reinforcement configuration. Stress concentration in weak soils was eliminated with a uniform distribution of applied footing load followed by the reduction in settlement with the incorporation of these planar reinforcements. Even though the emergence of geosynthetics have aided in an effortless strengthening of weak soils, the increasing cost, and its adverse effects on environment cannot be ignored easily. Thus, extensive researches are being carried out to overcome the detrimental effects of artificial polymers, which led to the research on natural fibers. The promising results achieved through natural fibers has encouraged the researchers to propose more innovative, effective and efficient natural fibers to serve the purpose of soil reinforcement (Deivanai and Arumairaj 2015, B. Das and Chetia 2022, Anggraini et al. 2017, Sayida, Evangeline, and Girish 2020). However, the performance of natural fibers is often underestimated due to the lack of detailed researches in the engineering field. Natural fibers being an affordable and easily available material in most developing countries can be effectively utilized to resolve the geotechnical problems that hinder the progression of infrastructure in such countries. Studies have demonstrated that natural fibers like coir, jute, sisal, kenaf, straw, and bamboo were also successful in increasing the tensile strength, shear strength and engineering properties of soil(Ahirwar and Mandal 2018; Hegde and Sitharam 2015; Lal, Sankar, and Chandrakaran 2017b; Panigrahi and Pradhan 2019; Sayida, Evangeline, and Girish 2020). Lal, Sankar, and Chandrakaran 2017a;Rajagopal 2009;Sridhar and Prathapkumar 2017;Vinod, Bhaskar, and Sreehari 2009 confirmed the application of coir geotextiles in soil bases caused a rapid improvement in bearing capacity. However, after conducting a comparative study on different types of natural fibers Shirazi et al. 2020 specified that bamboo grid reinforced soil performed superior to coir reinforced soil in terms of bearing capacity and settlement. Ahirwar and Mandal 2018; Akhil, Sankar, and Chandrakaran 2019; Hegde and Sitharam 2015 performed parametric studies on bamboo mats with and without openings and confirmed that strength improvement was dependent on the geometry and arrangement of reinforcement layers. Thus Bamboo mats have gained considerable attention in the recent years and numerous studies in this field have motivated us to focus on natural fibers which are much cheaper with tremendous growth potential.

Rattan being an eco-friendly and abundantly available material, are fast-growing plants that are indigenous to tropical regions of Asia, Africa, and Australia and there are about 600 species of rattan representing 13 genera mainly in Southeast Asia and the Pacific(Wahab et al. 2019). The ultimate tensile strengths, bond strength and Young's modulus of Rattan were reported by Adewuyi et al. 2015; Mahzuz et al. 2013, 2014 through several experimental investigations. Studies conducted by Adewuyi et al. 2015; Mahzuz et al. 2013, 2014 indicated that rattan exhibited comparable material properties to enhance the load bearing capacity of soil. Adewuyi et al. 2015 compared the flexural and tensile performance of bamboo and rattan reinforced concrete elements and satisfied the requirements for reinforcements in light weight RCC structures. Although, the interfacial bond strength between rattan and concrete were determined by researchers the scope of using rattan as an engineered soil reinforcement material is completely unaware due to the lack of researches. The studies reported so far did not consider the load settlement behaviour of sand reinforced with planar forms of reinforcement mats fabricated using rattan. In this context, it is attempted to conduct a detailed experimental study on the bearing capacity and settlement reduction of sand with rattan mats and the results so obtained is presented in this paper. Even though durability aspects of natural fibers are often doubted, ongoing researches in this field have shown that life span of natural fibers can be extended through several chemical treatments. Putri and Dewi 2020 stated that the degradation period of bamboo can be extended to a great extent, after proper preservation methods. Surface modification treatment of bamboo for preventing moisture adsorption using Copper chrome arsenic solution by (Hegde and Sitharam 2015, Ahirwar and Mandal 2018), bitumen coating by (Prasad 2010; Wei et al. 2018) have been effective in enhancing the durability of bamboo fibers. The results indicate the suitability of natural fibers for long term use in adverse conditions after suitable treatment methods.

In this research work, it is aimed to study the performance of sand beds strengthened with rattan in the form of planar reinforcement layers. The species of the rattan culms used in this research work is Calamus acanthospathus. The locally available rattan culms were shredded in to thin strips for fabricating rattan mats. The objective of the research is to investigate the potential of rattan culms in improving the bearing capacity and load settlement response of sand in the form of woven rattan mats through models tests at varying parameters. The paper discusses the strength improvement at varying depth of first layer, width, number of reinforcement mats and effect of using apertures of varying sizes.

RESULTS AND CONCLUSIONS

The results were encouraging enough to support the incorporation of rattan mats in the sand, as the experimental investigations exhibited a substantial improvement compared to unreinforced sand.. Following conclusions were incurred from the experimental investigations.

Enhancement in load settlement behavior was observed with the inclusion of multilayer reinforcements. Incorporating four layers of closely woven rattan mats resulted in an improvement factor of 7.14 and a settlement reduction of 88%. From the graph it is evident that incrementing the number of layers beyond 4 was found to be ineffective and impractical.

The incorporation of apertures into the closely woven rattan mats leads to better enhancement in strength and settlement when compared to the provision of closely woven rattan mats.

However, increasing the size of aperture mats beyond an optimum dimension, affected the overall performance of sand bed. After the inclusion of rattan mats of aperture sizes beyond 2*z* x 2*z*, the strength improvement and settlement reduction of sand bed reduced when compared to the sand bed reinforced with closely woven rattan.

Maximum improvement in strength ad settlement reduction was observed after the inclusion of rattan mats of aperture size 1*z* x 1*z*.

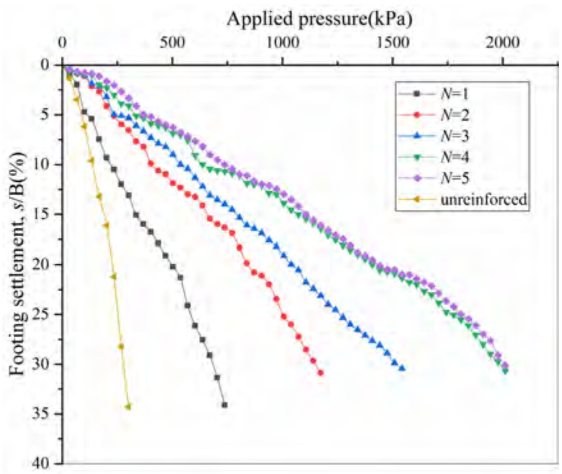

Fig.1 Applied pressure vs footing settlement for different number of reinforcement layers

The prime objective of the present research paper in understanding the reinforcing mechanism of rattan mats to improve the bearing capacity and settlement characteristics were satisfied through the series of plate load tests. From the results it can be inferred that there exists a wide potential for rattan fibers in substituting the synthetic fibres for improving the ground conditions and simulating the rural and economic growth of a country.

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ID 76
STANDARDISED SCHEME FOR SAMPLE PREPARATION TO ANALYSE NATURAL FIBRES AND RECYCLATES OF NATURAL FIBRE TEXTILES BY STATIC IMAGE

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ABSTRACT
The characterisation i.e. of natural fibres, nonwovens, textile recyclates etc. can be done by static image analysis. Although this is a versatile method, its usage is actually limited due to incomplete standardization (esp. in the area of sample preparation). Within the project StaPAFaB two research institutes are engaged to compile a reference manual listing typical classes of materials and optimised methods of sample preparation for each of the classes. This is combined with recommendations for reasonable parameters in image acquisition / processing and possible limitations for each type of material. Aim of the project is to enable reproducible and consistent analyses on an inter-laboratory level as well as to reduce the demand of time for the analyses. The article will focus on typical classes of natural fibre-based textile materials and adapted methods to enable their quick and reliable sample preparation.

INTRODUCTION
Static image analysis is an interesting option to analyse natural fibres, since it delivers distributions instead of average values for i.e. length, width and several shape factors. Unfortunately, up to now standardisation has taken place only incompletely for static image analysis. ISO 9276–1 to –6 [1] define parameters for evaluation and graphical presentation of the results. Image acquisition and calibration of the experimental setup are only specifically described by the equipment suppliers. However this does not cover the way how to prepare samples for the static image analysis.
This leads to problems in commercial and research laboratories: different ways of sample preparation, combined with various parameters in image acquisition and analysis accomplish strongly different results. At the worst there is neither reproducibility of results in one laboratory, nor comparable results between different laboratories. But this is not a general problem of static image analysis: it has been shown by inter-laboratorial round trials, that a well-described procedure can guarantee identical results within a small tolerance [2].
Within the project StaPAFaB two research institutes are engaged to publish a reference manual as a guideline for scientists as well as practitioners. This manual will comprise a list of typical classes of materials combined with well-documented optimised methods of sample preparation for each of those classes. This article presents results for two typical natural fibre-based sample materials to give a brief insight into the project aims.

RESULTS AND CONCLUSIONS

Two typical examples are given for an easy sample preparation: wet dispersion and a quick separation using compressed air for complex recyclate mixtures enabling at least access to essential data of material or fibre composition. Both are shown in Fig. 1.

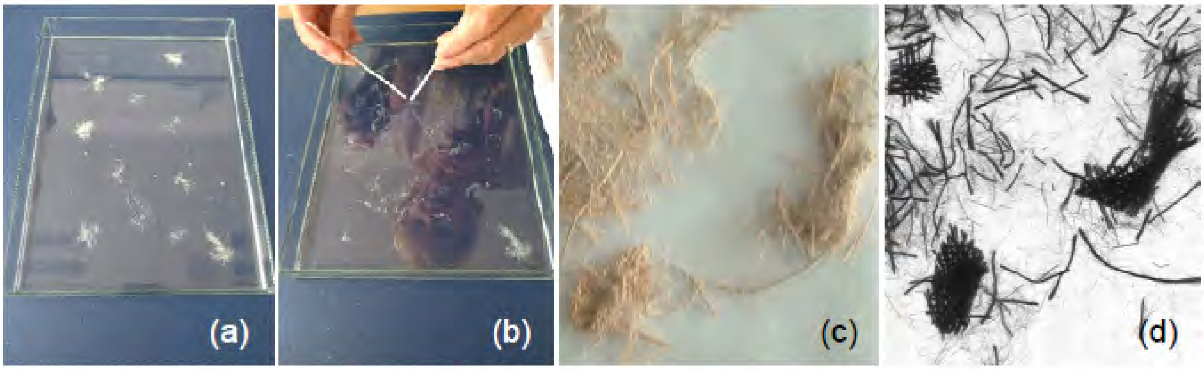

Fig.1 Natural fibres (a) immersed in water with surfactant, (b) after dispersion and jute carpet recyclate dispersed by compressed air (c) photo, (d) greyscale scan.

Agglomerated natural fibres can be dispersed in water with surfactant using plastic spatulas as shown in Fig. 1 (a) before and (b) after dispersion. This method is useful for fibres up to 20 mm to assess the length distribution. The glass trough used here can be placed directly on a flatbed scanner to acquire a transmission image.

The approach using compressed air to disperse material mixes is useful i.e. for textile recyclates like the jute carpet recyclate shown in Fig 1 (c) as photograph and (d) as transmission greyscale scan in 2400 dpi. In the scan fabric pieces appear black, yarn sections dark grey and single fibres light grey. From this type of image it is quickly possible to quantify the shares of these three textile fractions. In comparison, the time demand for an exact analysis by manual separation and weighing single fractions can be more than one day per sample.

Finally the reference manual, which is compiled during the project, will give an easy access to quick and reliable methods of preparation for a broad range of samples to be analysed by static image analysis.

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ID 83
EFFECT OF SODIUM LAURYL SULFATE (SLS) TREATMENT ON THE PROPERTIES OF RAMIE FIBERS

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ABSTRACT
An innovative treatment is applied for the regularisation of ramie fibres, namely a 5% sodium lauryl sulphate (SLS) solution was employed for the purpose. Chemical composition, physical properties, morphology, thermal degradation, and crystallinity were investigated.

INTRODUCTION
Ramie, one of the longest and strongest plant fibres, allows single fibre spinning, unusual for bast fibres, and retains its strength when wet [1]. However, because of its lower durability, it is typically combined with more resilient fibres like cotton or wool, after degumming, normally with sodium hydroxide (NaOH) [2]. Sodium lauryl sulphate (SLS) has been used for alternative treatment on some natural fibres [3]. This work proposes the use of SLS on ramie.

RESULTS AND CONCLUSIONS
Chemical composition of the treated fibres (Table 1) shows a very high cellulose content obtained by SLS application, though other studies on NaOH treatment on ramie reported up to around 90% cellulose [4]. The density obtained after treatment is still considerably lower than for other textile fibres, such as flax or hemp. Average fibres diameters (Figure 1) are mostly between 20 and 50 μm: this consistency is related to their effective degumming via SLS treatment, apparent from comparing the surfaces of untreated and treated fibres (Figure 2).

Table 1 Measured physical and compositional properties for SLS treated fibres (wt.%)

Cellulose (wt.%)	Hemicellulose (wt.%)	Lignin (wt.%)	Ash (wt. %)	Density (kg/m^3)
81.28	8.78	5.50	2.52	1.23

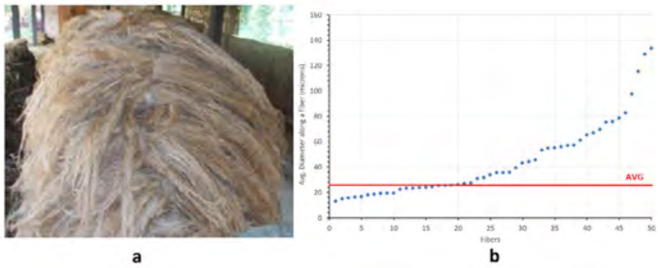

Fig.1 **(a)** Untreated ramie fibre; **(b)** Average diameter of the fibres along their length (50 fibres measured)

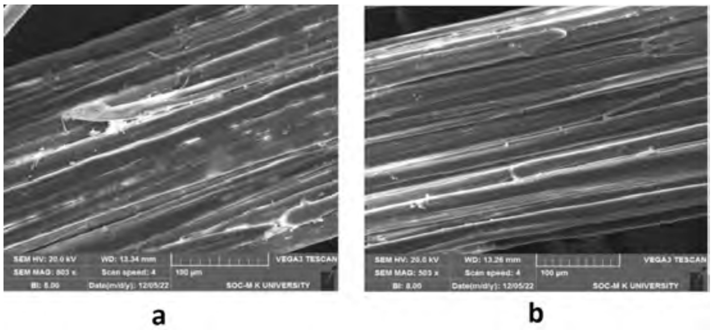

Fig. 2 SEM micrographs of fibre surfaces: **(a)** untreated; **(b)** SLS treated

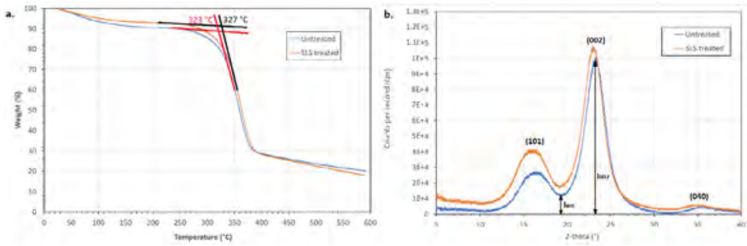

Fig.3 (a) TGA curves in nitrogen; **(b)** XRD spectra, of untreated and SLS treated fibres

Thermogravimetric analysis (TGA) (Figure 3a) suggests a degradation onset at close temperature for untreated and SLS treated fibres, determined as from method applied in [5]: the char residue at 600 °C was around 20% in both cases. X-ray diffraction (XRD) spectra (Figure 3b) did allow investigating crystallinity: the respective peaks, analysed as suggested in [6], indicated a slight increase of crystallinity in SLS treated fibres.

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ID 85
NEW INSIGHTS ON FLAX FIBER AGEING THROUGH THE STUDY OF ANCIENT EGYPTIAN TEXTILES

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ABSTRACT
Flax is one of the first domesticated plants, and its cultivation goes back to the ancient Egyptian period. At that time, flax fibers were already used due to their fineness and/or mechanical properties, for clothing, household linen, funerary uses such as mummy strips or funeral clothes, or even fishing nets and ropes. Today, the use of flax fibers for composite reinforcement or textile is strongly increasing, but their ageing behaviour remain a fundamental question. Through an original and transdisciplinary approach, this work focuses on the effects of ageing on the composition and mechanical performances of flax fibers from samples of archaeological interest. To this aim, fiber biochemical composition was investigated through synchrotron deep UV autofluorescence imaging and Raman spectroscopy, and results were correlated with mechanical properties evaluated by atomic force microscopy (AFM) in peak-force quantitative nano-mechanical property mapping mode (PF-QNM).

INTRODUCTION
Flax textiles were very valued in ancient Egypt, mostly due the comfort and strength provided by the fineness and high mechanical properties of flax fibers. Thus, flax was widely used for clothing as well as for fishing activities, but also for funerary purposes such as embalming through mummy strips, funerary clothes, or ornaments (Melelli, Shah, et al., 2021).
Despite high average mechanical performances (Baley & Bourmaud, 2014), flax fibers often exhibit some defects, known as “kink-bands”, being weak points for the fiber itself and more generally in the material made out of flax fibers (Melelli, Durand, et al., 2021). Thus, the effect of ageing on the characteristics of such defects, and to a greater extent on the performances of ancient flax fibers, is of interest for the present work.
Some pieces of Egyptian flax textiles, from Pharaonic and Roman periods, have survived for millennia up to the present day thanks to specific environmental conditions. The samples studied in this work come from Le Louvre Museum, Paris, France (E 27370 ; E 27376 ; AF 11260) and from Museo Egizio, Turini, Italy (TUR 2226); they were carefully selected for their archaeological contexts of great interest and for

the storage conditions they were subjected. Contemporary samples were also investigated and used as references for the study.

RESULTS AND CONCLUSIONS

Using Deep UV Fluorescence imaging, we observed a large difference between samples in terms of fluorescence intensity and emission wavelength, depending on the change in the fiber composition. This difference in the biochemical composition of flax fibers is also highlighted by spectra from Raman analysis, illustrating a change of the parietal composition through ageing, depending on the environmental conditions and duration.

In additional, Fig. 1 shows an example of results of the indentation modulus obtained by AFM PF-QNM on a flax fiber from an ancient Egyptian textile. If the average indentation modulus (17 GPa) of the fiber cell wall from the ancient fibers remains comparable to average modulus of contemporary fibers, the topography shows additional defects, such as cracks in the cell wall that can be observed on ancient fibers.

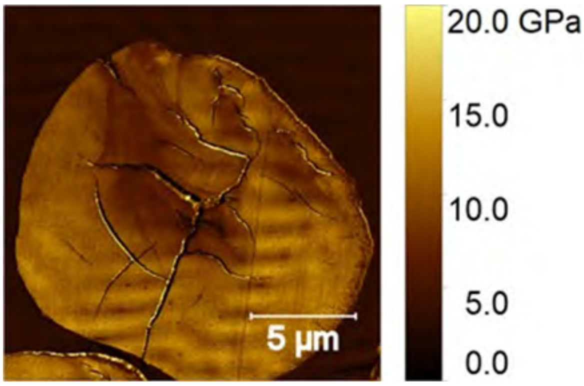

Fig. 1 Indentation modulus obtained by AFM PF-QNM show defects in the cell wall of an ancient flax fiber

Further analysis as well as complementary tests will be performed, such as micro-tomography and second harmonic generation (SHG) microscopy, to further investigate the effect of ageing on the composition, mechanical performances and ultrastructure of ancient Egyptian textiles from flax fibers.

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ID 87
ANALYSIS OF FIBERS OF INVASIVE PLANT SPECIES FROM THE MACARONESIA WITH POTENTIAL INDUSTRIAL APPLICATIONS

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ABSTRACT
In the scope of the Project Inv2Mac, which main goal is the potential use of biomass generated from invasive plant species of Macaronesia, several fibers samples of plants, namely, *Hedychium gardnerianum*, *Opuntia ficus indica*, *Opuntia dillenii*, *Pennisetum setaceum,* and *Acacia farnesiana* were studied. The fibrous components of the plants were analyzed through different characterization techniques, including scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and contact angle (CA), in order to evaluate their surface morphology and properties (roughness parameters, with 3D reconstruction), chemical composition/distribution and wettability. The analyses revealed samples composed mostly of carbon, oxygen, and nitrogen, of different hydrophilic nature (wettable surface), which can be potentially used to prepare composite materials for industrial applications with or without surface modifications.

INTRODUCTION
Invasive species are problematic and a real threat to any natural ecosystem since these spread without any control, creating an unbalance in all systems and available resources for the endemic species (Ortega, 2021). Thus, the biological, chemical and physical characterization of invasive plant natural fibers may be part of the solution for this environmental problem by incorporating them in composite materials, contributing to reducing the area affected by the propagation of these non-native species. The samples (fibrous parts of the plants, after extraction treatment) were provided in the dehydrated state from Gran Canaria (Canary Islands, Spain). Small portions of the samples were cut and placed onto the SEM stud pins, previously prepared with copper tape, cleaned with compressed air, and observed without any conductive layer deposition for imaging, 3D reconstruction, and elemental composition/distribution. For the contact angle, the samples were placed as flat as possible, aligned with the apparatus syringe, and the procedure for the contact angle measurement was performed using ultrapure water using the sessile drop method.

RESULTS AND CONCLUSIONS
In Fig 1, it is possible to observe different morphological structures characteristic of each mentioned plant. In the *H. gardnerianum* and *O. dillenii* (Fig 1a, 1c), it is shown axial fibrils and large longitudinal tracheid with pit apertures, while in *O. ficus indica* (Fig 1b) is visible periderm tissue (pourous hexagonal-like structures) surrounded by pit apertures tissue. In *P. setaceum*, (Fig 1d), the presence of silica cells is quite visible, a feature of plant silicon absorption as mono-silicic acid that is released to the soil by the weathering

of siliceous minerals (Kumar, 2017). *A. farnesiana* (Fig 1e) did not reveal any distinctive structures.

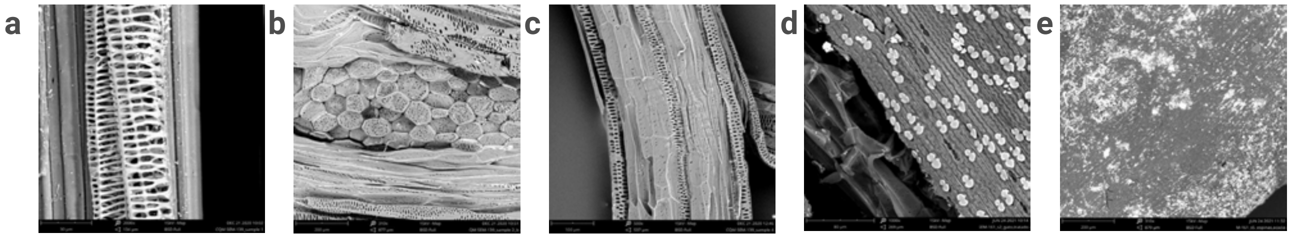

Fig.1 - SEM images of invasive plant samples. **a)** *Hedychium gardnerianum*, **b)** *Opuntia ficus indica*, **c)** *Opuntia dillenii*, **d)** *Pennisetum setaceum* **e)** *Acacia farnesiana*

In Table 1 is presented the elemental composition of the analyzed samples, which shown to be composed, in its majority, of carbon, and oxygen. It is also evident the presence of silicon in the sample *P. setaceum* (Fig 1d). On the other hand, the roughness and contact angle results show that the samples are hydrophilic.

Table 1 - Representative SEM images of the invasive plants studied

	a	b	c	d	e
Element composition	C: 65.53% O: 29.28% N: 3.94% K: 0.73%	C: 56.73% O: 38.05% N: 4.80% Ca: 0.10%	C: 61.24% O: 31.75% N: 6.64% Na: 0.12%	C: 55.29% O: 36.12% Si: 4.67% N: 3.18%	C: 76.16% O: 18.84% N: 3.62% Si: 0.59%
Roughness (µm)	Rz: 12.27 ± 5.34 Ra: 4.37 ± 0.38	Rz: 15.65±1.34 Ra: 2.99 ± 0.35	Rz: 9.69 ± 1.49 Ra: 2.39 ± 0.28	Rz: 6.80 ± 2.1 Ra: 1.7 ± 0.3	Rz: 7.90 ± 1.3 Ra: 1.7 ± 0.2
Contact angle (º)	68.46 ± 11.54	94.69 ± 9.62	86.03 ± 10.29	-	-

These preliminary results showed that the invasive plants present different hydrophilic natures, even after the extraction treatment, with the less hydrophilic ones potentially (*Opuntia dilleni*) being more suitable to form composites with plastic matrices than the more hydrophilic ones (*e.g. Hedychium gardnerianum*), possibly without the need of post-extraction chemical treatment. Its integration into polymeric materials and evaluation of its surface and mechanical properties is currently underway.

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ID 88
ACOUSTIC AND AIRFLOW RESISTANCE CHARACTERISITCS OF FIBRE-REINFORCED COMPOSITES MADE OF POLYLACTIC ACID (PLA) AND THE NATURAL FIBRES FLAX AND COTTON

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ABSTRACT
This work compares the airflow resistance and acoustic properties of composites with different mixing ratios of PLA and the natural fibres flax and cotton for the application in construction as lightweight structures, car door linings or seat pans. The composites are made from the binder fibre PLA and the bast fibres flax. The addition of cotton is intended to improve the acoustic effect (absorption) by increasing the number of air pockets (pores) and reducing their shape due to the fineness of the cotton. The airflow resistance and the acoustic parameters of samples with different mixing ratios were analysed and compared. The study shows that there is a dependency of the two parameters.

INTRODUCTION
Fibre-reinforced plastics made from degradable and renewable raw materials are increasingly replacing materials made from petrochemicals. Petroleum-based materials are dependent on the price of crude oil, are not renewable, and are mostly not biodegradable. Therefore, the structural shift from petroleum-based products to a bio-based industry is extremely relevant. In this work, industrially compostable composites based on renewable resources are being developed for the application in architecture as lightweight structures, car door linings or seat pans, with the aim of improving the acoustic properties through the use of cotton. The fibres that were used consists of renewable resources: the bioplastic polylactide is produced from lactic acid through a fermentation of sugar or starch. (Kayser, Averesch, 2015; RWTH,2004) The composite can be industrially composted without prior separation.
During the sound absorption, the sound energy that migrates through the component is converted into heat. The developed absorber leads to an improved acoustic by sound absorption through the interior of the component. This active principle is reinforced by an increased fineness of the fibres, whereby the use of cotton leads to improvements due to its properties: Cotton is finer than other natural fibres, which means that the overall weight can be reduced. The specimens used in this work are made from composite sheets fabricated with a two-step thermoforming process. In this process, needle punched nonwovens are treated under pressure and heat in a mould and then cooled under pressure. The basis weight of the samples was 800 g/m². More parameters of the composites are listed in the following table.

Table 1 Parameters of the composites

Materials	Mixing ratio			Consolidation parameters		
	PLA	Flax	Cotton	Temperature [°C]	Pressure [bar]	Time [s]
2	25	37.5	37.5	195	50	10
6	50	25	25			
10	75	12.5	12.5			

RESULTS AND CONCLUSIONS

The following figure shows the airflow resistance σ according to DIN EN ISO 9053-2:2021-02 of the materials with different PLA contents. The airflow resistance is approx. 18300 Pas/m² by a content of 25% PLA and more than doubles with a PLA content of 50%. If the PLA content increases further by 25%, the airflow resistance almost doubles.

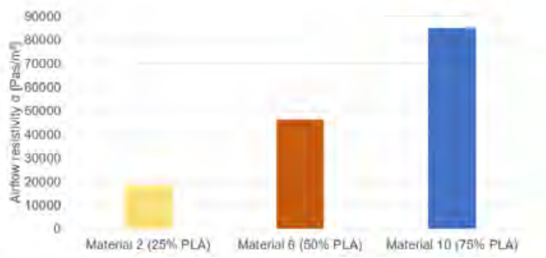

Fig. 1 Airflow resistivity of the materials 2,6 and 10

Figure 2 shows the absorption coefficient α of the materials 2, 6 and 10. Material 2 has a higher absorption coefficient, especially at high frequencies. The test was carried out according to DIN EN ISO 10534-2. The coefficient decreases with increasing PLA content, which is also due to the surface properties of the specimens with a high matrix content.

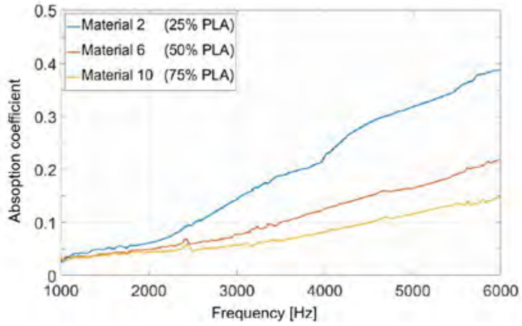

Fig. 2 Absorption coefficient of the materials 2, 6 and 10

This study shows that the absorption coefficient and the airflow resistivity rise in similar proportions with an increasing PLA content. This confirms the dependence of the two parameters.

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ID 89

CRYOGEL BASED ON CHITOSAN AND MICROCELLULOSE FOR HEXAVALENT CHROMIUM REMOVAL FROM CONTAMINATED WATER

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ABSTRACT

This work evaluates cryogels based on chitosan reinforced with microcellulose for Cr^{6+} sorption. The incorporation of microcellulose fibers contributes to increased mechanical properties with good interaction with chitosan. The highest Cr^{6+} sorption capacity was close to 1.4 $mmol.g^{-1}$, being related to the availability of amino groups from chitosan. Therefore, cryogels are efficient alternatives for the treatment of contaminated waters.

INTRODUCTION

Chromium is widely used in various industrial sectors (e.g., metallurgy, electroplating, textile, and leather). Due to its toxicity and persistence in the environment, there is serious concern about water contamination with chromium by industrial activity or improper disposal of waste (Peng, 2020). Cryogel based on chitosan and microcellulose is an efficient alternative for removing potentially toxic metal ions from the water, even at low concentrations, due to the high contact surface and functional groups available. In addition, the polysaccharides used have a biodegradable, biocompatible and high abundance character, favoring more environmentally friendly and affordable technologies (Chartier, 2022; Dragan and Dinu, 2020). In this work, cryogels were evaluated for Cr^{6+} sorption, varying the percentage of chitosan (C), microcellulose (M), and glutaraldehyde (G) in their formulations.

RESULTS AND CONCLUSIONS

The results from sorption tests are shown in Fig. 1. It is noted that solutions with an initial Cr^{6+} concentration of 0.3 mmol. L^{-1}, all formulations had excellent performance removing 100% of the ions in the aqueous medium. However, when evaluating the sorption capacity at higher concentrations, with the increase of the microcellulose and crosslinking agent content, there was a reduction in the capacity. The result is related to the availability of protonated amino groups of chitosan. The increase in crosslinking with glutaraldehyde and secondary bonds with microcellulose occupy active sites capable of interacting with chromium oxyanion. Through sorption isotherm analysis, it is clear that the cryogel developed has a highly favorable behavior for Cr^{6+} sorption. The Sips model was the one that obtained the best fit to the experimental data, indicating a heterogeneous surface, but with adsorption behavior in monolayer at high concentrations, possible by the preference in the interaction of Cr^{6+} with the amino groups (Al-Ghouti, 2020). The maximum sorption capacity observed for cryogels was close to 1.4 $mmol.g^{-1}$.

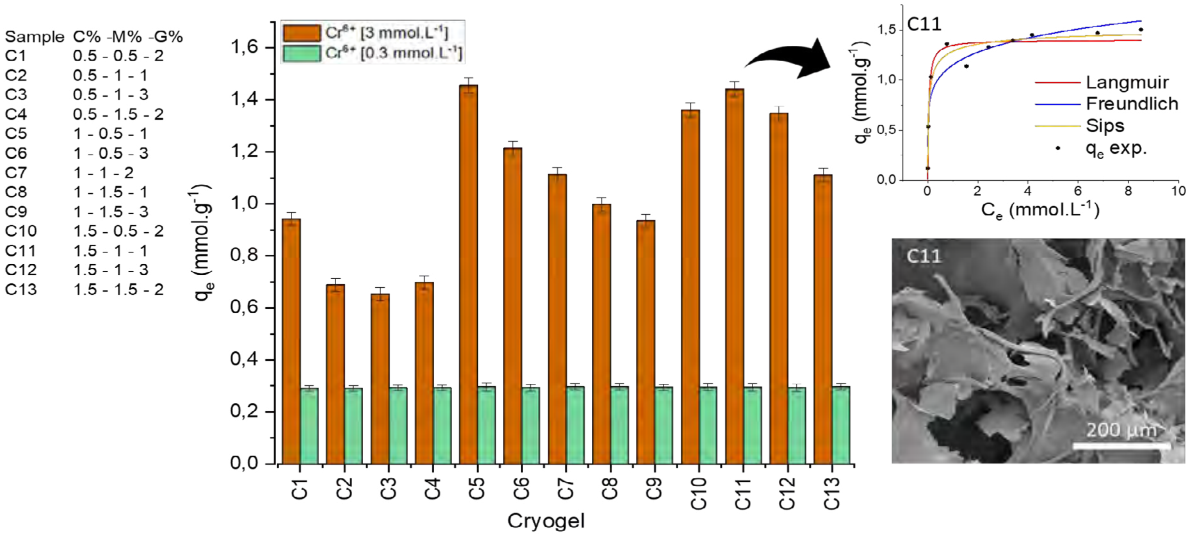

Fig.1 Sorption capacity (q_e) for cryogels based on chitosan and microcellulose at 25 °C and pH 4.0, Cr^{6+} sorption isotherm and micrography for C11 sample.

It is worth noting the importation of microcellulose for preserving the structure of the cryogels developed. The good compatibility between polysaccharides allowed the connection between chitosan sheets by microcellulose fibers, forming a three-dimensional structure with open and interconnected pores. In addition, compared to cryogels without microcellulose, there was an increase of 180 and 60% for the maximum stress supported by cryogels and 135 and 50% of the Young module, with the incorporation of 0.5 and 1.5% of microcellulose, respectively. This is a significant result because it favors the handling and reuse of cryogels in the treatment of water contaminated with metal ions.

ACKNOWLEDGMENTS

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ID 90
IMPACT OF NON-CELLULOSIC AND CELLULOSIC CELL WALL POLYMERS ON THE MECHANICAL PROPERTIES OF FLAX BUNDLES

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ABSTRACT
Fibre bundles are groups of elementary fibres glued together thanks to the middle lamella, and are the main fraction in plant fibre composites. In this study, relationship between the mechanical properties of flax fibre bundles, chemical composition and cellulose structure were investigated. To do so, a sequential biopolymer extraction was implemented. Fibre bundles were first depectinated by oxalate extraction, and then the hemicelluloses were extracted by LiCl/dimethyl sulfoxide (DMSO) and KOH. The oxalate extract consisted of homogalacturonans and type I rhamnogalacturonans, while the LiCl extract was composed mainly of glucomannans and the KOH extract of xyloglucans. The KOH stage resulted in the appearance of cellulose II in flax bundles. The extraction of pectin and hemicelluloses led to the disappearance of the middle lamella concomitant with a decrease in the tensile Young's modulus and maximum strength. Finally, the fibre bundle composition, ultrastructure and mechanical properties are discussed together in view of the thin middle lamella.

INTRODUCTION
Within the stem, the flax fibres are located in the phloem area and are present as bundles of several tens of single fibres (Akin, Gamble, Morrison, Rigsby, & Dodd, 1996). Fibres are individual cells characterized by an elongated polygonal shape, a reduced lumen volume and a thick cell wall. Their length can reach several centimetres with a diameter between ten and twenty microns (Pillin et al., 2011). At maturity, the plant cell wall of flax fibre consists, from the outside towards the inside, of the primary cell wall, secondary cell wall. Finally, in the middle of the fibre is a central cavity called the lumen (T. Gorshkova & C. Morvan, 2006), which can be irregular (E. Richely et al., 2021). The pectin-rich middle lamella ensures cohesion between fibres (Lazic, Janjic, Rijavec, & Kostic, 2017), but this thin layer is also considered as a weakness point that flax producers try to degrade and eliminate through retting, scutching and combing stages to provide composite reinforcements as individualised as possible.
If many studies have examined the mechanical properties of the secondary wall sublayers (Arnould, Siniscalco et al. 2017), few studies are available regarding the weakness points that glue strong individual fibres

together. If many studies have examined the mechanical properties of the secondary wall sublayers (O. Arnould, D. Siniscalco, A. Bourmaud, A. Le Duigou, & C. Baley, 2017), few studies are available regarding the weakness points that glue strong individual fibres together. The ambitious aim of this research work is to fill a gap in the knowledge of the relationships between the biochemical composition of bundles, polymer ultrastructure and mechanical properties on the scale of flax fibre bundles, including the middle lamella.

RESULTS AND CONCLUSIONS

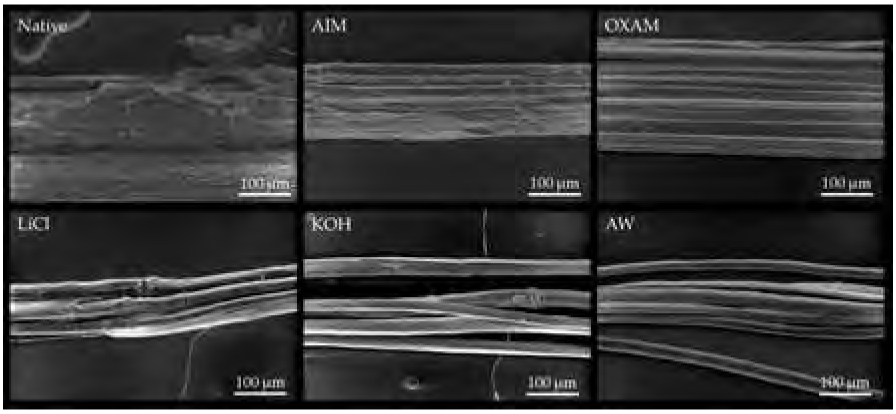

Fig.1 SEM images of each stage of the sequential extraction (×350)

SEM was applied to the flax fibres bundles to reveal changes induced by the different extraction stages (Figure 1). For native and AIM-treated bundles, the middle lamella is very clearly visible and surrounds the flax fibres within the bundles. As the sequential extraction progresses, the middle lamella is being less visible until it disappears completely. The fibre bundle is apparently still cohesive with a smoother outer surface, which effectively suggests that the middle lamella is only partially removed after pectin extraction by oxalate (OXAM). After the KOH stage, there was no longer trace of the middle lamella.

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ID 91
EXPERIMENTAL AND NUMERICAL APPROACH TO UNDERSTAND THE RELATIONSHIP BETWEEN THE STRUCTURAL FEATURE AND THE MECHANICAL PERFORMANCE OF FLAX FIBERS

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ABSTRACT
Flax fiber is a natural, eco-friendly material that is widely used in various industrial applications such as textiles, and composites. Understanding the relationship between the microstructure (lumen and other porosity) of flax fibers and their tensile properties is critical to improve the performance of these fibers for composite reinforcement. By integrating mechanical testing with X-ray microtomography experiments and finite-element modelling, the tensile properties of flax fiber at elementary and bundle scales are determined. The 3D-Finite Element models created using the images from X-ray microtomography are used to simulate the tensile characteristics of flax fibers. The Young's modulus calculated from numerical results are 39.5 ± 24 GPa for elementary fibers and 21 ± 12.2 GPa for bundle fibers. However, the robust hypothesis of this model disregards non-linearities. It is evident from the findings that length and porosity have an inverse relationship with the Young's modulus. The future prospect is to analyse the flax fibers at yarn scale which helps to better understand how tensile characteristics varies from elementary to yarn scales, as well as how inter and intra yarn porosities relate to a yarn's mechanical Properties.

INTRODUCTION
Flax fiber is a natural fiber obtained from the stem of a flax plant. It is known for its high strength, stiffness, and low density, making it a popular choice for use in various industrial and textile applications. The mechanical properties of flax fiber are influenced by several factors such as microstructure, chemical composition, and processing conditions. The lumen, which is the hollow core of the flax fiber, can also have an impact on mechanical properties. The presence of the lumen affect the strength and stiffness of the fiber, as well as its flexibility and elongation at break [1]. The mechanical properties of flax fibers are influenced by the lumen diameter, lumen wall thickness and the lumen percentage. The lumen percentage is the ratio of the lumen area to the total area of the fiber [2]. Overall, understanding the influence of the lumen on the mechanical properties of flax fibers is important for optimizing their use in various industrial applications.
The mechanical properties of flax fibers are generally studied at different scales. Into the stem, the el-

ementary fibers are glued together in bundles linked by a pectin-rich interphase, called middle lamella. Therefore, the mechanical properties of flax fibers are studied not only by measuring them on elementary fibers but also when fibers are still glued in bundles. In this work, we will discuss the tensile properties of flax fiber and the influence of the lumen on its tensile properties at elementary and bundle scale.

RESULTS AND CONCLUSIONS

The Young's modulus calculated numerically for elementary and bundle flax fibers are 39.5 ± 24 GPa and 21 ± 12.2 GPa which matches with the experimental values obtained by [3], however the considered model is linear elastic which does not include any plastic and viscous behaviour. The young's modulus as a function of porosity and length of the fiber is shown in Fig. 1. It can be observed that young's modulus is inversely proportional to porosity and length, confirmatory to experimental reported data. Numerical results for one fiber bundle are shown in Fig. 2.

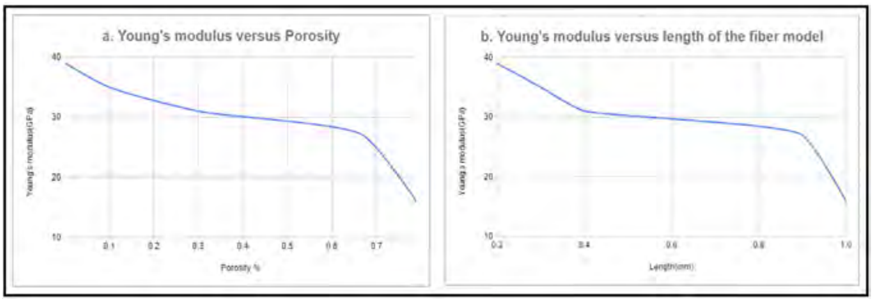

Fig.1 (a) Young's modulus as a function of porosity percent of the fibers **(b)** Young's modulus as a function of length of fiber model

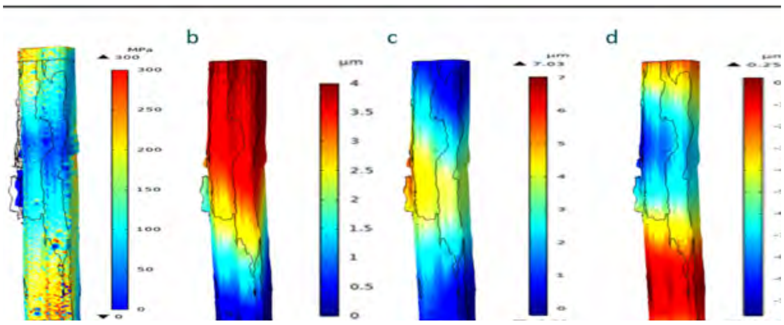

Fig.2 Numerical results of flax fiber bundle ((a) resulting axial stress component σ_{33} **(b)** displacement in tensile direction z **(c)** displacement in direction y **(d)** displacement in direction x)

This study demonstrates that the lumen influences the tensile characteristics of flax fiber. Further research at the yarn scale is needed to understand how tensile characteristics change from elementary fiber to bundle and to yarn.

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ID 92
NATURAL FIBER AND TEXTILE REINFORCED CEMENT COMPOSITES: WHERE WE ARE AND WHAT IS NEXT

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ABSTRACT
During the last ten years, in the framework of the research line on cement-based composites of the Textile Technology Research group (TECTEX), we have been carrying out research to develop new natural fiber- and textile-reinforced composite materials. In this sense, we have been performing experimental works on composite materials of a cementitious matrix and reinforced with fibers from different plants and wood —ranging from reinforcements such as nanofibrillated cellulose, through pulp, flock or continuous filaments— and with textile structures in the form of non-woven fabrics. In this paper, we summarize the main results obtained as well as which are the main challenges and research trends regarding the natural fiber- and textile-reinforced cement composites.

INTRODUCTION
In recent years, interest in the use of vegetable or cellulosic fibers —as an alternative to other reinforcements in composite materials— has grown considerably. Some of the advantages of these fibers compared to those commonly used are their availability, renewable nature, low cost, good thermal and acoustic insulation properties, and a specific resistance and modulus comparable to some synthetic fibers like glass fibers. These reinforcements led to cement-based materials with improved tenacity, flexural strength, and resistance to crack propagation. The improvements in these properties depend to a great extent on the fiber length —which can vary from the pulp (a few mm long); staple fibers (a few cm long); slivers (from around 20 to 100 cm)— and on how these fibers conform the reinforcement —in the form of fibers randomly distributed on the matrix, or in the form of textile structures like woven or nonwoven fabrics—. One of the main limitations of these systems continues to be the durability of cements reinforced with vegetable fibers when subjected to dry-wet aging cycles. To this end, different strategies have been developed, ranging from the modification of the matrix, to the modification of plant fibers through both chemical and physical processes. Our research group has been working since 2010 on this topic, carrying out numerous experimental works that have allowed us to acquire a comprehensive knowledge about these systems. In this paper we present the main results obtained so far, with the aim of serving as a starting point for future research.

RESULTS AND CONCLUSIONS

The first works done by our research group were focused on the preparation and characterization of fiber-reinforced cements with the reinforcement in the form of pulp, working both with softwood pulp and cotton linters. For these materials it was found that subjecting the fibers to a previous simple water treatment (hornification process) consisting on 5 cycles of wetting the fibers at room temperature followed by drying at 40ºC led to an improvement of the durability of the composites. This improvement on durability is mainly attributed to a better dimensional stability of the fibers in front of to changes in the humidity (Claramunt et al. 2010, 2011; Ardanuy et al. 2011). Subsequently, in addition to fibers in the form of pulp, the use of fibers in the form of nanofibrillated cellulose was studied, analyzing the effect of incorporating these fibers separately or in combination with conventional pulps. It was found a synergetic effect since the nanofibrillated fibers could act reinforcing the matrix and the microfibers after the first cracking of the matrix (Claramunt et al. 2015). Different strategies to improve the durability of materials were also studied like incorporating silica fume and autoclave curing (Ardanuy et al. 2012; Fernández-Carrasco et al. 2014; da Costa et al. 2019). In later years we continued working with staple fibers —fibers with greater length and suitable for processing with textile technologies—, with the aim of increasing the reinforcement capacity due to the greater length of the fibers. On the other hand, compounds that incorporated the fibers in the form of non-woven fabric were also developed. This allowed us to advance in the knowledge of the preparation of optimized textile structures so that the cement could penetrate through the structure (Ardanuy et al. 2015b) and in the manufacturing processes and characterization of the compounds. Moreover, a previous treatment with plasma was studied as strategy to improve the durability of the composites (Ventura et al. 2016). As was expected, the nonwoven-reinforced composites led to flexural and tensile strengths much higher than those values found for composites reinforced with fibers in the form of pulp (Claramunt et al. 2016, 2017). In addition, we studied new strategies for improving the durability such as the selection of alternative cement matrix and/or additives and/or using different curing systems (e.g. autoclave, CO_2 rich atmosphere) (Claramunt et al. 2018, 2019; Ramírez et al. 2020) or by treating the fibers with natural oils González-López et al. 2020).

In all these studies it can be verified how natural fibers —both in the form of fibers dispersed in the matrix and in the form of textile structures— constitute an optimal reinforcement to improve tenacity, flexural strength and resistance to crack propagation, being possible to tune-up the reinforcement capacity by using the reinforcement in different forms. Moreover, they have appropriate thermal and fire resistance (González-López et al. 2021). From all these studies we could conclude that, with small variations, the reinforcement capacity was mainly driven by the fiber form (length and thickness) and how these fibers conform the reinforcement (randomly dispersed or in the form of nonwoven fabrics). It is to say, the results in terms of mechanical performance, will be quite similar independently if the pulp comes from a eucalyptus tree or pine tree, or if the staple or slivers used are from flax, hemp, fique or other bast fibers, among others. In our opinion, it is not necessary to go further in this regard. The main problem and limitation of these cement composites reinforced with vegetable fibers is their degradation when subjected to accelerated aging. Therefore, the future work should be focused in this direction. If the durability problem is solved, these materials could transfer to the market in the form of construction products for multiple applications such as façade or sandwich panels, paving and thin or sandwich tiles for false ceilings or covers, or plates for reinforcing masonry and ceramic structures, among others.

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ID 97

TOWARD ADHESIVES-FREE BIO-BASED COMPOSITES VIA UVASSISTED INTERFACIAL CROSS-LINKING

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ABSTRACT

In the present research, industrially developed continuous regenerated cellulose fibres and polyethene (potentially of bio-based origin) are used to manufacture fibre-reinforced composites by introducing an intermediate step of UV irradiation of semi-finished products. This approach assumes that a cross-linked transition layer can be formed at the interface between the UV transparent matrix and UV adsorbing fibres, thus avoiding petrochemical adhesion promotors. The influence of the UV exposure and the exposure time on the interfacial and interlaminar shear strength (IFSS and ILSS) of viscose fibres or fabrics embedded in a PE matrix has been characterised by single fibre fragmentation and double-notched tensile test, respectively. The performed mechanical tests confirm the potential of the proposed fabrication toward high-performance adhesives-free bio-based composites.

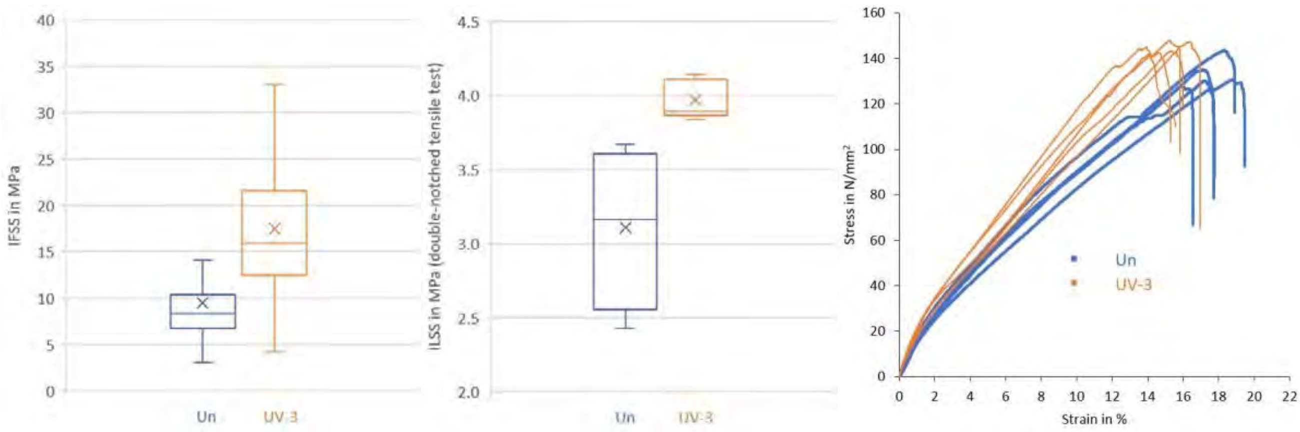
INTRODUCTION

In bio-based composites, the incompatibility issues between the hydrophilic cellulose reinforcing fibres and hydrophobic polyolefin matrix affect the fibre/matrix adhesion and decrease the composite performance. In the past, extensive studies have been carried out to improve fibre/matrix adhesion by modifying the fibre surfaces (Eichhorn et al., 2001) or adding additives (Mechraoui et al., 2007). These modifiers and additives are usually petrochemicalbased and can be problematic during processing and disposal. Despite optimising the fibre/matrix adhesion in composites, there is often a sharp transition in stiffness at the thin fibre/matrix interface. Therefore, achieving gradient transition of mechanical properties, i.e., tensile, shear and elastic properties, is also essential, along with optimising the compatibility between the fibre surface and the matrix.

In this research, a total of three different test series have been performed to investigate the influence of the UV-irradiation on the fibre/matrix adhesion, i.e., single fibre fragmentation test (SFFT), double-notched tensile test, and tensile test. All these tests were performed on two different samples, i.e., Untreated (Un) and treated samples (UV-3), where the semi-finished composite samples were exposed to a broadband UV lamp for 3 minutes on each side of the samples, with a main emission band from 250 to 300 nm (UVACUBE 2000, Dr Hönle, Germany).

RESULTS AND CONCLUSIONS

The single fibre fragmentation test, double-notch tensile test, and tensile test results show that UV exposure and exposure time positively affect fibre/matrix adhesion. An explanation of the observed phenomenon is seen in a mechanism proposed in a paper by Bahners et al. (Bahners & Gutmann 2012), where model composites made of PET fabric and PE matrix were UV irradiated with a mercury broadband lamp. It resulted in the formation of cross-linking at the PE/PET interface through the penetration of photons via the transparent PE matrix. Müssig et al. (2018) investigated the influence of photo-polymerised thin layers of 1 % and 5 % PETA on the fibre/matrix adhesion of cellulose/PP composites. However, in this research, the 3-minute UV-irradiates samples resulted in a similar effect as that of 1 % PETA on the fibre/matrix adhesion in cellulose/polyolefin composites. Given this background, the performed experiments confirmed the influence of UV radiation on the fibre/matrix adhesion of cellulose fibres in polyethene matrix without adhesives.



(a) (b) (c)

Fig. 1: Comparison of IFSS from the single fibre fragmentation test **(a)** and ILSS from the double-notch tensile test **(b)**, and stress-strain behaviour **(c)** of cellulose fibre/PE composites between untreated (un) and 3 minutes UVirradiated simples (UV-3)

ACKNOWLEDGMENTS

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EFFECT OF WEAVE STRUCTURE ON THE AUXETICITY AND COMFORT PROPERTIES OF 2D WOVEN STRUCTURES

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ABSTRACT

The demand for performance qualities connected to human health and sporting activities, including as compression, stretch and recovery, moisture management, water and air permeability, and thermal extensibility, has increased the potential for multi-functional textile products. Consumers are more interested in extensible features when buying clothing items like pants, t-shirts, and maternity belts. Using elastic yarn is one of the traditional methods for introducing extensibility, stretch, and recovery qualities into the fabric. Because conventional mono-stretch fabrics do not provide the needed extensibility, and the creation of bi-stretch fabrics takes more time and machine settings. The development of auxetic woven materials with dual-directional stretchability is one potential remedy for these issues. In the current study five different 2D woven auxetic structures were developed (with stiff and elastic weft separately) and their auxeticity and other mechanical features (tensile, tear and stretch & growth) were examined. All of the created auxetic woven constructions increased the wearer's overall extensibility by displaying a negative Poisson's ratio of up to -1. The tensile strength and tear strength of these constructions were up to 470 N and 72 N, respectively. Comparing stretchability of elastic fabric samples to stiff fabric samples, elastic fabric samples have a higher stretchability of up to 80%. The results showed that novel auxetic structures showed sufficient auxeticity even without elastic yarn.

INTRODUCTION

These are different classes of materials including rigid and stretchable materials. Stretchable materials can be further categorized as auxetic and non-auxetic materials. Auxetic materials are somehow different materials as compared to conventional materials (Alderson & Alderson, 2007; Hu & Zulifqar, 2020; Tobergte & Curtis, 2013) These materials exhibit a negative Poisson's ratio (NPR)(Ho et al., 2016; Ravirala et al., 2006) When the Auxetic materials are stretched in the axial direction they will expand in the lateral direction. In the recent few years, the use of auxetic textiles and development of auxetic fabrics and materials have attracted much attention (V V et al., 2022). Different researchers have worked on 2D woven auxetic geometries but as per the author's knowledge, very little research reported in the field of hybrid 2D woven auxetic textiles. In this research, five novel 2D woven auxetic designs (Pique plain weave (S1), Pique Twill weave (S2), Huckaback weave (S3), Bedford cord weave (BFC) (S4), and Mockleno weave (S5)) were used to develop fabrics having stretchability in both directions (warp and weft) for sportswear and activewear. All the fabrics were characterized by negative Poisson's ratio, tensile strength, tear strength, and stretch and recovery behavior. One-way ANOVA statistical analysis was performed to analyze the statistical significance of different results.

RESULTS AND CONCLUSIONS

It was observed that the yarn type, weave structure, and float length have a significant impact on the physical and mechanical properties, as well as the auxetic behavior of the woven fabric. S5 (Mockleno weave) showed the highest auxetic behavior with the lowest negative Poisson's ratio of -1. It was also noted that to achieve the auxeticity in the final product, there should be an optimized ratio of longer and shorter float lengths. S1 (Pique plain woven fabric) showed the highest tensile strength as compared to other woven structures. Tear strength depends upon the float length of the weave structure, if the float is higher, the number of yarns under that float will behave like a bunch and provide strength against tear force. Hence, S5 (Mockleno weave) showed the highest tear strength due to the longest float in the weave design. Rigid woven structures showed higher stretch and growth mostly in the warp direction as compared to the weft direction. When the weft yarn was changed from rigid to elastic yarn, most of the woven structures showed a remarkable increase in stretch and growth in the weft direction. The results of one-way ANOVA statistical analysis showed that weave structure and material type have a significant effect on the NPR and mechanical properties of developed samples because the p-value is less than 0.05 for each test result.

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ID 99
ECOFRIENDLY FLAX AND FLAX/BASALT HYBRID COMPOSITES PRODUCED WITH A LOW MOLECULAR WEIGHT BIOPOLYAMIDE 11 FOR AUTOMOTIVE APPLICATIONS

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ABSTRACT
The automotive sector put many efforts in increasing polymer composites eco-friendliness exploiting constituents from renewable resources. In this attempt, the present work investigated a bio-based PA11 matrix reinforced with a flax and an intraply flax/basalt hybrid woven fabric assessing quasi-static and dynamic performance through 3-point bending and low-velocity impact tests considering the effects of stacking sequence and fiber orientation. The effect of temperature on the impact response was also addressed. Hybrid composites outperform significantly flax ones thus ensuring satisfying mechanical properties while preserving composite lightness and partial biodegradability. Among the two hybrid configurations available, i.e., [0/0] and [0/90], the [0/90] ensures a good bending stiffness and an improved impact resistance with a reduced permanent indentation.

INTRODUCTION
The design of eco friendlier components is the new challenge of most industrial fields to counteract pollution and waste disposal issues. National and international regulations endorsed greener policies aimed at exploiting materials from renewable resources, discouraging the use of landfills and promoting a circular economy perspective oriented towards materials reuse and recycling (Shogren, 2019).
Many efforts were made by the automotive sector which massively exploits polymer composites to reduce vehicles weight and fuel consumption. The main attempts focused on the replacement of traditional glass fibers with vegetable ones, to reduce composite carbon footprint and to achieve a partial biodegradability at the end of the life cycle (Boland, 2016).
Despite specific tensile properties comparable with glass, vegetable fibers are not able to ensure the same mechanical performance when embedded in the polymeric matrices due to their hydrophilic nature which leads to a poor interfacial adhesion with the hydrophobic matrix. The will to achieve bio-based composites competitive with glass ones endorsed the research of another natural solution, and a suitable one was found in basalt characterized by mechanical properties comparable with glass and by a greener production process where no chemicals are required and energy consumption is reduced (Fiore, 2015). Despite satisfactory mechanical properties, basalt fibers are not biodegradable therefore hybridization is the best

solution to exploit the points of strength of both natural fiber types. The challenge against environmental pollution can be also faced from the polymer matrix perspective by selecting biodegradable polymers or polymers synthetized from natural resources (Mohanty, 2002).

In this framework, the present work assessed the 3-point bending and impact properties of a low molecular weight bio-polyamide 11 synthesized from renewable resources and reinforced with flax and intraply flax/basalt hybrid woven fabrics. Impact resistance is a key factor in automotive therefore all composites were studied considering the effect of impact energy, i.e., 10, 20 and 30 J, and temperature, i.e., -40 °C, room temperature and +45 °C. The experimental campaign was supported by a profilometric post-impact analysis and by thermal analysis, i.e., DMA and DSC.

RESULTS AND CONCLUSIONS

DSC revealed a double melting peak for the bio-polyamide 11 Besno Rilsan® at around 180 °C and 190 °C. An important conclusion can be drawn on the manufacturing process, in fact the higher melting temperature at around 190 °C ensures a good processability of the matrix already at 200 °C thus preventing the degradation of the thermal sensitive vegetable fibers.

Flax composites are characterized by lower flexural performance than hybrids due to the intrinsic higher mechanical properties of basalt thus confirming hybridization as a suitable solution to achieve satisfying mechanical properties while preserving lightness and partial biodegradability. Among hybrids, [0/0] composites displayed the highest and the lowest flexural properties when tested along basalt and flax directions, respectively. The [0/90] hybrid configuration featured intermediate quasi-static performance with a bending stiffness closer to the [0/0] upper limit.

Even in dynamic conditions, hybrids outperformed significantly flax which is characterized by the lowest maximum force, the highest maximum displacement, i.e., the lowest linear stiffness, and the highest amount of absorbed energy, i.e., a higher damage degree. Among hybrids, the [0/90] configuration showed a better impact response due to the lower anisotropy which allows basalt fibers to bear the impact load along two perpendicular directions thus delaying flax fibers overloading and breakage. This is confirmed by the profilometric analysis where [0/90] hybrid displayed the lowest permanent indentation. Concerning the effect of temperature, the lower impact properties of flax are confirmed being the only one to undergo perforation at -40 °C and 20 J. Hybrids disclosed a progressive softening of the matrix from 40 °C to +45 °C which caused a reduction in material stiffness due to a progressive approach of PA11 glass transition temperature. The latter is also responsible for the involvement of a wider area of the sample allowing to dissipate a higher amount of energy while preventing damage localization.

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ID 100

EFFECT OF MICROFIBRILLATED CELLULOSE (MFC) ON THE MIGRATION OF BIOBASED PLASTICIZERS IN POLY (LACTIC ACID)(PLA) BIOCOMPOSITES.

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ABSTRACT

This work aims to analytically investigate the effect of the micro-fibrillated cellulose (MFC) in regulating plasticizer migration when added in plasticized PLA-biocomposites, by employing the Fick's Second Law. The diffusion coefficient (D), will be linked to variations in the mechanical and thermal properties of cast extruded MFC-bio-composites over time, validating the MFCs addition predicted benefits gathered over a 50-day period, as performant industrial production approach.

INTRODUCTION

Poly(lactic acid) (PLA) has caught the attention of bio-composites industry and scientific research owing to its thermal and mechanical properties. However, its main drawbacks related to the inherent significant brittleness hinders its employment in industrial packaging applications. Nevertheless, PLA brittleness is commonly overcome by adding low-to-medium molecular weight plasticizers, still bearing in mind their possible over time migration.

Different approaches may be adopted to limit the plasticizer migration and one of the most promising is the micro/nano filler addition thanks to the formation of tortuosity paths that may limits the phenomenon. Among different fillers, cellulose is particularly appealing also because of its well-known contribution to enhance the barrier and mechanical properties of biobased composites.

In this context, the present research aims to investigate the plasticizer migration ability of cast extruded PLA films reinforced with two commercially available micro-fibrillated celluloses (MFCs) and two biobased plasticizers (polyethylene glycol (PEG) and lactic acid oligomer (OLA)), used as dispersants.

Thermal, mechanical, and migration tests are performed on flat-die cast extrusion bio-composites to evaluate the potential plasticizer migrations over a period of up to 50 days by estimating the diffusion coefficient of Fick's second law and evaluating the MFC effect in reducing the plasticizer migration.

RESULTS AND CONCLUSIONS

The adoption of biobased plasticizers as dispersing aids resulted fundamental to obtain an improvement of the final bio-composites mechanical properties with an easy scale-up from laboratory to industrial scale. The rheological characterization carried pout in this work was fundamental to set the flat die cast extrusion to produce films. Subsequently, on the films obtained, migration tests were carried out to evaluate the short- and long- term film performances. Interesting was the calculation of the diffusion coefficient, analytically evaluated applying the second Fick's law. The exponential decay trends of the diffusion coefficients over time are reported in Fig. 1. It can be noticed that a significant mass loss occurred in a short period (about 72 h). Compared to the formulations in which MFC are not present, it can be observed the positive effect of MFC in limiting the plasticizer migration. Migration outcomes were also associated with variations in the bio-composites mechanical properties (elongation and stress at break) and crystallinity changes, detected by Differential Scanning Calorimetry (DSC). Indeed, the sudden increase in PLA crystallinity that occurs in the first three days, ascribed to a drop in the elongation at break and to an increase in the stress at break, is what mostly contributes to the plasticizer mass loss. Moreover, MFC presence inhibits the diffusion process stabilizing the mechanical properties along time despite of the crystallinity increment.

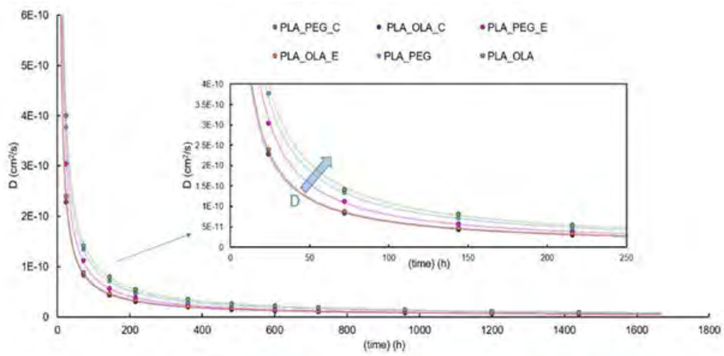

Fig.1 Trend of the diffusion coefficients over the time

The study proves how the approach adopted can be valuable, in the industrial production practices, to achieve a modelling procedure that can predict and optimize the behavior of bio-composites, in the perspective of biobased and biodegradable manufacturing as well performant as possible.

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ID 101
INFLUENCE OF ADDITIVE MANUFACTURING PROCESS PARAMETERS ON DYNAMIC MECHANICAL BEHAVIOUR OF FLAX FIBRE REINFORCED POLYLACTIC ACID POLYMERS

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ABSTRACT
This work investigated the influences of fused filament fabrication (FFF) process parameters on dynamic mechanical behaviour and microstructure of flax fibre reinforced polylactic acid polymers. For this purpose, a commercial filament for FFF 3D printing was selected. Specimens were manufactured by varying several process parameters. The quality of the printed specimen was assessed by characterising the microstructure (density measurements) and the dynamical mechanical behaviour (DMA). The interaction between the process parameters, the mechanical behaviour and the microstructure was analysed using experimental design in order to obtain statistically relevant observations. It was showed that the key parameters to control printed specimen quality is the nozzle temperature. Cold crystallisations of flax reinforced PLA printed specimens occurred.

INTRODUCTION
Additive Manufacturing has become a promising and innovative processing technique to manufacture complex geometry with increase production efficiency and cost saving (Wang, 2017) (Cheng, 2023). Additive Manufacturing of polymeric structures has gained a lot of interest as shown by the number of reviews available in literature. Among all the technologies, fused filament fabrication (FFF) and continuous fibre coextrution (CFC) are the most studied. The FFF is used to manufacture short fibre reinforced polymer composites. However, the main issue of FFF is the defects appearance, such as porosities, due to extrusion. These porosities caused by poor interfacial bonding limit the mechanical properties at higher fibre contents (Aydın, 2011). In order to improve the mechanical properties of FFF printed components, continuous fibres are increasingly introduced into FFF processing. The approach is referred as continuous fibre coextrution (CFC) (Pizzorni, 2022). Among different matrices, Poly (Lactic Acid), also known as polylactide (PLA), is a common filament feedstock material for 3D printing because of its renewability and biodegradability.
The main objective of the research project is to optimize long flax fibre to manufacture loadbearing structures via additive manufacturing. The presented work is a first step to explore the interactions between the 3D printing parameters, the dynamic mechanical behaviour and the microstructure of the material. For this purpose, the evolution of the behaviour and the microstructure in accordance to the most statistically rele-

vant process parameters is proposed. The influence of the printing parameters on the quality of the printed specimens is assessed by measuring the apparent density. A correlation between the process parameters and the dynamic behaviour is then established.

RESULTS AND CONCLUSIONS

Among all parameters that govern the 3D printing process (slicing strategy, line path, temperature etc.), the paper explores the effect of parameters that control the thermal history of composite materials. Indeed, the nozzle temperature, the printing speed and the build plate temperature are expected to influence the physical structure of polymeric materials and therefore their microstructure and dynamic mechanical behaviour. The other parameters were selected according to preliminary tests to ensure the printability of specimens. In a first approach, the quality of printed parts is evaluated by measuring the apparent density. A decrease of density reflects an increase of defects such as porosities. The experiments were conducted several times in order to ensure that the results are not due to intrinsic variability of vegetal fibres. The results in Fig.1 shows that the main factor that statistically govern the quality of printed specimens is the nozzle temperature.

DMA analyses shows that a cold crystallisation of PLA matrix occurred. The cooling time during printing is too fast to allow polymers to crystallize in a stabilized structure.

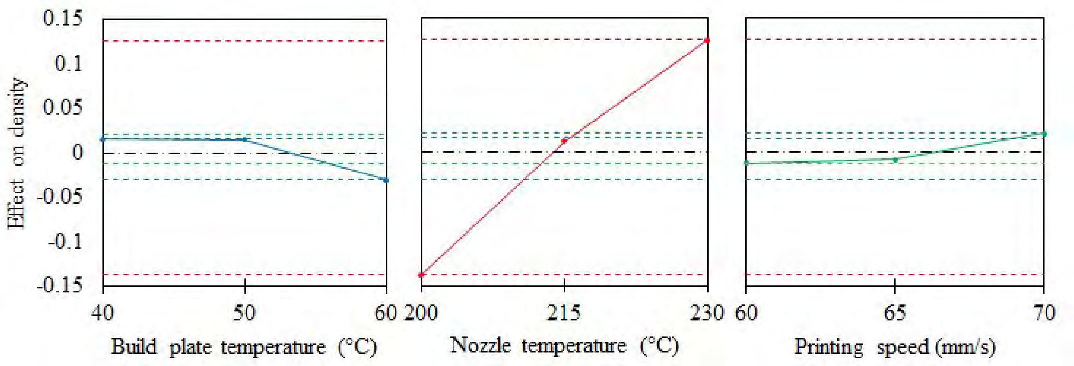

Fig.1 Influence of printing parameters on Flax/PLA composite density

Deeper analyses will be discussed in the presentation, to highlight the influence of parameters coupling on apparent density of printed specimens. Additional points will be also discussed such as the interaction between process parameters and dynamic mechanical behaviour.

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ID 103
STATIC AND FATIGUE PROPERTIES OF NATURAL FIBRE COMPOSITES UNDER HYGROSCOPIC CONDITIONS

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ABSTRACT
An experimental investigation was performed to study the effect of humidity on the static and dynamic mechanical properties (fatigue) of non-pre-dried flax/polyester biocomposites. A better understanding of the relationship between environmental factors, particularly humidity, and mechanical properties allows to assess the long-term durability of these materials. This work aims to provide a better insight into the effect of humidity, evaluated at 50%, and 85%(RH), on the evolution of the tensile and fatigue properties of flax/polyester biocomposites. Results show that the reduction in tensile modulus at high humidity is remarkably lower than usually reported in literature. Furthermore, the results of the fatigue test under high humidity conditions demonstrate that the modulus of natural fibre composites during the dynamic loading increases compared to the normal humidity condition.

INTRODUCTION
In recent years, the use of natural fibres especially flax fibres as a suitable alternative to glass fibre in fiber reinforced polymer (FRP) has been more attractive. Lightness and a lower environmental impact are two crucial factors which can distinguish Natural Fibre Composites (NFCs) from the synthetic fibre reinforced polymers (Lu, 2019). Among various types of natural fibres, bast fibres, especially flax, are attractive and used in industry due to their high specific stiffness(Azwa, 2013). But their applications have been limited due to the nature of these materials. Although, the effect of humidity and water absorption on the mechanical properties of NFCs have been investigated by many researchers, few researchers have embedded non-dried fibre in their composites. Lu et al. found that the durability of these materials increases by making composites without drying the fibres (Lu & Van Vuure, 2019). Hence, in order to have a better understanding of the effect of humidity on the NFCs with non-dried fibre, a biocomposite with unidirectional flax fibre and partially biobased polyester resin was prepared using the vacuum-assisted resin infusion (VARI) technique with a theoretical fibre volume fraction of 35%-40%. Then, tensile and fatigue tests were done at 50% and 85%RH.

RESULTS AND CONCLUSIONS
Figure 1 depicts the tensile and fatigue properties of Unidirectional NFCs. Shown in Figure 1A, the higher tensile strength of NFCs at 85% RH in comparison with standard condition could be due to a plasticization effect. While on the other hand a 14% reduction in tensile modulus due to the increasing humidity is observed, which is much lower than generally reported in literature (Réquilé 2019). Furthermore, as shown in Figure 1-B, the modulus of NFCs during fatigue divided by the modulus in the first cycle (Ed/E1) enhances by 5% and 8% at 50% RH and 85% RH respectively, at 1 million cycles.

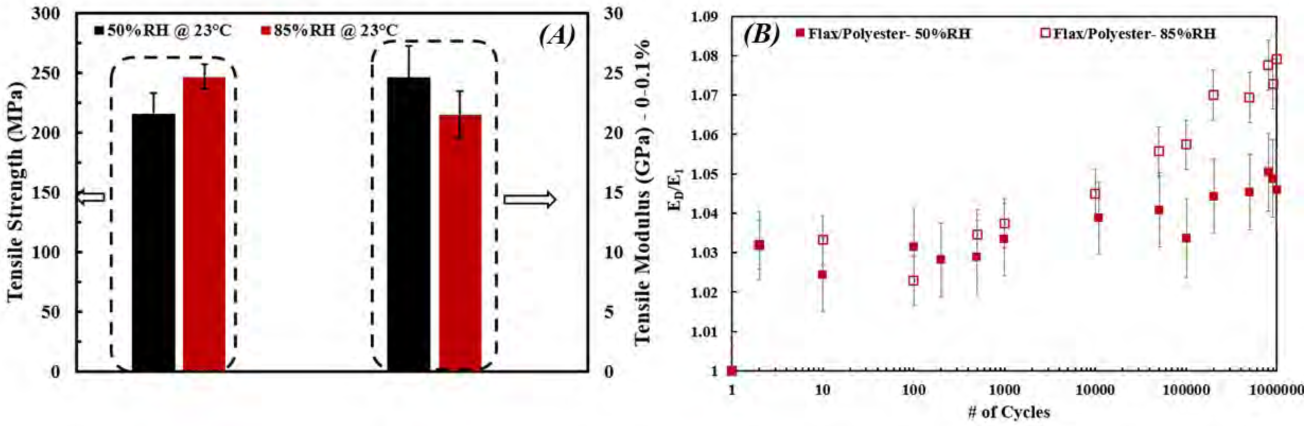

Fig.1 (A) Tensile properties of NFCs at different relative humidity conditions **(B)** Ed/E1 of NFCs at different relative humidity conditions

Moreover as seen in Figures 2-A and 2-B, the residual strain as well as energy dissipation in each cycle during fatigue at 85% RH are greater than at standard conditions. Higher energy dissipation and residual strain at higher humidity conditions suggest more plastic deformation and sliding of polymer chains over each other. Alternatively, they might represent more crack initiation (Bensadoun, 2016), and then fatigue life could be shorter at 85% RH in comparison with 50% RH. This is currently further evaluated.

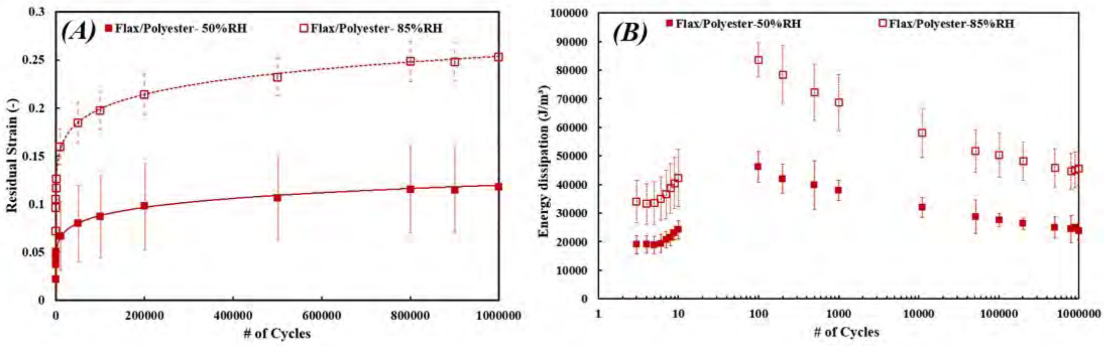

Fig.2 (A) Residual strain of NFCs **(B)**Energy dissipation of NFCs at different relative humidity conditions.

ACKNOWLEDGMENTS

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ID 104
EFFECT OF BANANA PSEUDOSTEM AND ANNATTO SHELL FILLERS ON CATALYTIC AND THERMAL PROPERTIES OF IMMOBILIZED MANNANASE ON BIOBASED COMPOSITE FOAMS

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ABSTRACT
A commercial mannanase was immobilized on a biobased epoxy composite foam (ECF) support reinforced with annatto shell (AS) and banana pseudostem (BP) fibers. The enzyme was immobilized in these matrices with moderate reductions in their activity with respect to the free enzyme, being the AS filled foam system the one that presented the best catalytic performance. According to the infrared and thermogravimetric analyses, the strength of the bond between the enzyme and the ECF support is affected by the type of filler without affecting the thermal stability of the catalytic system.

INTRODUCTION
The immobilization of enzymes in lignocellulosic materials has been proposed for chemical, pharmaceutical, biotechnological, food and environmental applications (Rodríguez-Restrepo & Orrego, 2020). Even though, the low mechanical resistance and the separation difficulties for the reuse of the catalytic system are the major reasons inhibiting their widespread adoption. However, when lignocellulosic materials are used as fillers for ECF of partially or totally biological origin, a porous matrix is obtained that is light and offers good chemical and mechanical resistance (Pradeep et al., 2022)
In this study, mannanase was immobilized on a foamed epoxy resin support (cubic shape pellets) reinforced with annatto shell (AS), and banana pseudostem (BP) dried powders. Biobased ECF were prepared using the method developed by (Pradeep et al., 2022) and enzyme was immobilized according (Rodríguez et al., 2018). AS and BP were incorporated at 5 wt% in each case, in order to study its effect on protein load, catalytic activity and activity retention. For the characterization of the catalytic system FT-IR measurements and Thermogravimetric Analysis (TGA) were performed.

RESULTS AND CONCLUSIONS
Table 1 shows the catalytic properties of the free enzyme and the immobilized enzyme systems. It is highlighted that the immobilization was satisfactory since the typical reduction of the activity with respect to the free enzyme was moderate, being the matrix foamed with AS the one that presented the best performance. The operational stability of both catalytic systems was also good (data not shown).

Table 1. Inmobilization of Rohalase® in bioepoxy foam with fibers from agricultural waste

Method	Immobilized protein (mg/g carrier)	Activity (U/mg protein)	Activity retention (%)
Free enzyme		0.65±0.09	
ECF+ BP	30.62±2.04	0.29±0.02	46.72±1.60
ECF+AS	30.46±2.18	0.48±0.04	77.62±3.40

± standar desviation (n=3). U= amount of enzyme that can produce 1 μmol of reducing sugars (mannose base) per min

Rohalase® is an enzyme product that contains mannanase as main activity.

Figure 1 presents the variation of the absorptivity bands measured by FT-IR of the pellets of the composite material without and with enzyme. When comparing the spectrum of the ECF+BP pellet with the ECF+BP+ Rohalase® pellet (Figure 2-A), a decrease in signal intensity is observed at 3410 and 2920 cm-1, which is associated with a decrease in hydroxyl bond vibrations and C-H stretching of CH2 and CH aromatic and aliphatic (Theophile, 2012). On the other hand, there are an increase in signals at 1515, 1250, 1183, 1042 and 838 cm-1 values that respectively correspond to C-C stretching vibrations in aromatic, C-O bond stretching, asymmetric C-O-C stretching, and C-O-C stretching of oxirane group (Theophile, 2012). This is explained by the formation of covalent bonds between the hydroxyl groups of the fiber and the aromatic or aliphatic carbon chains of the compounds present in the pellet. In addition, when contrasting the spectra of the ECF+AS and ECF+AS+Rohalase® pellets (Figure 2-B), the results are similar, with less noticeable decreases in the intensity of the 3400 and 2920 cm-1 bands but with an increase in signals at 1042, 830 and 776 cm-1 , which corresponds to symmetric C-O stretching, C-O-C stretching of oxirane group amines and NH wag (Muthukumar et al., 2022). This seems to indicate that the strength of the bond between the enzyme and the fiber is affected by the type of fiber used as the filler of the pellet.

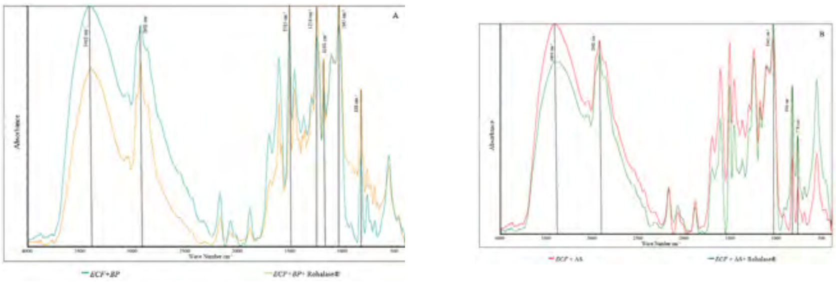

Fig.1 FTIR Spectra for ECF pellets with Rohalase® and different fibers

The results of the thermogravimetric analyzes (data not shown) do not show significant differences for the temperatures at which the maximum rate of weight loss occurred regardless of the type of fiber (365±1 °C). In conclusion, the use of different types of lignocellulosic fiber can alter the activity of the immobilized enzyme without affecting the thermal stability of the catalytic system.

ACKNOWLEDGMENTS

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ID 105
BIODEGRADATION OF FLAX FIBRES: IMAGING TECHNIQUES FOR AN INTERDISCIPLINARY STUDY

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ABSTRACT
Nowadays, flax fibres are studied and used in biobased composites and clothing, but they were also largely used in the past and textiles are often found as archaeological finds and supports in artworks and paintings. A point in common between modern and ancient materials is the biodegradation of flax fibres due to fungi and bacteria that can occur over time. In this work, several imaging techniques, such as second harmonic imaging microscopy (SHG) and atomic force microscopy (AFM PF-QNM), are used to better understand the effects of biodegradation and their impact on the cell walls and the fibre structure. A palette of naturally aged flax fibres extracted from artworks or archaeological fabrics, and raw modern fibres or coupled with PLA in a biocomposite were investigated. This cross-disciplinary approach brings to light the common problem of flax fibre durability in different fields and emphasizes the advantage of imaging techniques to understand the consequences of biofilm on the fibre structure and mechanical properties.

INTRODUCTION
Like other types of plant fibres, such as hemp or palm, flax fibres can be used as reinforcement when coupled with resins or mortars. Unfortunately, cellulose fibres are highly hygroscopic, and the water absorption favours a suitable environment for the action of microorganisms, which mainly use the cell walls as a source of food. The growth of biofilm is also a problem in the cultural heritage field that wants to prevent biodegradation to preserve textiles at their original state.

Up to now, fungi and bacteria have been identified and studied on different wet and dry wood species (Schmidt & Liese, 1994) but less is known about the effects of biofilm on the cell walls of bast fibres (Crawford et al., 2017; Elamin et al., 2018). Imaging techniques, like AFM PF-QNM and SHG, can help us to explain changes in mechanical behaviour and biochemical composition obtained by other more common characterization techniques, such as tensile testing or FTIR-ATR.

In the present study, a complementary approach of imaging techniques, like AFM PF-QNM and second harmonic imaging microscopy, are used to better understand how fungi and bacteria interact with the flax fibre cells.

RESULTS AND CONCLUSIONS

In Fig. 1 the degradation process of modern flax fibres naturally biodegraded on soil (a) and fibres extracted from an Italian painting on canvas (b) are compared.

Fungi and bacteria can attack the surface of the fibres, but some species are also able to grow throughout the lumen. SHG provides information on the state of the crystalline cellulose and on the general preservation of the fibre ultrastructure (Fig. 1a, b), which appears strongly compromised due to porosities, especially around the lumen (G layer). AFM, on the other hand, has the advantage of providing information on local mechanical properties; in Fig. 1c few fibres from the same yarn extracted from the ancient canvas are illustrated and it is possible to observe the biodegradation near the lumen. Although a new pore is generated in the G layer (see the detail of Fig. 1c), the whole area near it is affected and the mechanical properties drop until to reach 11 GPa. The rest of the fibre has however preserved an indentation modulus with a mean of 18 GPa.

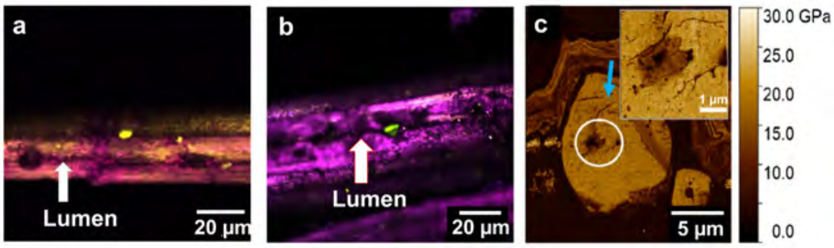

Fig.1 a) SHG microscopic imaging of modern flax fibres naturally degraded in soil. The white arrow highlights the lumen affected by microorganisms. **b)** SHG microscopic imaging of fibres extracted from a flax yarn of a painting on canvas dated 18th century. The fibre is degraded starting from the lumen. **c)** Map of stiffness acquired by AFM PF-QNM. The white circle highlights the detail in top right and the blue arrow underlines fractures.

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ID 106
DESIGN FOR SUSTAINABILITY AND INDUSTRIAL HEMP APPLIED TO TEXTILES IN BRAZIL

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Textile material is the second most-damaging product category to the environment (Lawson et al., 2022), and Design is committed to sustainability in the textile industry through recycling, reducing, reusing, and developing materials (GFA & BCG, 2017; TED, 2018). In this context, the interest for natural fibers has increased because they are less hostile raw materials to the environment during production, use and disposal of garments. In 2019, 30% of the fibers produced in the world were natural (Zimniewska, 2022).

An example of a natural fiber with a promising environmental performance is Industrial Hemp for its: (a) quick growth in the planting process; (b) lower consumption of water and harmful inputs to the environment during fiber processing stage; (c) higher resistance to traction, abrasion and UV rays during the use of textile product; (d) biodegradability if disposed of properly (Modefica, 2022).

There are records of the use of hemp in different times and places, but as it is extracted from the Cannabis sativa L. plant's stem, the textile fiber faced production restrictions since the 20th century in a significant part of the West (Westerhuis, 2016; Modefica, 2022). To change national legislations in this matter and start a new economic cycle for this fiber, it was necessary to recognize Industrial Hemp and psychoactive substances derived from the same plant species, but from different botanical variants (Freire et al., 2021). So, it is gradually being reintroduced into the productive chain. A little more than 30 countries produce hemp fibers, but on a limited scale, representing 0.05% of the textile fibers produced in the world, in 2019 (Westerhuis, 2016; Zimniewska, 2022).

While China and France seek to play a leading role in growing markets (Lawson et al., 2022), the textile production with Industrial Hemp in Brazil is incipient. Brazilian regulation is ambiguous, once is not allowed to plant any type of Cannabis sativa L. in the country since 1932, although the textile industry can use imported inputs (Modefica, 2022).

The Brazilian potential both in agribusiness and in the textile industry to explore industrial hemp as a natural and intrinsically sustainable fiber must be highlighted. For example, Brazil is fourth largest cotton producer and second top exporter in the world (Aragão & Contini, 2021). Also, it is the largest and most complete production chain in the West, lying among the major knitwear and jeans producers, as well as a global reference for swimwear and jeans fashion design (Abit, 2023).

Therefore, the objective of this study was to investigate the existing relations in the triad "design for sustainability, Industrial Hemp, textiles" based on bibliographical and documentary research, from a multidisciplinary vision and a strategic design approach.

The study is structured in two sections. The first one is focused on the properties of industrial hemp in textiles (including comparisons with cotton), sustainable development goals, and design strategies oriented to sustainability and circular economy (ONU, 2015; GFA & BCG, 2017; TED, 2018). Moreover, the second section investigates industrial hemp textiles in Brazil. In this case, the analytical phase of the Three-dimensional Strategic Design Matrix (Bergmann & Magalhães, 2022) is applied. Technologies, cultural principles, and consumption goods are addressed in three contexts: (a) Past and neglected initiatives, (b) Present and limited applications, (c) Future and perspectives for innovation.

The research results illustrate the following:
● The existence of favorable conditions for industrial hemp in textiles in response to contemporary environmental challenges. However, only its usage as a natural fiber will not ensure alignment with the circular reasoning. Even less if this is only the reflection of an ephemeral fashion movement.
● The convergence of design strategies for sustainability and the process of textile product development with industrial hemp. This fiber stands out from a holistic approach for innovation and choice of materials that challenge obsolete production and consumption patterns.
● The recognition of the technological potential of the Brazilian production chain to incorporate industrial hemp competitively. This requires systemic efforts committed to the responsible use of Cannabis sativa L. derivatives, which includes not disregarding the cultural resistance to the use of controversial materials.

Additionally, to reconcile economic development and environmental Sustainability, this study allows us to understand that all textile chain links must adapt to changes resulting from innovations, from the rescue of old traditions, or both together.

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ID 107
PHYSICAL AND THERMAL CHARACTERIZATION OF CASHEW NUT SHELLS (ANACARDIUM OCCIDENTALE L.) FROM VICHADA DEPARTMENT IN COLOMBIA

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ABSTRACT
This work presents the physical and thermal characterization of cashew nut shells (CNS) from the Vichada Department in Colombia. The influence of the type of pre-processing (roasted and boiled) on nut shelling was studied. Physical characterization showed that the pre-processing type affected CNS's bulk density, porosity, and color. Thermal analysis displayed that CNS degradation has three steps, starting above 130°C, 180°C, and 220°C for CNS-B, CNS-R, and defatted CNS, respectively. CNS's properties showed that this natural fiber may be used to develop green materials.

INTRODUCTION
Cashew trees (*Anacardium occidentale* L.) can be found in Colombia's eastern plains, especially in the Vichada Department. Cashew production by-products include cashew nut shell liquid (CNSL), cashew nut testa, and cashew nut shells (CNS), which are usually wasted. Among these, CNS represents approximately 50% of the complete nut after extracting the CNSL, which is burned or simply left on the ground (Orduz-Rodríguez & Rodríguez-Polanco, 2022). However, the use of CNS as a filler in composites can be an alternative for this waste, but its applications have been limited mainly to CNS ash (Jannat et al., 2021), although its lignocellulosic structure is suitable for the development of natural fiber composites.
Hence, this work presents the physical and thermal characterization of CNS samples from the Vichada Department as a first step in developing new environmentally friendly products such as green composites. The influence of pre-processing type: roasted CNS (CNS-R) and boiled CNS (CNS-B), on nut shelling was studied. Moisture content (ASAE S410.3); CIELab color (ASTM D7856); bulk and true density, and porosity (Kilanko et al., 2018) were also measured. Finally, thermal performance was evaluated by TGA analysis (ASTM E1131).

RESULTS AND CONCLUSIONS
The results of the physical characterization are shown in Table 1. The moisture content and true density

were not significantly different between CNS-R and CNS-B because the pre-processing process changes CNSL content, but it does not affect the lignocellulosic composition and nature. The CNS-B's bulk density was higher than CNS-R owing to CNSL presence; thus, the porosity value of CNS-B was lower. In addition, CIELab color parameters (L*: lightness, a*: green-red, and b*: blue-yellow) also showed variations after pre-processing. Also, Fig. 1 displays the thermal behavior of CNS from the pre-processing stage and CNS after CNSL extraction: defatted CNS-R (CNS-RD) and defatted CNS-B (CNS-BD). The preprocessing type led to dissimilar behaviors in the degradation peaks between CNS-R and CNSB due to the different amounts of CNSL in the samples. CNS-B curves suggest the presence of carboxylated CNSL because an early loss step was observed at 130°C, in contrast to 180°C in CNS-R. On the other hand, defatted samples have similar performance regardless of the preprocessing type, and the first loss step is observed at approximately 220°C, a temperature similar to that of other natural fibers. Therefore, defatted CNS fibers can be used to process new green composites at a higher temperature range, in contrast to CNS-R/B.

Table 1. Physical properties.

Sample	Moisture content [%]	True density [g cm^{-3}]	Bulk density [g cm^{-3}]	Porosity [%]	L*	a*	b*	Color
CNS-R	6.212 ± 0.713	0.640 ± 0.022	0.234 ± 0.004	63.347 ± 0.684	38.05 ± 3.99	10.60 ± 1.80	13.53 ± 3.21	
CNS-B	8.280 ± 1.308	0.685 ± 0.040	0.355 ± 0.004	48.201 ± 0.630	32.31 ± 3.80	5.95 ± 1.79	6.50 ± 3.06	

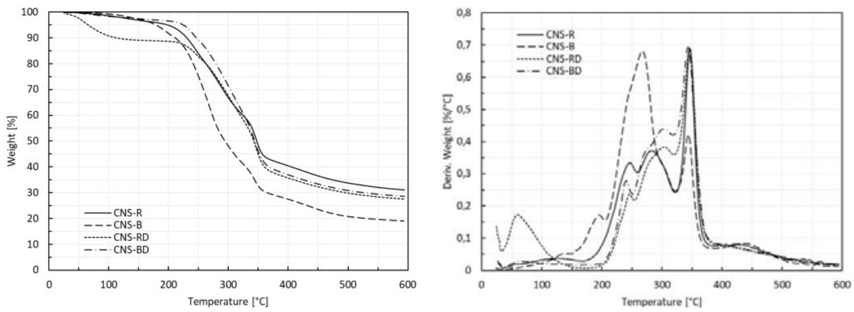

Fig.1. Thermal behavior of CNS from pre-processing and after CNSL extraction. **(a.)** TGA **(b.)** DTGA

Finally, the low density of CNS compared to other natural fibers and the thermal behavior of CNS from the CNSL extraction process show that this natural fiber is a promising candidate for developing new green composites in Colombia.

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The authors wish to express their gratitude to Sistema General de Regalías, OCAD de Asignación de Ciencia, Tecnología e Innovación ACTeI and Ministerio de Ciencia, Tecnología e Innovación which funded this research through the Project "Aprovechamiento de los subproductos Agroindustriales en la producción del marañón en el departamento del Vichada - BPIN 2020000100571.

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ID 109
DAMAGE EVOLUTION ANALYSIS OF FLAX FIBRE BIOCOMPOSITE UNDER CREEP LOAD BY ACOUSTIC EMISSION AND μ-CT

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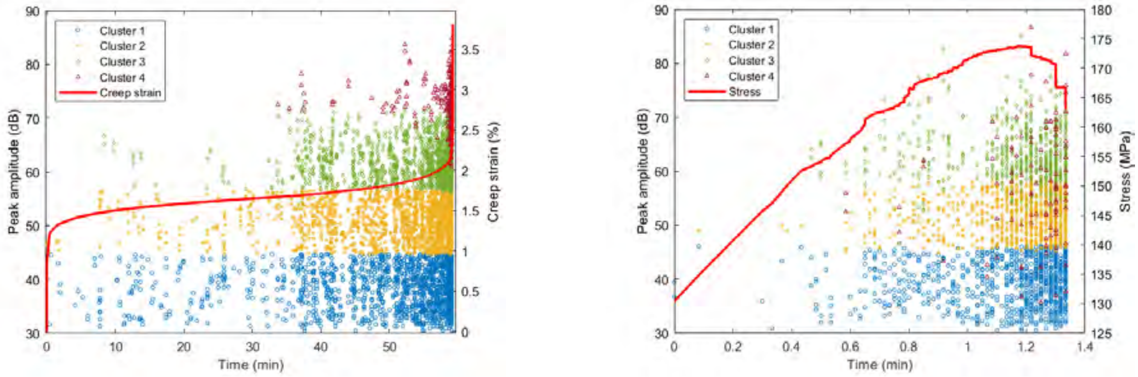
ABSTRACT
This work aims to reveal the damage mechanisms and evolution of unidirectional flax fibre composites when subjected to creep load. The acoustic emission technique (AE) was used to monitor the failure progress during flexural creep tests. Clustering analysis was carried out to recognize the AE events by the unsupervised machine learning algorithm k-means ++. Then the correlation between clusters with damage modes was established based on elaborate coupons with targeted failure mechanisms and computed tomography analysis. The identified AE events showed that the damage initiated from matrix cracking in the primary creep stage, and then fibre-matrix interface debonding combined with fibre pull-out occurred sequentially during the steady stage. In the tertiary creep stage, the explosive increase in fibre fracture emerged and eventually triggered catastrophic failure. Additionally, the considerably more cumulative AE events detected during creep tests when compared to quasi-static tests, showed that plenty more damage was generated under creep load, and therefore caused the strength degradation.

INTRODUCTION
Plant fiber biocomposites have been adopted as an alternative to synthetic fibre composites in certain engineering fields in the context of sustainable and circular economy. One of the main obstacles for plant fibre biocomposites to extend their value to structural applications is the lack of knowledge on the durability and long-term performance (Koolen et al., 2019; Sala et al., 2021). The stiffness and strength degradation under a constant load (creep behavior) has been observed for polymer composites and is particularly evident for plant fibre biocomposites in which both the fibre and matrix exhibit viscoelastic behavior. However, mechanisms behind this phenomenon have not been well- studied and there is still no clarity about the damage progress of composite materials under creep load.

The composite material used in this work was manufactured with unidirectional flax tape with an aerial weight of 110 g/m^2 and polypropylene as the matrix. The composite plates were produced by thermal compression molding by stacking flax UD tapes between polymer films. The creep tests were performed on a universal testing machine (Instron 5567) in 3-point bending mode. The AE sensors were clamped on the ends of the samples during testing.

RESULTS AND CONCLUSIONS
Four different clusters were obtained based on the AE events detected during creep tests and quasi-static tests. Fig. 1 (a) presents the peak amplitudes of these AE patterns with respect to time, superposed with the evolution of the creep strain on the right y-axis. In the primary creep stage, only a small amount of

AE events from cluster 1 (matrix cracking) and a very few amounts from cluster 2 (fibre-matrix interface debonding) were detected. Most of events from cluster 2 and all events from cluster 3 (fibre pull-out) emerged successively at the beginning of the steady stage and constantly appeared until the sample failure. The cluster 4 (fibre breakage) occurred sporadically during the steady stage and explosively grew in the tertiary creep stage, which is believed to be the dominant inducement to failure. The clusters for quasi-static tests depicted in Fig. 1 (b) show a similar evolution progress. With the applied load (right y-axis) increasing to 131 MPa, the events from cluster 1 started to appear which implied the initiation of damage. Then the cluster 2 and cluster 3 were observed sequentially. In the end, the specimen failed with substantial events from cluster 4 rapidly emerging when the applied load reached to the strength value of 174 MPa.



Creep test (b) Quasi-static test

Fig. 1 Clustering results for AE events detected in (a) creep test and (b) quasi-static test

Fig. 2 compares the number of AE events from different clusters based on the creep test and quasi-static test. It can be found that the number of events detected during the creep test is substantially higher than in the static test for all clusters, which indicates considerably more damage generated when subjected to creep load, and therefore resulted in failure when the load was below the quasi-static strength.

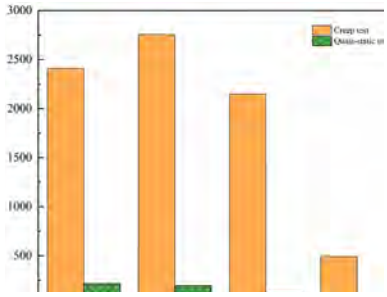


Fig. 2 Number of events from different events under creep load and quasi-static test

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support from the China Scholarship Council and the EU Interreg NWE Smart Circular Bridge project.

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ID 112
BIOHARDENING WITH PLANT GROWTH PROMOTING MICROORGANISMS (PGPM'S) AS BIOFERTILIZER FOR ABACA

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ABSTRACT
Growth performance of abaca was improved with PGPM application. At the maturity stage of abaca plants, different growth parameters were higher with the combined application of vermicompost and *Lactobacillus* based bio-fertilizer materials. In like manner, this particular intervention had yielded the highest leaf biomass of the experimental plants. Better growth and higher biomass production could be attributed to the better P uptake of the plants with the combined application of vermicompost and *Lactobacillus* based bio-fertilizer materials.

INTRODUCTION
Abaca fiber is one of the major sources of foreign exchange earnings of the country. Among the producers of natural fibers, Philippines contributed about 85% in the world market of abaca fiber. At the beginning of the 21st century, the country had an average production of approximately 70,000 tons of abaca fiber (FAO, 2007) of which the bulk of the fiber produced comes from the Visayas region. However, several problems beset the abaca industry that deter the production of high yield and good quality abaca fibers. Aside from the destructive bunchy top disease that almost wiped-out abaca plantation in the region, decline in soil fertility aggravates the situation.

Nutrient depletion could be one of the possible causes in yield reduction in intensively cultivated abaca growing areas since growers consistently extract more nutrients (i.e. soil mining) from the soil than is usually returned. Approaches such as the use of beneficial organisms such as plant growth promoting microorganisms (PGPM) could possibly improve these the soil condition. The most widely used and popular PGPM's are indigenous microorganisms (IMO's), effective microorganisms (EM's) and AMF which believed to be a more effective and practical approach in soil fertility restoration, water relations and increased disease resistance (Spagnoletti, et al, 2021; Irankhan , et al., 2021). Reports showed that hyphae of the AM fungus also stabilize soil aggregates by both enmeshing soil particles (Miller and Jastrow 1990) and excreting a glycoprotein ("glomalin"), which may act as a glue-like substance to adhere soil particles together (Wright and Upadhyaya, 1998).

RESULTS AND CONCLUSIONS
Hardened abaca plantlets inoculated with bio-fertilizers were assessed and monitored under field condition. Promising treatments were planted and considered as potential material for better performance of abaca plants.

Towards the end of the life cycle of the mother plants (i.e. at 17 to 23 MAT) plant height, number of leaves, leaf area, base circumference, and number of suckers of abaca were significantly affected by the addition of the different BOF materials. Tallest plant height was obtained in abaca amended with vermicompost and *Lactobacillus* (T5) while those plants applied with vermicompost alone, showed the shortest plant height (Figure 1). Likewise, this particular treatment showed more vigorous growth compared to the rest of the treatments.

The same observation was noted in the number of leaves and leaf area in which plants amended with vermicompost and *Lactobacillus* showed the highest number and bigger area compared to the rest of the treatments.

Similarly, bigger base circumference and more number of healthy suckers of the abaca were obtained in abaca amended with the combined application of vermicompost and *Lactobacillus*.

These observations suggest that the combined application of vermicompost and *Lactobacillus* could be a promising amendment in rehabilitating abaca growing areas. This particular treatment obtained the highest leaf biomass (Fig 2). Favorable growth and biomass production could also be attributed to the higher Phosphorous uptake (Fig 3) of the plants treated with the combined application of vermicompost and *Lactobacillus*.

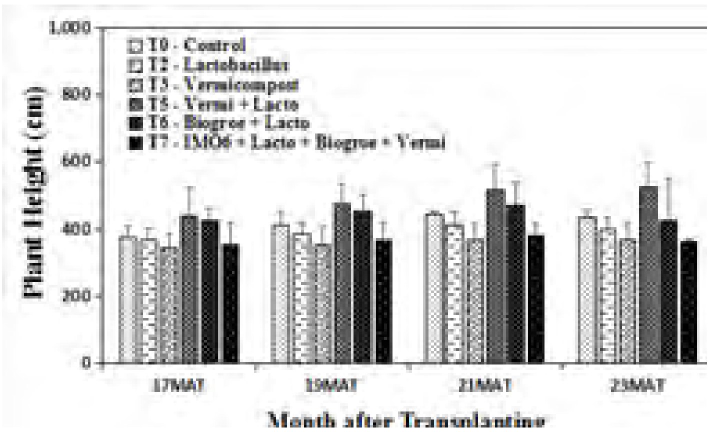

Fig 1. Plant height (cm) of abaca at maturity stage as influenced by the different biofertilizer application

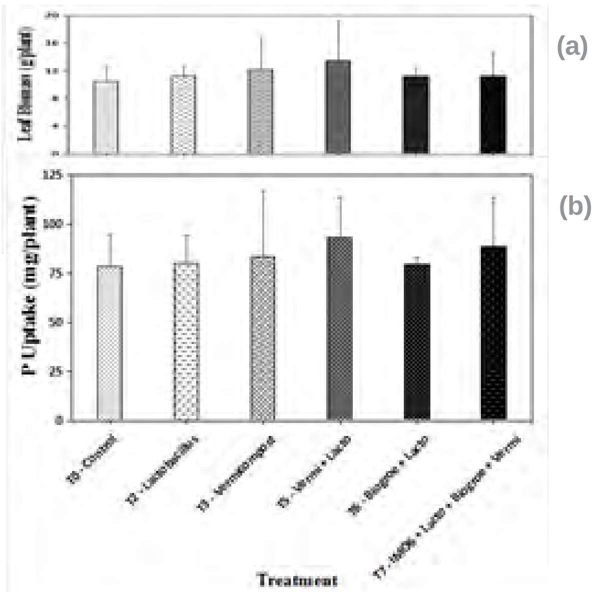

Fig 2. Leaf biomass (g/plant) **(a)** and P uptake (mg/plant) **(b)** of harvested abaca as influenced by the different biofertilizer application

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ID 113
POTENTIAL OF ASH FROM PELAGIC SARGASSUM SPP. AS STABILISER IN EARTH-BASED MATERIALS: PHYSICAL, CHEMICAL AND THERMAL ASSESSMENT

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ABSTRACT
This study evaluates the effect of the pelagic Sargassum desalination process on its physico-chemical and thermal properties as well as on the chemical and mineral composition of the ash. Fresh pelagic Sargassum was subjected to two cycles of immersion in water at 40 min each to dissolve excess salt in the water. Characterisation of the raw and desalted Sargassum shows an increase in volatile matter from 43.74% to 50.82%, fixed carbon from 3.29% to 8.34% and porosity from 84.59% to 86.18%, while there is a reduction in ash content from 28.44% to 17.25% after desalination. Furthermore, the TGA and DTG results show a slight reduction in the degradation temperature, which is associated with a reduction in the amount of residual char from 35% to 27%. The results of the XRF analysis of the Sargassum ash showed a very low alumiosilica content ($SiO3+Al2O3$ < 4%), but a significant increase in CaO content after desalination from 21.96% to 50.17% while K and Cl contents decreased to 48% for K and 73% for Cl. These chemical and mineral analyses of the ash suggest the possibility of using desalinated Sargassum ash as a partial substitute for cementitious materials for soil stabilisation to reduce greenhouse gas emissions from cement production.

INTRODUCTION
The massive invasion and proliferation of *Sargassum* algae on beaches and coasts in recent years is endangering people's livelihoods, climate-sensitive areas and local ecosystems (Tobío-Pérez et al., 2022). This *Sargassum* outbreak problem has prompted several Caribbean countries to declare a national state of emergency (Desrochers et al., 2020). Due to the severe environmental, health and economic impacts associated with the presence, mechanical cleaning and storage of *Sargassum* on costs and on the beach (Tobío-Pérez et al., 2022), the Caribbean Regional Fisheries Facility has estimated the cost of *Sargassum* clean-up in 2018 at US$210 million (Davis et al., 2021). The complexity, economic impact and regional scope of *Sargassum* biomass management should lead to synergy between different actors (national and international) through interdisciplinary and transdisciplinary approaches (Robledo et al., 2021). The use of Sargassum as fertiliser has raised concerns about the bioaccumulation of metals such as Al, Ca, Fe, Mg, K, Na and Mn at concentrations higher than those in the surrounding seawater (Milledge et al., 2020). Research to develop sustainable value chains for *Sargassum* to promote economic opportunities that could help mitigate the impacts of mass influxes seems commendable. Although these algae represent a harmful marine resource for many Caribbean countries, they also represent a high-potential energy and building material opportunity that has been little explored.

RESULTS AND CONCLUSIONS

Fig. 1 show the thermo gravimetric (TGA) and derivative analysis (DTG) curves of pelagic Sargassum before and after pretreatment. The results reveal that TGA curve (Fig. 1a) is constituted of three zones in correlation to the pyrolysis results of various marine seaweed materials (López-Sosa et al., 2020). Zone I shows a very low percentage of mass loss (≈17% for S1 and ≈20% for S2) due to dehydration in the temperature range of 40 to 204°C. The degradation of zone II accuses between temperatures 204 and 602°C and displays the highest mass degradation (≈35% for S1 and ≈41% for S2), which is related to the removal of various amounts of biopolymers from the algae (López-Sosa et al., 2020). The final degradation above 602°C (zone III) is attributed to the combustion of carbonaceous residues triggered by intense heating (Kumar et al., 2021).

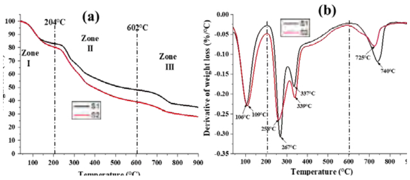

Fig.1 (a) TGA curve and **(b)** DTG curve of Sargassum seaweed before (S1) and after pretreatment (S2)

ACKNOWLEDGMENTS

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ID 114
NUMERICAL MODELING OF NANO-FIBRIL ALIGNMENT IN ELECTRIC FIELD ASSISTED FLOW FOCUSING DEVICE

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ABSTRACT
The effect of applied electric field on nanoscale ordering of CNFs in the electric field assisted flow-focusing device was numerically clarified. The equations of translational and rotational motion were solved for individual 1000 ellipsoidal solid particles assuming CNFs. The flow field in the flow-focusing channel is previously simulated by VOF method. The numerical simulation revealed that the CNF order parameter increases up to 0.36 by electric field and 0.62 at the exit of the channel by elongational flow for CNFs of 600 nm with applying 600 V. With the pre-alignment by electric field, the CNFs order parameter improved by 16.6 % under this condition.

INTRODUCTION
In recent years, cellulose nanofibrils (CNFs) have received considerable attention as one of the most advanced nanosized biomass materials. Fibrils can be obtained by disintegrating paper pulp through a combination of mechanical shearing and high-pressure homogenization, resulting in controlled fibrillation down to a width of around 4-20 nm and length of around 1 μm. The cellulose filament fabricated by integrating the CNFs through flow-focusing method has been reported to express the outstanding mechanical properties. As is well known, the microscopic alignment inside the cellulose filament leads to the strong filament (Håkansson, 2014).
Recently, Takana et al. proposed an innovative method of controlling CNF alignment by coupling an alternating electric field with conventional fluid dynamical alignment control (Takana, 2020). In this device, electrodes are installed upstream from the flow-focusing section. CNFs in the channel are pre-aligned by the alternating electric field along the flow direction, and then they are transported into the flow-focusing section to be further aligned. It was successfully demonstrated the impressive enhancement in mechanical properties of cellulose filament with stiffness up to 25 GPa and more than 63% and 120% increase in tensile strength and toughness, respectively with improved nanoscale ordering (Wise, 2020).
In this study, the effect of applied electric field on nanoscale ordering of CNFs in the electric field assisted flow-focusing device was numerically elucidated.

RESULTS AND CONCLUSIONS
In this study, 1000 sample ellipsoidal solid particles assuming CNFs were introduced in the electric and flow fields. The CNF behaviour in the channel were simulated by solving the equations of translational and rotational motion for individual particles. The flow channel has 1 mm x 1 mm rectangular cross section, and the alternative electric field is applied in axial direction by a pair of electrodes installed in upper and lower walls at 8 mm upstream of the flow-focusing location. At the flow-focusing point, the elongational flow is

induced by introducing sheath flow from right and left side of the channels as shown in Fig. 1.
The flow field in the flow-focusing device is solved by VOF method in the 1/4 domain with symmetry boundary conditions as shown in Fig. 1. The volume fraction has values between 0 to 1.0. The volume fractions of 0 and 1.0 correspond to water phase and CNF dispersion, respectively, and the volume fraction of 0.5 is defined as the interface between water and CNF dispersion. The CNF dispersion firstly detaches from the side wall by injection of sheath flow at z = 2 mm and then separated from the upper and lower walls to form the liquid thread by elongational flow.
The instantaneous image of the particles (CNFs) in the electric field region of the channel is shown in Fig. 2. The color bar in the figure corresponds to the particle orientation angle along the z-axis. Because of the electrostatic torque acting on the electrically polarized particles, particles rotate to be parallel to the electric field. The numerical simulation revealed that the order parameter s (with s =1.0 being perfect alignment) increases up to 0.36 by electric field and 0.62 at the exit of the channel by elongational flow for CNFs of 600 nm for applied voltage of 600 V. With the pre-alignment by electric field, the CNFs order parameter improved by 16.6 % under this condition.

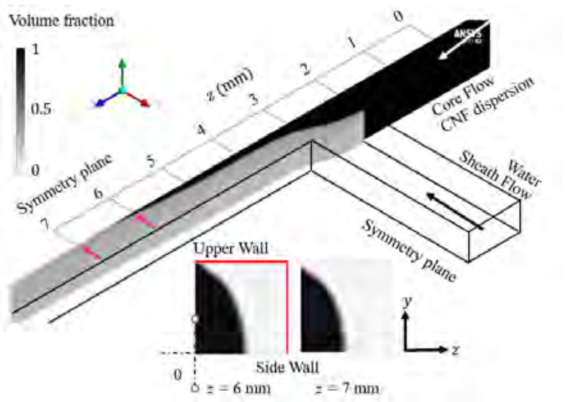

Fig. 1 CNF dispersion flow in the flow-focusing channel solved by VOF method.

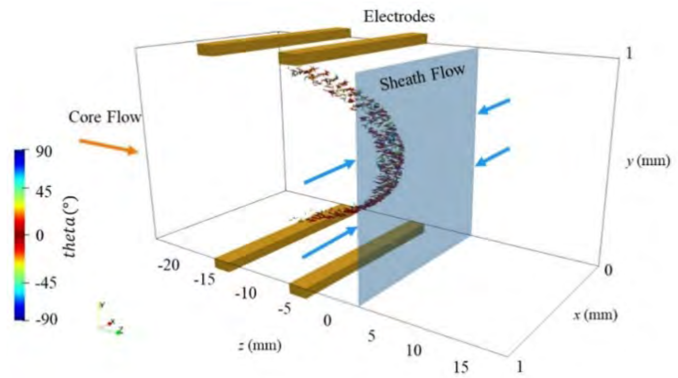

Fig. 2 Visualization of particles (CNFs) in the electric and flow fields.

ACKNOWLEDGMENTS
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ID 116
STATIC AND FATIGUE PROPERTIES OF CROSS-PLY FLAX COMPOSITES UNDER HYGROSCOPIC AGEING CONDITIONS

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ABSTRACT
An experimental investigation was performed to study the effect of hygroscopic aging in realistic moisture conditions on the static and dynamic properties of natural fibre composites(NFCs). Cross-ply $[0/90]_{4s}$ flax-epoxy composites were prepared with resin transfer molding (RTM) and exposed to the summer and winter average relative humidity of the Western European outdoor climate by making use of a climate chamber. The ageing protocol counted 12 hygroscopic aging cycles between RH65% and RH85% at 40°C. Results showed that the reduction in tensile and flexural stiffness of composites was 9% and 13%, respectively. While moisture aging, in this regime, had no effect on the static and dynamic properties of cross-ply biocomposites.

INTRODUCTION
In recent years, natural fibres (NF) have received considerable attention to replace synthetic fibres in fibre reinforced polymer composites for advanced applications. However, the hygroscopic nature of natural fibres might limit the application of NFCs especially in outdoor conditions where the moisture conditions are typically harsher compared to the indoor climate [1]. Moisture uptake can cause severe softening of plant fibre reinforced composites since intermolecular interactions are weakened leading to increased chain mobility; hence, the mechanical properties could be changed by moisture uptake. Berges et al. [2] studied quasi unidirectional flax fibre - epoxy composites and reported a decrease in flax yarn stiffness of 40% when comparing the stiffness at 85% humidity with the value at standard conditions, RH 50%. Hendrickx et al. [3] used a harsh ageing protocol of ten wet/dry cycles between oven dry and 80% relative humidity and reported a 35-40% decrease in the longitudinal strength of unidirectional flax fiber-reinforced epoxy and polypropylene composites. To sum up, it is well established in literature that moisture can have a severe effect on the properties of NFC, however, ageing studies which simulate realistic ageing conditions are limited. Therefore, cross-ply flax-epoxy composites were prepared and aged in a climate chamber at the summer and winter average humidity in the Western European outdoor climate, corresponding to RH65% and RH85%, respectively. The ageing protocol comprised 12 wet-dry cycles at a temperature of 40°C. The static properties were evaluated throughout the ageing process both in the wet state RH85% and after saturation at standard conditions RH50%, 21°C. The fatigue properties were evaluated at standard conditions at two moments, namely, after production and after 12 hygroscopic ageing cycles.

RESULTS AND CONCLUSIONS
The evolution of tensile and flexural properties of cross-ply [0/90]4s flax-epoxy composites during 12 hygroscopic ageing cycles is depicted in Figure 1. Evaluating the effect of moisture absorption by comparing the properties in the wet state (Cycle 0.5, 3.5, 6.5, 9.5, 12.5) with the properties determined at standard

conditions (Cycle 0, 6, 12) shows an average decrease in tensile and flexural stiffness of 9% and 13%, respectively (please see Figure 1). In addition, the composite failure strain followed the opposite trend compared to the evolution of stiffness, while strength is largely unaffected. Furthermore, humidity has a greater effect on the flexural properties compared to the tensile properties, raising the presumption that the compressive properties are more influenced by moisture. The effect of moisture ageing evaluation is demonstrated by comparing the properties at cycles 6 and 12 with the properties after production (cycle 0), which are all determined under standard conditions. All static properties in both tensile and flexural loading are statistically unaffected.

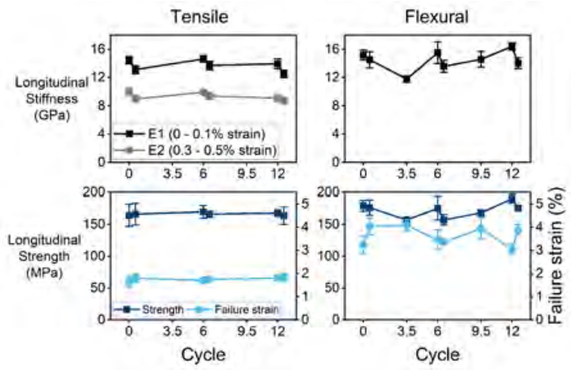

Figure 1: Influence of moisture ageing on the tensile and flexural properties of cross-ply [0/90]s flax-epoxy composites (Vf= 39 ± 0.5 %)

Figure 2 shows the fatigue properties of cross-ply $[0/90]_{4s}$ flax-epoxy composites which were recorded after production and after 12 hygroscopic aging cycles, both after equilibration at standard conditions. The S/N curve results show limited differences in the slope of the regression line for both samples, indicating that damage propagation during fatigue loading is not significantly altered. Furthermore, the static strength after production and after moisture ageing were statistically equal, hence, the differences in fatigue resistance were also limited.

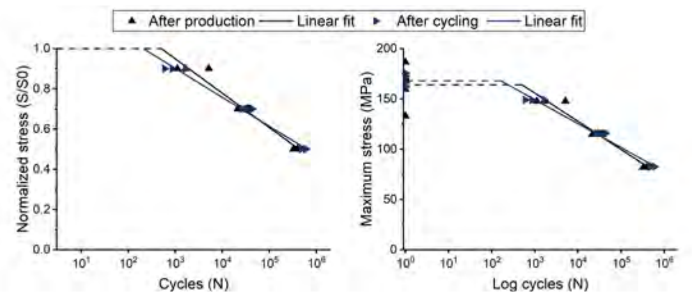

Figure 2: S-N curves of cross-ply $[0/90]_{4s}$ flax epoxy composites, Vf = 39 ± 0.2%.

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ID 118
INFLUENCE OF COMPACTION PROCESS PARAMETERS ON NCF FLAX FIBRE PREFORM WITH HYGRO-THERMAL MONITORED CONDITIONS

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ABSTRACT
This work aims to investigate the influence of thermo-hygro-mechanical parameters during an experimental compaction study of a NCF flax fibre stack. Temperature and relative humidity conditions applied for samples aging are maintained during compaction tests, in order to evaluate their influence on the flax fibre preform behaviour subjected to through-thickness compressive forces.
The study is focused on reporting the influence of compressive strength, variable HR% and T°C, for a regular number of NCF flax fibre layers, with a compaction to a minimum thickness of 3mm.

INTRODUCTION
Currently, composites manufacturing processes are not dedicated to plant fibre-reinforced specificities. Their particular structural and chemical compositions result in a thermo-hygromechanical behaviour, which is significantly different than synthetics fibres ones (Baley C, 2019).
During a compaction step, occurring in a fibre-reinforced composite material manufacturing process, fibres are subjected to through-thickness compressive forces (Yong AXH, 2021). As a response to mechanical load, the fibre stack is likely to be warped, which could modify the preform permeability, due to the morphology of porous structure (Ouagne P, 2010).
While compaction is taking place, fibres could be also exposed to various hygro-thermal conditions. Due to hydrophilic behaviour of plant fibres, their moisture absorption mechanisms have been widely studied, both in immersion and relative humidity (Célino A, 2014a, 2014b). In their study, authors have highlighted that "water sorption in fibres and their composites has been found to significantly affect their dimensional and structural properties". Therefore, during a compaction of plant fibre preform, hygro-thermal conditions must be also monitored, in order to take into account thermo-hygro-mechanical specificities of plant fibres.
The current work is dedicated to the analysis of a NCF flax fibre stack. Values corresponding to the maximum compaction stress, the thickness at maximum compaction stress, and real deformation are correlated with the set of experimental compaction process parameters.

EXPERIMENTAL APPROACH
To lead the study to reliable results in replicable experimental conditions, a dedicated step by step protocol has been established. Moreover, in order to enable results comparison, either with textile compaction or

plant fibres hydrophilic behaviour studies, protocol is in accordance with corresponding literature benchmark and review (Yong AXH, 2021; Célino A, 2014a).

Main steps require attention in order to limit scatter in results: the fibre stack forming, the hygro-thermal samples aging, and the compaction test process parameters monitoring.

Therefore, requirements and dedicated tools has been designed to obtain preforms in replicable conditions. Environmental chambers (HR%, T°C) and universal testing machine (force, displacement) have been previously calibrated. During the compaction test, with hygro-thermal regulation, all parameters are monitored and recorded.

Ranges of speed compressive strength (0.1 to 10 mm/min), relative humidity (10 to 80 HR%) and temperature (23 to 80°C) for a regular number of NCF flax fibre layers, with a compaction to a minimum thickness of 3mm are analysed.

Thanks to this first set of parameters, authors established correlations between mechanical fibre stack responses and the hygro-thermal experimental conditions previously highlighted. In accordance with results, more parameters are investigated in order to establish a ranking of their respective influence.

ACKNOWLEDGMENTS

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ID 119
UNDERSTANDING THE FATIGUE RESPONSE OF HYBRID FLAX COMPOSITES UNDER LOAD CONTROL CONDITIONS

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ABSTRACT

In this study, the fatigue behavior of two composite materials, Glass/Flax and Kevlar/Flax, was evaluated. Samples with a 12-ply unidirectional flax core, encased by a 2-layer skin, with similar fiber volume fractions, were subjected to tension-tension fatigue testing. The results showed that the Kevlar/Flax specimens had a higher fatigue life and endurance compared to the Glass/Flax specimens. This difference was attributed to the synergistic properties that Kevlar/flax hybrids display as a result of their similar physical characteristics.

INTRODUCTION

The use of natural fibers has a long history of enhancing the properties of various materials. In recent years, natural fibers have gained attention as a viable alternative to synthetic fibers. Flax, being one of the most widely grown natural fibers, offers a unique combination of strength, stiffness, lightness, and cost-effectiveness [1,2]. The blending of natural and synthetic fibers creates a hybrid solution, as each material can offset the disadvantages of the other. The assessment of long-life fatigue behavior and fatiguing mechanical properties is crucial for the design of components and structures that can provide a long-term durability prediction [3,4].

However, the fatigue mechanism of flax fiber composites and their hybrid composites is complex and not well understood due to a lack of systematic and detailed information. This study aimed to investigate the fatigue performance of Kevlar/Flax and Glass/Flax epoxy composites under stress-controlled amplitude fatigue testing in two different flax fiber orientations (0° and ±45°).

The hybrid composites were manufactured from 12 layers of dry Flax sandwiched in between four layers of Kevlar 49 and Glass Fibers. The matrix used is an Araldite® LY 1565 (epoxy) + Aradur ® 22962 (polyamine hardener) thermosetting resin system (Huntsman). Two 305 mm x 305 mm laminate configurations were manufactured for each: with the following stacking sequences; $[0_{K2}/0_{F6}]_S$ and $[0_{G2}/\pm45_{F6}]_S$ where the *K, G* and *F* represent the Kevlar, Glass and the Flax fibers respectively. Samples from both hybrid configurations were tested at 5 Hz.

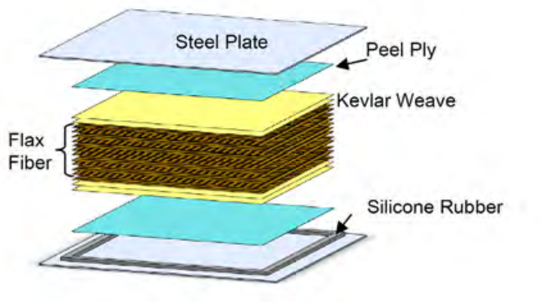

Figure 1. Manufacturing assembly for hybrid flax composite

RESULTS AND CONCLUSIONS

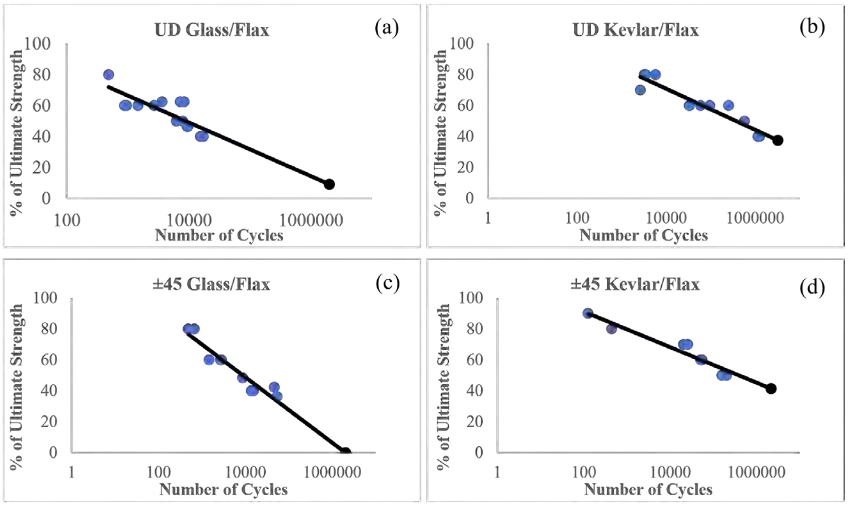

Figure 2: S-N plots of Unidirectional (a) Glass/Flax, (b) Kevlar/Flax and ±45 (c) Glass/Flax and (d) Kevlar/Flax displaying runout/fatigue strength at 2 million cycles

These preliminary results display the differences in the fatigue perfromance based on the fiber orientations and the fiber types. While Glass/Flax hybrids may offer greater strength and stiffness, the figure above shows the importance of fatigue analysis. Kevlar having similar physical properties to flax i.e. density, ultimate strain, and modulus, pairs extremely well with flax, whereas glass fibers being of brittle nature, with a lower breaking strain, do not allow flax to share the load, hence resulting in earlier fracture.

ACKNOWLEDGMENTS

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ID 120
NANOCELLULOSE BASED MATERIALS FOR FOOD PACKAGING

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ABSTRACT
The present communication describes innovative nanocellulose-based materials for active food packaging, fabricated following simple and eco-friendly approaches. Focus will be given to nanocellulose films impregnated with a bioactive extract through supercritical impregnation, and thermoplastic starch-bacterial nanocellulose films enriched with gallic acid. Overall, the films presented good mechanical performance, UV-blocking properties, and antioxidant and antibacterial activities, that are fundamental for the intended application.

INTRODUCTION
The use of active packaging technologies and biopolymeric materials are among the emerging trends for a more sustainable food packaging industry. Nanocellulose substrates, e.g., nanofibrillated cellulose (NFC) and bacterial nanocellulose (BNC), have gained relevance in this field due to their unique properties (biodegradability, tailorable surface chemistry and excellent mechanical properties), which, combined with bioactive compounds (or extracts) allows the fabrication of advanced nanomaterials for sustainable active food packaging.

RESULTS AND CONCLUSIONS
In the first study, bioactive free-standing films composed of nanofibrillated cellulose (NFC) and a phenolic compounds-rich extract, viz. mango leaf extract (MLE), were fabricated via supercritical CO_2 impregnation (SSI) and conventional solvent casting for comparison purposes (Figure 1) (Bastante, 2021). The SSI-assisted impregnation of NFC with MLE originated robust films with thermal stability up to 250 ºC, good mechanical properties (Young's modulus > 4.7 GPa), UV-light barrier, antioxidant capability with maximum inhibition percentage of ca. 84%, and antimicrobial activity against *Staphylococcus aureus* (growth inhibition ≈ 37%) and *Escherichia coli* (growth inhibition ≈ 91%). The comparison of the NFC/MLE films prepared by SSI with those fabricated via solvent casting shows that the antioxidant and antimicrobial activities are noticeably higher in the films obtained by the SSI-assisted impregnation of MLE into NFC, because the MLE components are mostly adsorbed at the surface and not in the bulk of the biopolymeric matrix, which results into faster migrations and, thus, higher active properties.

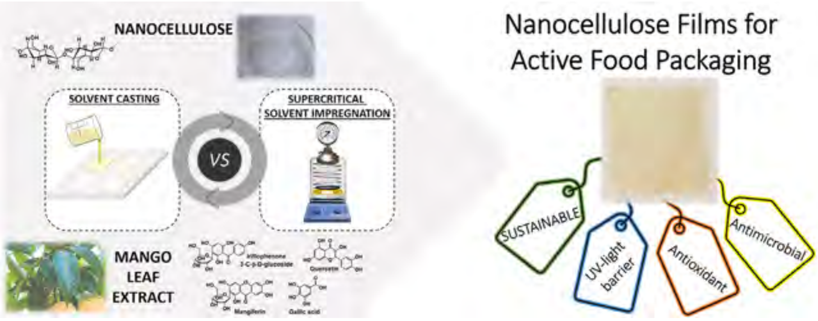

Figure 1- Scheme summarizing the preparation of the NFC-MLE based films (Bastante, 2021).

In the second approach, bioactive transparent nanocomposite films of thermoplastic starch (TPS) reinforced with bacterial nanocellulose (BNC) (1%, 5% and 10% w/w, relative to starch) and enriched with a phenolic compound, viz. gallic acid (1 and 1.5% w/w, relative to starch) were prepared by solvent casting (Figure 2) (Almeida 2023). The addition of BNC (≥ 5% w/w) and GA (1 and 1.5% w/w) improved both the mechanical properties (Young´s Modulus: 1.2 -2.0 GPa vs. 1.0 GPa in TPS; tensile strength: 23 - 39 MPa vs. 20 MPa in TPS) and the water resistance (moisture absorption and solubility in water) of the films. All nanocomposite films are thermally stable up to 125 °C. It was also noticed that the addition of GA imparted the TPS-BNC nanocomposite films with UV-blocking properties and antioxidant activity (DPPH scavenging activity above 80%). Moreover, the film with 10% w/w of BNC nanofibers and 1% w/w of GA revealed to have good oxygen barrier property with a coefficient of permeability of 0.91 ± 0.12 $cm^3 \mu m\ m^{-2}\ d^{-1}\ kPa^{-1}$ and antibacterial activity against *S. aureus* (reduction of about 4.5 $\log_{10}$ colony forming units (CFU) mL^{-1} after 48 h).

Figure 2- Scheme summarizing the fabrication of TPS_BNS_gallic acid-based films (Almeida, 2023).

Both studies evidenced the potential of nanocellulose substrates for the development of eco-friendly functional bio-based materials, following different methodologies, for application as sustainable active food packaging.

ACKNOWLEDGMENTS

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ID 121
ABACA/PE ROTOMOLDED PARTS MADE IN 1-, 2- OR 3- LAYERS

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ABSTRACT
1-, 2-, and 3-layer parts were produced by rotomolding with different loadings of abaca fiber (5-20 %), performing a comparison on the mechanical properties with the fiber content and shots. Rotomolding cycles were analyzed, finding an increase in heating time for all composites. The impact properties are reduced for all composites, while the tensile Young's modulus increases for the parts made in 2-layers at 10 % loading of fibers. The higher energy consumption needed for the different layers would only be justified by an increase in the composite properties.

INTRODUCTION
Rotational molding allows for obtaining hollow parts with good mechanical properties and surface quality, despite no pressure being commonly applied in the process. This sector is expected to have an annual increase of about 6 %, expecting to reach 8.500 million euros in 2031. Some of the main drawbacks of this technology are the long cycle times, the relative difficulty in obtaining complex geometries, and the lack of a wide range of materials. Very few materials are used in rotational molding due to the process's particularities and sensitiveness, with polyethylene (PE) accounting for more than 80 % of the rotomolding market. In the last years several authors have proposed using biobased or biodegradable materials to increase products sustainability, being currently PLA the second most commonly rotomolded material. Few works about using recycled materials have been performed in rotomolding; for example, PLA fractions (Aniśko et al., 2022), post-consumer plastic residues (Cestari et al., 2021) or cable wastes (Díaz et al., 2018). The introduction in rotomolding of lignocellulosic materials has also been explored in literature, as a way to produce more sustainable products (Suárez et al., 2021), by using waste or side-stream products. Banana and abaca (Ortega et al., 2013; Monzón et al., 2012), agave (Cisneros-López et al., 2018), buckwheat husks (Andrzejewski et al., 2020) or wood dust (Abhilash et al., 2022) has been assessed at different loadings, generally not exceeding 10 %. The comparison of mechanical properties of parts obtained in the different shots the assessment of the viscoelastic behavior of the different parts, and the analysis of cycle times are the aims of this communication.

RESULTS AND CONCLUSIONS
Cycle time for one layer is significantly shorter than for 2- and 3-layers parts. Longer cycle times are directly related to higher energy consumptions, but also to higher possibilities of fiber degradation or polymer oxidation. The use of fibers, even at low loadings (5%) provokes a delay in the induction and sintering time, reaching the desired internal temperature at longer times with the higher fiber content. Similar conclusions are observed for the 2- and 3-layers parts: the higher fiber content, the longer the cycle times, and the longer the heating required. However, if comparing the heating time versus the final content of the fiber, it

is observed that the effect of working with layers is more significant than the fiber content. Only for 3-layers the total heating time is increased (about 10 %), while parts made in 2-shots have a similar total cycle time than parts made in one. So, considering the longer heating times, higher energy consumption and bigger processing difficulties, this strategy would only make sense to increase the ratio of foreign material or to improve the final properties of the part.

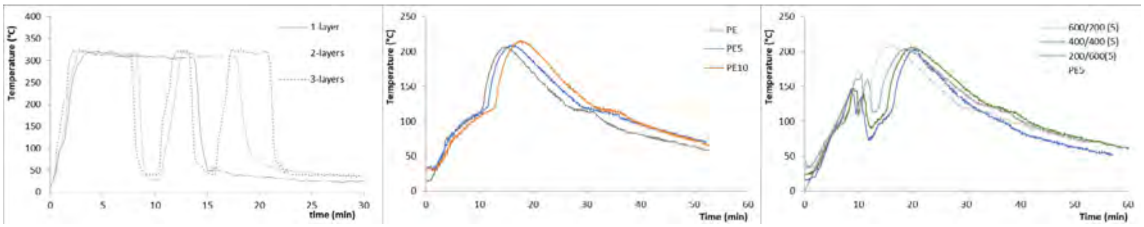

Fig. 1 External and internal temperature profiles for 1-, 2-, and 3-layers parts

Next figure shows the relationship between the tensile and flexural elastic modulus with the fiber content; the tensile elastic modulus only increases for the 2-layers parts with fibers at 10 %, remaining unchanged for 1- and 3-layers parts, regardless of the fiber content or the use of PEMA as compatibilizer. The 2-layers structure with only 5 % of fiber does not has fiber enough to achieve this reinforcing effect. Besides, the outer skin of well-consolidated homogeneous PE, provides better results than having a similar amount distributed in the entire part thickness. Impact test is drastically reduced for all composites, regardless the layers or fiber content. Tensile and flexural strength show similar behavior than elastic modulus.

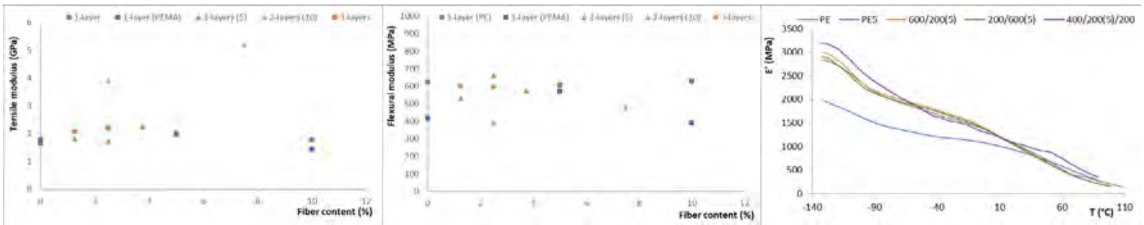

Fig. 2 Comparison of tensile and flexural modules with fiber content, by number of layers (above). Storage modulus for composites with fiber and 1-, 2-, and 3-layers (below)

The storage modulus (E') for the composites with 5% fiber made in 2 and 3-layers show by a closer behavior to the neat PE than for the 5% loaded part made in a single layer. The 3-layers parts show a less smooth curve, which might be due to the irregular distribution of the internal PE layer. Besides, the viscoelastic behavior of the composites does not show any significant differences for the parts with 5 or 10% fiber in the different layers, as seen above for E'.

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ID 122
APPLICATION OF GREY BASED TAGUCHI ANALYSIS FOR OPTIMIZING PROCESSING PARAMETERS OF COMPRESSION MOULDING TO PRODUCE PLA BASED JUTE COMPOSITES

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ABSTRACT
In an attempt to encourage the sustainability and reduce the pollution due to landfills caused by the composites, researchers are inclined towards the usage of green composites. Low mechanical properties of the composites hamper their wide spread of application. Hence, an attempt was made to improve the mechanical properties (tensile, flexural and impact) of Polylactic Acid (PLA) composites reinforced with jute woven fabric by optimizing the processing parameters of compression moulding. To accomplish it, Taguchi combined with Grey Relation Analysis (GRA) was implemented. The use of GRA is to obtain the composite with optimised tensile, flexural and impact properties simultaneously.

INTRODUCTION
Green composites are produced by reinforcing biopolymer with natural fibers. By understanding the life cycle of green composites, it is observed that these composites do not cause harm to the environment as they are degradable at the end of their life. Polylactic Acid (PLA), Polyhydroxyalkanoates (PHA), Polyhydroxybutyrate (PHB), Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) and starch are some of the known biopolymers being investigated for the development of green composites. Among all, PLA is well known due to their good mechanical properties in comparison with other biopolymers and its properties is more or comparable to polypropylene. PLA can be reinforced with natural fibers to improve its mechanical properties. Compression molding was considered as viable manufacturing technique to produce composites with long fibers and woven fabrics as reinforcements. Temperature, pressure and time are the vital processing parameters to be considered. It is very much essential to optimize these processing parameters to obtain the composite with good performance (Sanivada, 2020).
As there are three processing parameters and these are varied at three levels as shown in the Table 1, it is required to carry out 27 experiments to find out the optimised processing parameters. Hence to reduce the number of experiments, Minitab was used to obtain the L 9 orthogonal array based on Taguchi Design and it is shown in Table 2. In this study, GRA was used in combination of Taguchi to find out the optimised

processing parameters for producing composite with optimised multi-performance. The normalization of response values was found out using larger the better criteria and grey relational coefficients and grades were calculated. The formula used for normalization of responses using higher the better criteria is shown in Equation 1 (Girish, 2019).

$$x_i(k) = \frac{y_i(k) - \min y_i(k)}{\max y_i(k) - \min y_i(k)}$$ Equation 1

Where $x_i(k)$ = value after the grey relational generation

$\min y_i(k)$ = smallest value of $y_i(k)$ for k^{th} response

$\max y_i(k)$ = smallest value of $y_i(k)$ for k^{th} response

Table 1 Processing Parameters of Compression Moulding

Level	Temperature (ºC)	Pressure (bar)	Time (minutes)
1	170	30	5
2	180	35	10
3	190	40	15

RESULTS AND CONCLUSIONS

The results from the mechanical tests (tensile, flexural, impact) GRA degree and rankings are shown in Table 2. It is observed that the experimental no 4, 3 and 1 are giving the highest tensile, flexural and impact strength. It is evident from the results that no single experimental run will give us the best values for all the properties. Hence, GRA was used to analyse the results and to predict the combination of processing parameters for the preparation of composite with optimum performance in terms of tensile, flexural and impact strengths.

Table 2 L9 Orthogonal Array with their outputs

Experiment Number	Temperature (ºC)	Pressure (bar)	Time (minutes)	Tensile Strength MPa	Flexural Strength MPa	Impact Strength kJ/m^2	GRA Degree	GRA Ranking
1	170	30	5	50.89	52.93	23.62	0.72432	1
2	170	35	10	45.29	102.66	21.00	0.646601	2
3	170	40	15	45.25	112.37	19.69	0.661855	5
4	180	30	10	54.05	92.43	18.37	0.693061	3
5	180	35	15	44.84	97.11	18.37	0.555367	6
6	180	40	5	50.03	106.44	19.69	0.691577	4
7	190	30	15	43.01	78.02	18.37	0.472826	7
8	190	35	5	39.41	77.53	17.71	0.434624	8
9	190	40	10	35.39	42.15	18.37	0.343616	9

Based on GRA results, grey ranks were determined. From Table 2, it was observed that the first experimental run has the first rank and the optimized processing parameters were 170ºC , 30 MPa and 5 minutes to produce the PLA composite with optimized tensile, flexural and impact properties. The results from analysis of variance suggested that the temperature was the most influencing parameter followed by pressure and time.

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ID 123
TOWARD A NEW METHOD, SAFE AND FAST, TO QUANTIFY LIGNIN IN FIBRE PLANTS ELEMENTS

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ABSTRACT
This work compares two methods of analysing the lignin content of plant fibres such as hemp and flax. This polyphenol, composed of three monolignols in different ratios depending on the plants, is one of the main components of vegetable fibers and its rate influences their stiffness and mechanical properties. Recently, a new quantification method was proposed, presenting many technological advantages, but it mainly relies on the molar absorption coefficient Ɛ (L.mol-1.cm-1). The traditional method uses Acetyl Bromide, which is carcinogenic, mutagenic and reprotoxic (CMR) to the user and hazard for the environment, diluted in sulphuric acid, to break down the plant fibres and measure their lignin content by absorbance in a three-hour step. Whereas in the more recent method, lignin solubilisation is assisted by the presence of L-Cysteine, an amino acid, improving the kinetic of the reaction to only one hour. A more suitable estimation technique of the different monolignols present in the plant, which influence the molar extinction coefficient, is used in the study. The results from the two methods are compared and discussed, and conclusions are drawn as to their accuracy and usefulness.

INTRODUCTION
The lignin content method is based on the dissolution of lignin into monolignols, and the Klason method is the current reference approach for measuring insoluble lignin in acid, (TAPPI 2006). However, this technique utilizes hazardous reagents and is both time consuming and damaging to the environment. An improved iteration of this method using acetyl bromide has been developed (Moreira-Vilar, Siqueira-Soares Rde et al. 2014). Despite the benefits of this method, it still utilizes an ecologically unfriendly reagent. A more recent technique to emerge is Cysteine-assisted Sulfuric Acid (CASA) (Lu, Wang et al. 2021) which has been shown to produce comparable results to the Klason method when applied to softwood. This method is, however, reliant on the monolignol G/H/S ratio and the ratio of these molecules is highly divergent in different fiber plants (Lupoi, Singh et al. 2015) such as hemp, flax and jute, and this paper aim to find more suitable values of absorption molecular coefficient (Ɛ) for these plants.
The lignin content of five plant fiber samples were analyzed using the acetyl bromide and CASA (cysteine assisted sulfuric acid assisted) methods. The results of the acetyl bromide method were deemed to be robust. The Ɛ used in the CASA method was calculated using a precise monolignols ratio determined by GC-MS, using thioacidolyse method. (Lapierre 1988)

RESULTS AND CONCLUSIONS

The influence of Ɛ on lignin content results is shown in Fig. 1. where seven different genotypes of hemp have been analysed using CASA method, and compare to the results obtained with the acetyl bromide method. The Ɛ used are these present in the original publication of CASA method, where Ɛ = 12,35 should be applied for G:S ratio superior or equal to 2 (as in fibre plants) and Ɛ = 17,25 L.mol-1.cm-1 should be employed for G-lignin. (Lu, Wang et al. 2021).

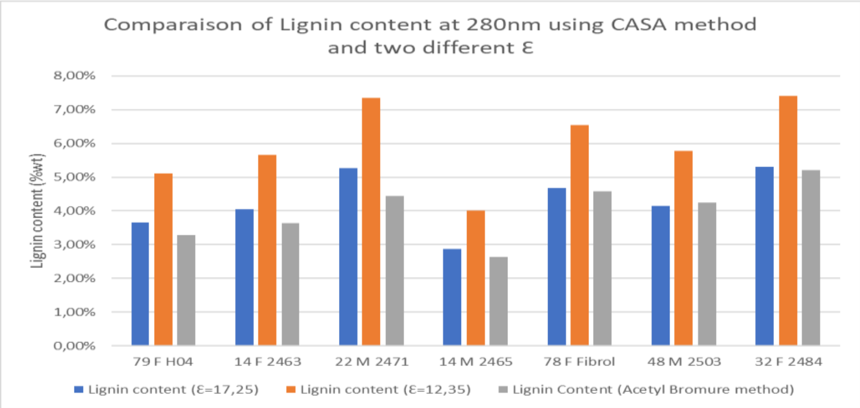

Fig.1 Influence of the Ɛ on the lignin content results in Cysteine assisted method, compare to Acetyl Bromide results, in different varieties of hemp

On average, the discrepancy between values obtained with acetyl bromide and CASA using Ɛ = 17.25 is 8%, while using Ɛ = 12.35, the estimation of the lignin content is overestimated by 50%. There is a significant influence of the molecular absorption coefficient in the measurement of lignin content. Therefore, its determination, derived from monolignol estimation, has to be accurate.

ACKNOWLEDGMENTS

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ID 125
LYSOZYME IMMOBILIZED ON FIBERS OF CELLULOSE SUGAR CANE BAGASSE CELLULOSE FOR MEDICAL APPLICATIONS.

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ABSTRACT
In this study, cellulose fibers from sugarcane bagasse were produced and used as support in the process of immobilization of the enzyme lysozyme. The best immobilization results for the enzyme lysozyme were using the activating agent aminopropyltriethoxysilane and glutaraldehyde at pH 9 for values of 73.4% and 84.6% for total protein content and enzymatic activity, respectively.

INTRODUCTION
In Brazil, sugarcane is one of the largest agricultural monocultures, with an estimated productivity for the 2022-2023 harvest of 572.9 million tons of sugarcane. Most of the sugarcane produced is used to produce sugar and alcohol. Sugar production is estimated at 333.9 million tons and ethanol is 25.83 billion liters (CONAB, 2023). The processing of sugarcane to produce sugar and alcohol generates various agricultural residues, such as straw, bagasse, filter cake, vinasse and wastewater. Of these residues, bagasse is the first in terms of the amount generated. The world production of bagasse is about 54 million tons per year (LIU et al., 2007). Sugarcane bagasse residues can have several applications such as energy production, second generation ethanol should generate strong competition for the use of bagasse with the cogeneration of electricity, the production of biogas (MALUF, 2014; VOLIPI et al, 2023). However, the burning of bagasse presents environmental problems such as, for example, the dragging of unburned particulate matter, known as soot. In addition to the pollution caused by burning, bagasse can generate waste during transport and handling (FORNARI, 1991). Thus, the composition of sugarcane bagasse has driven several research groups to develop technologies aimed at its use (SANTOS et al., 2018; COSTA et al., 2013). Cellulose extracted from sugarcane bagasse can be applied in the textile sector to produce fibers with the incorporation of enzymes to produce bandages for medical applications (COSTA et al. 2020). In this study, fibers produced with sugarcane bagasse cellulose were used as support and three different activating agents (epichlorohydrin (4% v/v) + glutaraldehyde (0,5% v/v), 1-ethyl- (3-dimethylaminopropyl) carbodiimide (EDC) + universal buffer and γ-aminopropiltrietoxisilano (γ-APS) + (0.5%) glutaraldehyde) at pH 7 and 9 were tested. Lysozyme enzyme immobilization reactions by covalent bonding at pH 7 and 9 to 3h of contact were performed and total proteins and enzymatic activity assays were conducted to yield calculate.

RESULTS AND CONCLUSIONS
Total protein and enzyme activity results are shown in the Table. 1. In general, the best results of immobilization of the enzyme lysozyme in the fibers were obtained for pH 9 values for all treatments carried out. The activating agent aminopropyltriethoxysilane and glutaraldehyde in showed total protein values of 73.4% and activity of 84.6% to (4%) epichlorohydrin + (0.5%) glutaraldehyde was 57.1% and 56.6%

and for the EDC were obtained 56.16% and 41.1% of enzymatic activity and total proteins. In this study, treatment with γ-APS showed a 33.68% yield difference in terms of activity when compared to the other treatments. Proving that the immobilization process was efficient, that the enzyme remained active during the connection with the support, and that the ideal pH value for the immobilization process was 9.0, which also shows that it is within the stability of the enzyme.

Table 1 Results of immobilization of lysozyme in fibers produced with sugarcane bagasse cellulose SH/H_2O_2

Activating Agent	pH	Protein yield (%)	Enzymatic activity (%)
(4%) epichlorohydrin + (0.5%) glutaraldehyde	7	36.0	48.0
	9	55.6	57.7
EDC + universal buffer	7	15.1	15.2
	9	41.1	56.1
γ-APS + (0.5%) glutaraldehyde	7	8.5	9.21
	9	73.4	84.6

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ID 126

PLASTIC DEFORMATION BEHAVIOUR OF NATURAL FIBRE COMPOSITES UNDER COMPRESSION LOADING AND THE OFFSET STRAIN

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ABSTRACT

This paper presents the elastoplastic deformation behaviour of enset fibre-epoxy composite under compression loading. The elastic and plastic deformation regions were investigated and the offset strain and yielding strain have been addressed. Fibres were chemically treated to examine improvement in the elastic limit of the composite and the introduced plastic deformation. The total plastic deformation and failure modes were determined. A prediction model has been devised to represent the nonlinear deformation behaviour.

INTRODUCTION

Unidirectional composite materials experience various fracture mechanisms such as micro buckling, fibre kinking, fibre breaking, and longitudinal cracking, under uniaxial compressive loading. In all these deformation behaviours there is the shear deformation of the isotropic polymer matrix which dominantly appears after the elastic limit of the composite structure due to the initial fracture that exists at the boundary of a misaligned fibre (Gutkin et al. 2010). This introduces plastic deformation during continuous progressive loading. Due to such fracture behaviour, fibre composites record lower compressive strength, 50-60%, compared to tensile strength during axial loading on unidirectional composites (Kyriakides et al., 1995).

This study aims to investigate the plastic compressive behaviour of natural fibre composites in uniaxial loading. Samples were produced from untreated and chemically treated enset fibres embedded with epoxy resin. A speckle pattern was generated on the frontside and backside of the 3mm thick samples with an atomized air painter. Samples were placed in a double-stage shear loading compression fixture and a progressive load was applied at speed of 1mm/min. The Euler buckling, strain and elastic modulus were measured by digital image correlation. The elastic and plastic deformation is dichotomized based on the elastic limit computation. The shear modulus of the fibre was back-calculated to give an empirical explanation of the value of the shear plane angle of the kink band during plastic deformation.

RESULTS AND CONCLUSIONS

Compression loading on composite samples results in an elastoplastic deformation with a clear transition point. After experiencing a linear elastic response with a small strain value, the composite structure experiences a significant elastoplastic deformation (Fig.1). The contribution of elastic deformation to the total deformation is around 12%, the remaining is therefore plastic deformation. The elastic limit stress is less than half when compared to the ultimate strength except for NaOH-treated composites (Table 1).

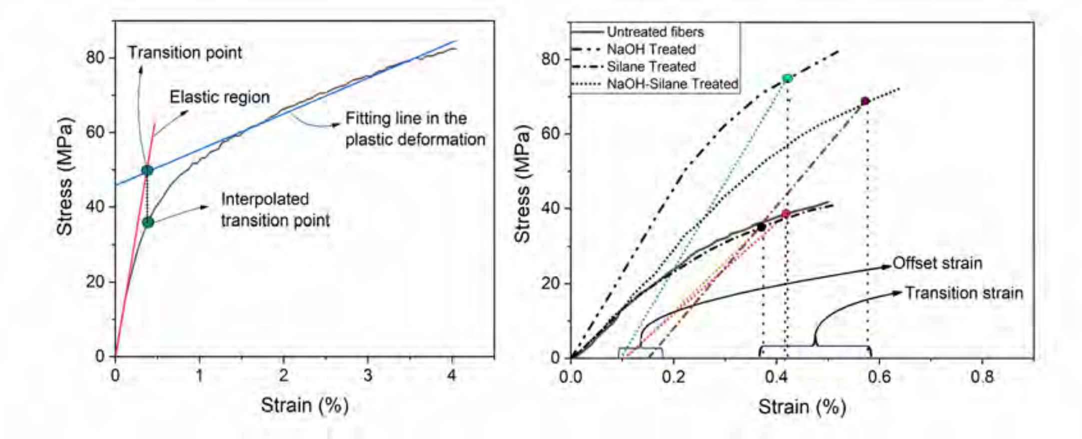

Fig.1 a) Elastic and plastic deformation during compression loading and **b)** the strain values at the elastic limit

Table 1 the elastic and plastic deformation of enset composites and the transition points

Composites	Transition strain (ε_T , %)	Offset strain	Transition Strain percentage	Transition Stress Percentage	Total plastic strain percentage
Untreated FPC	0.42 ±0.06	0.10 ± 0.02	7.23 ± 0.98	44.05 ± 3.81	88.04 ± 2.03
NaOH TFPC	0.43 ± 0.06	0.12 ± 0.01	8.11 ± 1.94	52.76 ± 5.19	89.14 ± 2.43
Silane TFPC	0.39 ± 0.04	0.11 ± 0.01	7.69 ± 0.93	41.20 ± 2.89	87.25 ± 1.89
NaOH-Silane TFPC	0.57 ± 0.06	0.15 ± 0.2	7.21 ± 0.87	48.07 ± 3.51	89.31 ± 1.39

These elastic and plastic deformations are utilized to foster a representative model to predict the stress-strain behaviour of the composite. The constants of the Ramberg-Osgood predictive model were obtained from these deformation values. A fibre kinking was observed in untreated and Silane treated composites with a shear plane angle nearly equal to Mohr's shear plane angle, which is generally less that the angle observed in synthetic fibre composites due to friction stress because of fibre rotation during fibre kinking (Pinho et al. 2005). Therefore, this considerably smaller angle of the kink band demonstrates that the shear modulus of the fibres should be less than or similar to the shear modulus of the matrix. This smaller shear resistance avoids the frictional stress due to fibre rotation during expanding the kink band.

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ID 127
BIODEGRADATION OF NONWOVEN MULCHES PRODUCED BY PLA BIOPOLYMER USING DIFFERENT PRODUCTION TECHNOLOGIES

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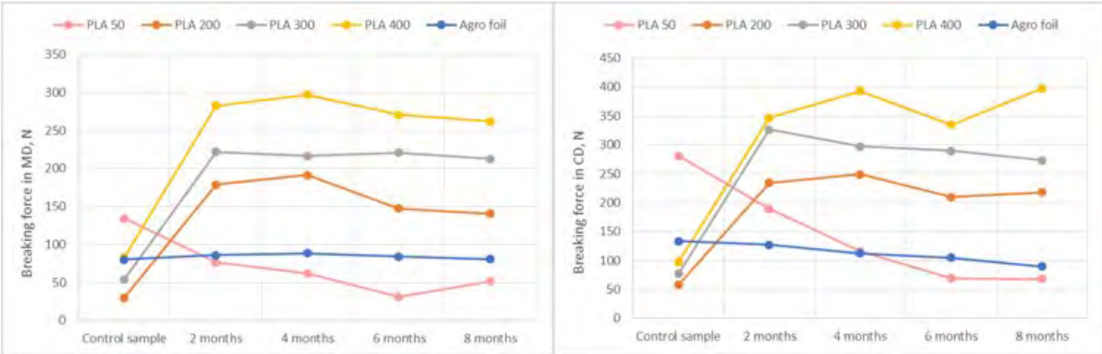
ABSTRACT
The influence of different nonwoven fabric processes mechanically produced nonwoven fabrics bonded by needle punching and spun-bonded, made of PLA polymer on biodegradation were investigated. The results revealed that the production process of non-woven mulches significantly affects the biodegradation process even though the mulches are produced from the same biopolymer. The mechanically produced mulches consist of oriented layers of fibres bonded by needle-punching into one structure where due to environmental conditions different changes of structure, regarding spun-bonded mulch and agro foil, occur.

INTRODUCTION
Plastic agro foil is widely used, thus contributing to agricultural plastic waste and soil pollution. Renewable resources-based biodegradable materials are preferred over petroleum-based materials due to environmental concerns (Yang 2020). PLA polymer made from renewable sources, mostly corn starch, emerged as a potential alternative to plastic films due to its biodegradability, biocompatibility and unique characteristics. The investigations on nonwoven mulches made from biopolymers are mostly related to spun-bond or melt-blown mulches made of PLA polymers and their blends where melt-blown PLA mulches tend to a greater decrease in mechanical properties, i.e. potential biodegradation (Dharmalingam 2016, Hablot 2014). Degradation of the PLA spun-bonded nonwoven mulch after 2 years was still in the early stages, where the degradation rate was dependent on microbes in soil and elevated temperatures (Puchalski 2019).
To investigate the influence of different nonwoven fabric processes, thus their structure, on biodegradability and weed suppression, in May 2022, mulches made of PLA polymer (NaturalWorks Ltd.) and conventional agro foil were placed on the soil by randomly arranged blocks of three replication plots. The mulches were mechanically produced nonwoven fabrics bonded by needle punching from PLA fibres of 250 gm^{-2}, 290 gm^{-2} and 360 gm^{-2}, spun-bonded PLA mulch of 50 gm^{-2}. The biodegradability of mulches by testing mass per unit area, thickness under the pressure of 0.5 kPa and 1 kPa, tensile properties and air permeability every 60 days over a period of 8 months was monitored.

RESULTS AND CONCLUSIONS
The mass per unit area and thickness of all mulches, including foil, increase during 6 months after which

decreasing tendency is visible. Unlike mechanically produced mulches, the small change in mass and thickness is recorded for spun-bonded PLA mulch and agro foil. The breaking force of spunbonded PLA mulches and agro foil decrease from the beginning of biodegradation testing, while the significant breaking force increase of mechanically produced mulches is visible (Fig.1.). The mechanically produced mulches consist of oriented layers of fibers bonded by needle-punching into one structure where due to environmental conditions different changes of structure, regarding spun-bonded mulch and agro foil, occur.



a) b)

Fig.1 Nonwoven fabrics and agro foil breaking force in a) MD; b) CD

This study shows that the production process of non-woven mulches, respectively the structure of non-woven fabrics, significantly affects the biodegradation process even though the mulches are produced from the same biopolymer. Since the layered structure has a significant influence on the results of conventional tests that usually confirm the biodegradability of textile fabrics, it is necessary to carry out further tests of the fiber's biodegradability (SEM, TGA and FTIR analyses) to obtain information on what extent the biodegradation of the fibers within the mechanically produced mulches has occurred.

ACKNOWLEDGMENTS

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ID 129
GREEN REGENERATION: CONVERTING ELECTROSPUN CELLULOSE ACETATE FIBERS TO CELLULOSE

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ABSTRACT
This study presents a novel method for the electrospinning of aligned cellulose acetate fibers and their subsequent regeneration into native cellulose to be used as reinforcement for composite applications. The method involves the use of a 10% (w/w) cellulose acetatesolution in a 1:1 binary mixture of acetone and acetic acid, and the use of potassiumhydroxide (KOH) as a deacetylation agent. The resulting fibers were characterized by SEM, and the successful regeneration of cellulose fibers was confirmed by FT-IR spectra. The findings of the study indicate that further optimization of the solution content and electrospin-ningprocesscouldleadtotheproductionofhigh-qualitycellulosefiberswith great potential applications in green technology.

INTRODUCTION
Electrospinning is a versatile and cost-effective method to produce micro to nano scale fibers with a large surface area to volume ratio, high porosity, and flexibility in surface characteristics. The production of aligned cellulose fibers has been of great interest in various fields due to their unique properties and potential applications [1]. However, the electrospinning of cellulose into fibers has proven to be a challenging task due to the high viscosity and poor solubility of cellulose in common solvents. The high molecular weight of cellulose also makes it difficult to dissolve in common organic solvents. Cellulose molecules are large and entangled, making it difficult to separate them and dissolve them in a solvent.
To overcome these limitations, cellulose acetate (CA) fibers are often electrospun and then deacetylated to regenerate the native cellulose fibers [1,2]. Cellulose acetate is a modified form of cellulose in which some of the hydroxyl groups are replaced by acetyl groups. This modification reduces the hydrophilicity of cellulose and makes it more soluble in common solvents, [2] such as acetone and acetic acid, which can be used in electrospinning.
The regeneration process in this study, involves the use of potassium hydroxide (KOH) as a deacetylation agent to remove the acetyl groups from the cellulose acetate fibers, ultimately reforming the native cellulose. The KOH reacts with the acetyl groups, breaking the esterbond and regenerating the hydroxyl groups, thus creating cellulose fibers. By using this approach, it is possible to benefit from the advantages of electrospinning process while obtaining aligned cellulose fibers. The alignment of fibers improves the mechanicalproperties, increases the surface area, and enhances the performance of the material.

RESULTS AND CONCLUSIONS
In this study, a solution of 10% (w/w) cellulose acetate in a 1:1 binary mixture of acetone and aceticacid-wassuccessfullyelectrospun.ThefiberswerecharacterizedusingaScanning

Electron Microscope (SEM) and the micrograph (Fig 1. (a)) shows their alignment with an average diameter of 1 µm. The cellulose acetate fibers were then deacetylated using KOH, to convert it to cellulose.
A Fourier Transform Infrared (FT-IR) was performed on the sample and a change in peaks of the spectra (Fig 1. (b)) confirmed the success of the deacetylation and cellulose regeneration process.IntheFT-IRspectraofthedeacetylatedsample,thepresenceofregeneratedcellulose was confirmed by the decrease of peaks attributed to acetate groups at 1745 cm^{-1}, 1375 cm^{-1}, and 1235 cm^{-1}, and the appearance of peaks at 3300 $cm^{-1}$and 1100 cm^{-1}indicating the presence of hydroxyl and ether groups.

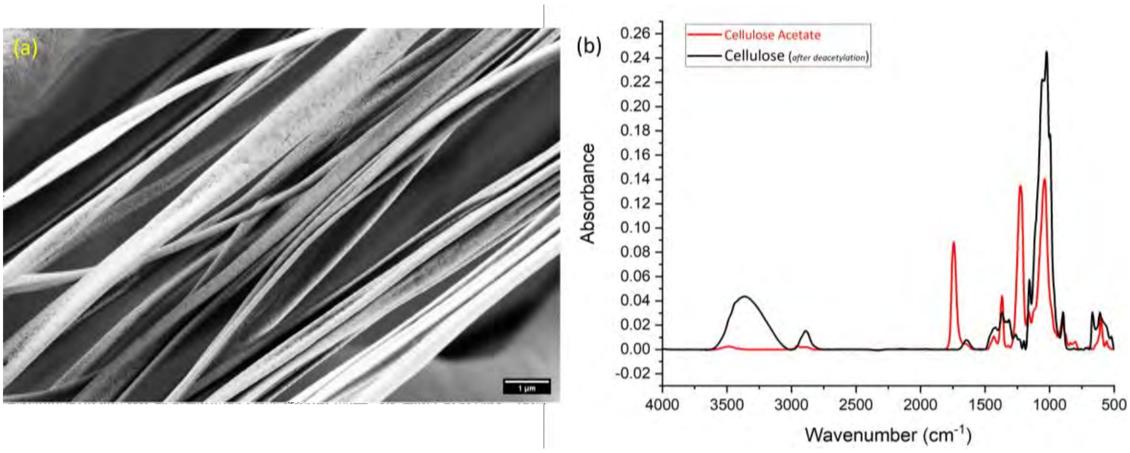

Figure1.(a) A SEM micrograph of Cellulose acetate fibers: **(b)** FT-IR spectra of the electrospun cellulose acetate fibers and the regenerated cellulose after deacetylation.

The results obtained are encouraging and indicate that further optimization of the electrospinning process can lead to the development of high-quality cellulose acetate fibers. The next step forward in this research would be to electrospin cellulose acetate fibers with aligned cellulose nanocrystals (CNCs) inside them. These CNC filled aligned fibers can then be regenerated back to cellulose, which would improve its properties and open doors to many potential green applications. The ultimate goal of this work in the framework of BioCel3D project is to incorporate an aligned electrospun cellulose-based reinforcement for continuous fiber 3D printing, resulting in the creation of a technical natural fiber with improved hierarchical organization and enhanced physical properties.

ACKNOWLEDGMENTS

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ID 131
PRODUCTION OF ALGINATE, CHITOSAN AND ALGINATE/CHITOSAN FIBERS: EVALUATION OF PHYSICAL-CHEMICAL AND MORPHOLOGICAL PROPERTIES AND CYTOTOXICITY

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ABSTRACT
In this work, alginate, chitosan and alginate/chitosan fibers were produced and their physical-chemical, morphological and cytotoxic properties were evaluated. The fibers showed values ranging from 4 to 10 cN/tex, and the use of glycerol is associated with a downward trend in tenacity values. The water absorption capacity values were above 100% and maximum weight loss of 28% in a period of 30 days. The fibers did not show cytotoxicity, being promising for biomedical applications.

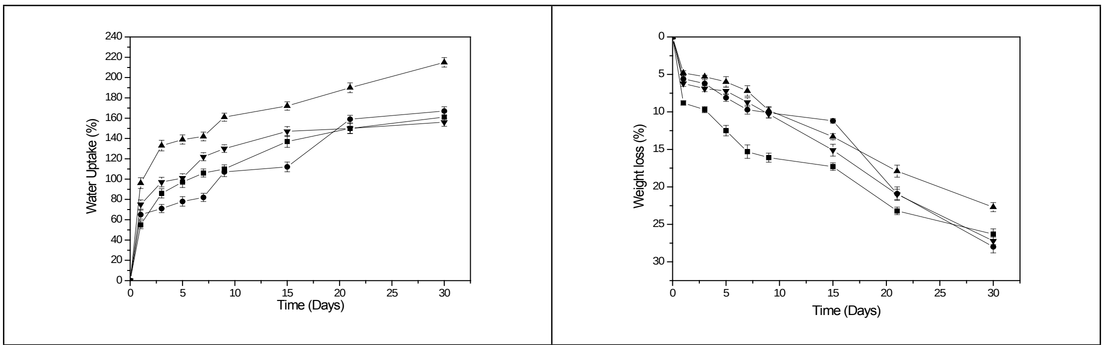
INTRODUCTION
In the medical area, textile materials are increasingly important in many applications of fibers, fabrics, nonwovens in the manufacture of medical protective clothing, implants, non-implants and extracorporeal devices. For example, a healing bandage must protect the wound from further damage, as well as relieve pain, absorb exudates and stop bleeding, accelerate healing, keep the wound at an ideal temperature, pH level and moist (free of infection and slough). excessive), maintaining high humidity at the wound/dressing interface, allowing gas exchange, free of toxic chemicals, particles or fibers that could be released from it and undisturbed by its structural changes. It should also be non-allergenic, non-toxic and easily removed. Thus, the use of biomaterials with antimicrobial and healing properties is interesting (Brown , Tonelli, 2001; Croisier, Jérôme, 2013.
In this work, alginate and chitosan were used separately and together to produce fibers. Alginate is biocompatible, has a relatively low cost, does not present toxicity and can form gels by adding divalent cations such as, for example, Ca^{2+} (Lee, Mooney, 2012).
Chitosan is a polymer obtained from the deacetylation reaction of chitin, whose chitin is extracted from the exoskeleton of crustaceans. Chitosan is an important abundant renewable resource and has some properties such as: biodegradability, good compatibility, low toxicity, great versatility to form films, gels, fibers, granules, scaffolds and has antimicrobial activity, showing potential for medical applications (Croisier, Jérôme, 2013).

RESULTS AND CONCLUSIONS

The results from the tensile tests are shown in Table1 and the Fig1 water uptake and weight loss.

Table 1. Tension test results

Fibers	Count number (tex)	Breaking load (N)	Tenacity (cN/tex)	Elongation (%)	Young´s Modulus (N/tex)
Alginate (Al)	27.6 ± 1.34	1.62 ± 0.11	7.25 ± 0.52	4.89 ± 2.43	4.77 ± 0.49
Alginate (AlGl)	29.2 ± 1.39	1.35 ± 0.07	7.75 ± 0.44	6.08 ± 1.85	5.02 ± 0.47
Chitosan (Ch)	17.2 ± 0.7	0.77 ± 0.05	4.47 ± 0.28	1.45 ± 0.97	4.16 ± 1.01
Chitosan (ChGl)	17.2 ± 0.65	1.49 ± 0.08	8.64 ± 0.51	4.25 ± 0.83	5.55 ± 0.41
Hybrid (H)	18.4 ± 0.82	1.64 ± 0.10	8.89 ± 0.55	5.44 ± 2.4	5.66 ± 0.77
Hybrid (HGl)	15.1 ± 0.79	1.53 ± 0.05	10.0 ± 0.33	6.17 ± 1.93	5.72 ± 0.54



(a) (b)

Fig.1. (a) Water uptake (%) and **(b)** Weight loss (%) versus time. Al (-■-), AGL (-●-) H (-▲-); H_GL (-▼-) fibers.

This study shows that the fibers had tenacity values ranging from 4 to 10 cN/tex, and the use of glycerol is associated with a downward trend in tenacity. The water absorption capacity values were above 100% and maximum weight loss of 28% in a period of 30 days.

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FUNCTIONALIZATION OF FLAX FIBERS TO DEVELOP ELECTRORESPONSIVE HYGROMORPH BIOCOMPOSITES

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ABSTRACT
The present study endeavors to investigate the feasibility of flax fibers as a material for the fabrication of electroresponsive hygromorph biocomposites (HBCs). Through a systematic approach, the homogeneous functionalization of flax yarns was optimized by conducting in-situ polymerization of either pyrrole (Py) or 3,4-Ethylenedioxythiophene (EDOT) monomers under controlled strain and humidity conditions. Additionally, the study evaluated the kinetics of sorption/desorption of the functionalized fibers using dynamic vapor sorption (DVS), as well as the Joule heating effect through the utilization of a high-resolution thermal camera of 20 m°K. The outcomes of this research could provide valuable perspectives on the properties and practical applications of HBCs in areas such as smart textiles, energy harvesters, and sensors.

INTRODUCTION
Cellulose-based composites have been garnering increasing attention as potential alternatives to more traditional materials due to their versatile properties and eco-friendliness. In particular, the modification of cellulose fibers, to exhibit conductivity, presents a multitude of potential applications from organic electronics and sensors to antistatic materials [1]. Among the various cellulose-based fibers, lignocellulosic fibers such as flax have been demonstrated to possess exceptional mechanical properties such as high tensile strength, good toughness, and stiffness, properties that are closely linked to the nature of cellulose and its crystalline structure [2]. These fibers are currently being evaluated as environmentally friendly and cost-effective alternatives to traditional glass for the use in polymer composites and concrete reinforcement. Recent research from Le Duigou et al. [3,4] has resulted in the development of HBCs utilizing designs inspired by the bilayer microstructure of natural hydraulic actuators. According to their research, flax fibers have been identified as the most suitable candidates for the fabrication of HBCs.

RESULTS AND CONCLUSIONS
The infrared (IR) thermal imaging analysis of a yarn functionalized with polypyrrole (Ppy) upon voltage application is depicted in Fig.1.A. The results indicate a maximum heating temperature of 62.5°C achieved at 5V applied voltage, with a uniform temperature distribution evident from the cylinder-shaped composites exhibiting a red interior layer, signifying homogeneous functionalization of PPy on the fiber surface. SEM analysis of the elementary fibers of the same functionalized yarn, presented in Fig.1.B, provides further confirmation of the uniform distribution and adhesion of PPy.

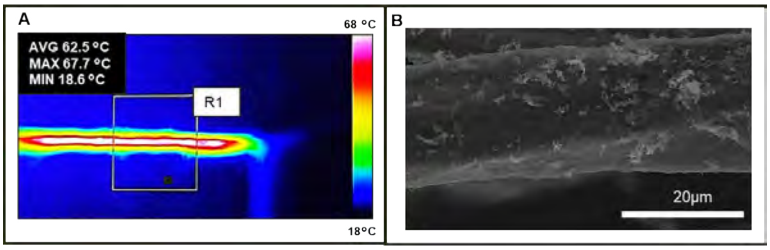

Fig.1.A. Infrared thermal image of PPy coated on flax yarn, of linear density 68 TEX, after applying certain voltage ;
Fig. 1.B.SEM image of the Ppy functionalized surface of the elementary fiber of the same yarn

An comprehensive evaluation of the hygroscopic behavior of yarns pre- and postfunctionalization necessitates the utilization of the DVS analysis to determine alterations in mass as a result of fluctuations in relative humidity, with a focus on adsorption or desorption. The DVS results depicted in Fig. 2 for poly(3,4-ethylenedioxythiophene) (PEDOT) and Ppy functionalized yarns, of 1:6 mass fraction of flax/polymer, evince that these composite materials possess a superior capacity for water adsorption as compared to non-functionalized yarns.

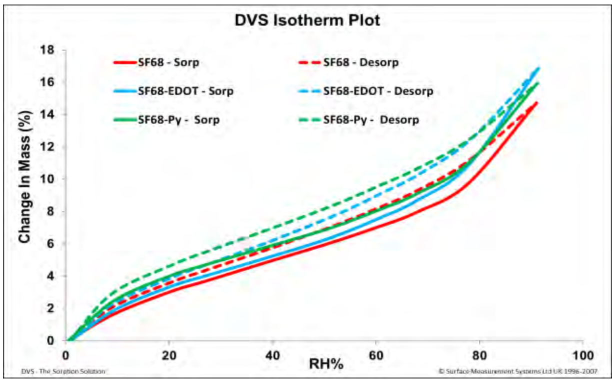

Fig.2. Desorption/sorption of the flax fibers from Safilin supplier (SF) of linear density 68 TEX : non functionalized, functionalized with poly(3,4-ethylenedioxythiophene) (PEDOT) or polypyrrole (Ppy)

This project presents a significant prospect for the development of a new generation of sustainable, shape-shifting electroresponsive biocomposites that can be used to create 3D printed architected materials which are capable of undergoing changes in shape or properties in response to temperature and humidity stimuli.

ACKNOWLEDGMENTS

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ID 138
HYGROSCOPIC DURABILITY OF FLAX FIBRE COMPOSITES WITH DIFFERENT MATRICES UNTER CYCLING CONDITIONS

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ABSTRACT
The flax fibre reinforced composite industry is rapidly expanding through high-performance structural materials with minimum impact on the environment, compared to the traditional synthetic fibre composites [1]. Despite this high potential, the inherent attraction of flax to moisture due to its composition [2] makes necessary the study of the mechanical performance under hygroscopic conditions. However, in realistic environments the long term moisture exposure is not stable but can be greatly affected by the location and the seasonal time e.g. in tropical climates [3]. It is generally accepted that for most outdoor environments in Europe the relative humidity (RH) level will fluctuate throughout the year between a high and a low value in winter and summer, respectively. This high-low cycling RH exposure introduces damage formation due to the cyclic swelling and shrinking of the fibres which are constrained inside the composite's matrix. This study focuses on this type of cyclic environmental loading while the flexural performance of the flax fibre composites with different matrices is monitored. One cycle consists of 1 week at 80% RH followed by 1 week at 30% RH while the temperature is constant at 60°C to accelerate the moisture diffusion. Prior to flexural testing the composite specimens were left to saturate at standard conditions at 50% RH and 21°C.

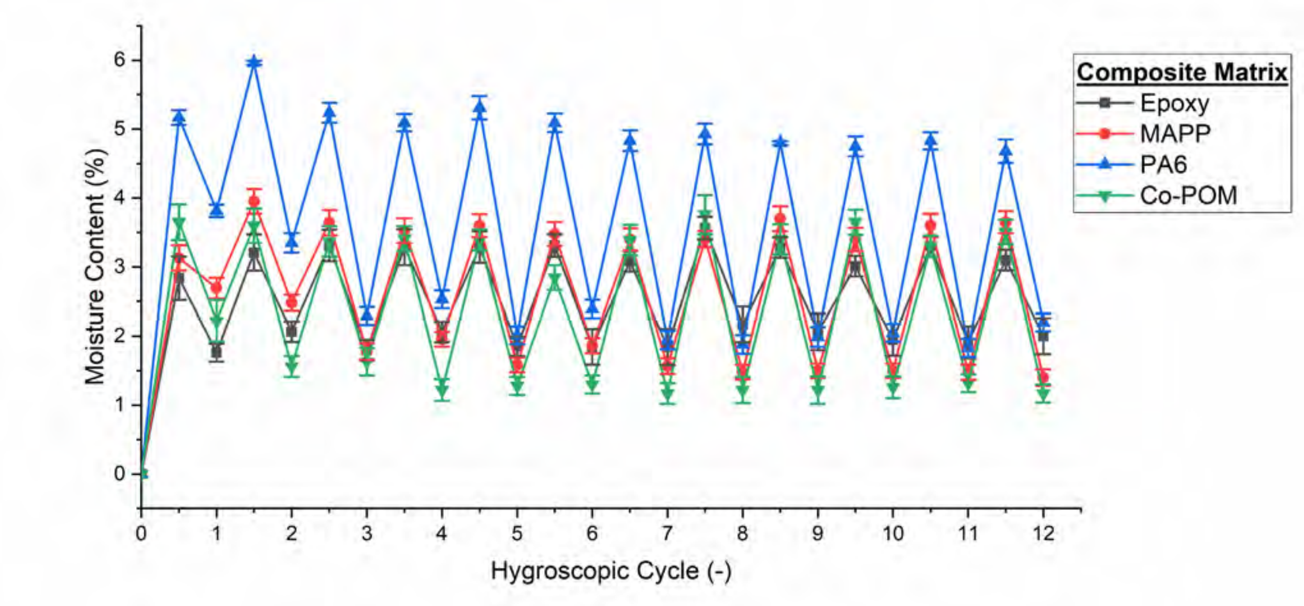

Fig.1 Moisture content at the end of each half cycle.

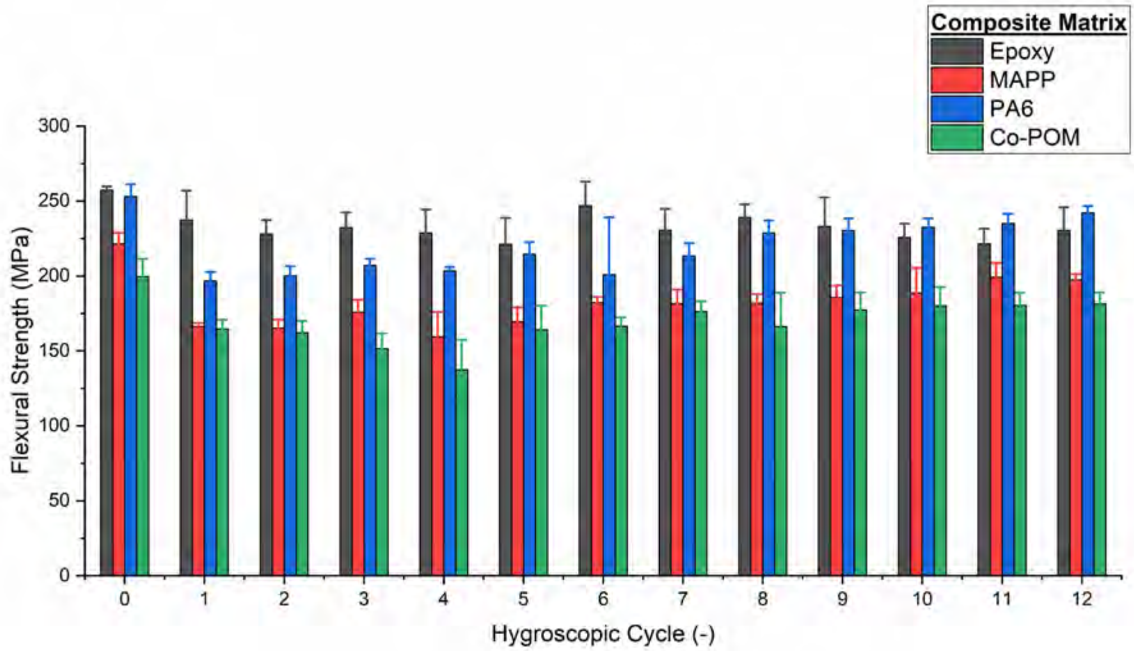

Fig.2 Flexural strength as function of the hygroscopic cycling

The results indicate that although after the first cycle there is a drop in mechanical properties, there is little degradation in the following cycles. This could be explained by monitoring moisture content (Fig. 1) and since the systems do not absorb higher water content in the following cycles and by extension they do not swell more, this would not create additional damage. Furthermore, a strengthening mechanism is being observed, particularly in the later stage of cycling, particularly for all thermoplastic matrices. This moisture self-strengthening phenomenon has been recently observed by Lu et al. [4] and been attributed to a similar hornification treatment in wood.

ACKNOWLEDGMENTS
The authors are sincerely grateful for the financial support of European Union's Horizon 2020 research and innovation programme provided within the framework of the Marie Skłodowska-Curie FibreNet project - grant agreement No. 764713

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ID 140
BIODEGRADATION, MECHANICAL AND MORPHOLOGICAL PROPERTIES OF PLANT FIBRES

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ABSTRACT
The paper presents the biodegradation, mechanical and morphological properties of plant fibres. An analysis of the biodegradation properties was performed by a simple soil burial test conducted under controlled conditions in the laboratory, for 2, 4, 7, 9 and 11 days. Mechanical properties of control and buried samples were determined on tensile testers Vibroscop and Vibrodyn 400. Morphological analyses were performed by using a scanning electron microscope at a magnification of 1000x. With the purpose of a better understanding of biodegradation, the number of total fungi and bacteria in the soil was also determined.

INTRODUCTION
Plant fibres are favourable resources for industrial applications, since they are ecologically efficient with high tenacity and possess the properties of biodegradability, renewability and sustainability. Plant fibres contain cellulose but differ in chemical composition, crystallinity, degree of polymerization and fibre production method. Furthermore, in addition to cellulose, plant fibres also contain various non-cellulosic admixtures in different percentages. Differences in chemical structure and physical characteristics between plant fibres affect the overall biodegradation process, which ultimately results in their different level of biodegradability. Therefore, it is important to investigate the biodegradation behaviour of plant fibres and their influence on their mechanical and morphological properties.
An analysis of the biodegradation properties of Hemp, Jute and Sisal fibres was performed by a simple soil burial test (farmland soil), conducted under controlled conditions in the laboratory for 2, 4, 7, 9 and 11 days. With the purpose of a better understanding of biodegradation, the total number of fungi and bacteria in the soil was also determined. Mechanical properties (tenacity) were determined on tensile testers Vibroscop and Vibrodyn 400. Morphological analyses were performed by using a scanning electron microscope at a magnification of 1000x.

RESULTS AND CONCLUSIONS
The mass loss percentage (Figure 1a) in all investigated plant fibres were in a linear relation with the progression of fibre biodegradation. Sisal fibres have the least decrease in mass loss (Figure 1a) and tenacity (Figure 1b) which can be explained by a small amount of cellulose, and about 11-19 % of lignin (which preserves fibres chemical structure). Since Hemp fibres have a large amount of cellulose (responsible for the fibres' exceptional mechanical properties) and small amount of lignin (approx. 6%), under influence of large amount of fungi in the soil, result in the greatest decrease in mass loss (44.51%) and tenacity

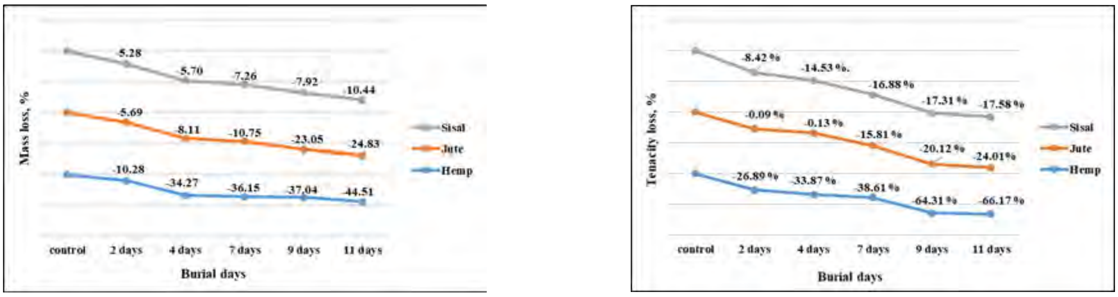
(66.17%).

Biodegradation,

Table 1 Determination of the number of total fungi and total bacteria after 11 days of burial in the soil

	Fungi		**Bacteria**	
	x̄, (CFU/ml)	**stDEV**	**x̄, (CFU/ml)**	**stDEV**
Control	8.50E+04	7.55E+03	1.28E+07	7.64E+05
Hemp	9.03E+04	3.21E+03	1.25E+07	5.03E+05
Jute	1.03E+05	3.21E+03	1.42E+07	4.36E+05
Sisal	9.07E+04	2.08E+03	1.78E+07	1.13E+06

Figure 1 Mass loss **(a)** and tenacity decrease **(b)** of fibres after burial in the soil

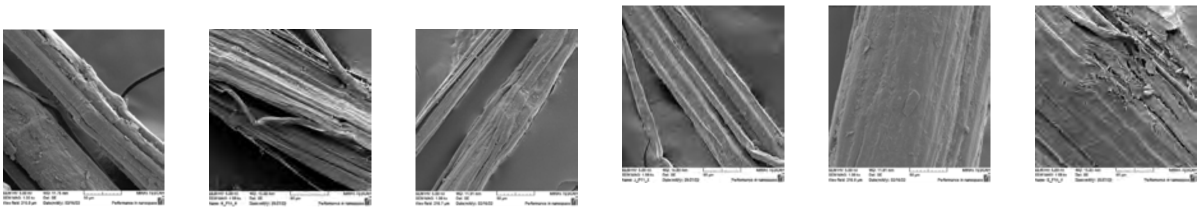


(a) **(b)**

The obtained results were confirmed by the surface microscopic images (Figure 2) of tested fibres before and after the biodegradation processes.

Figure 2 Morphological analysis of fibres after 11 days of burial in the soil

Hemp **Jute** **Sisal**



Control **11 days** **Control** **11 days** **Control** **11 days**

The obtained results are significant for further specific research, considering that they indicate the speed and level of biodegradation of individual plant fibres, which is useful for making the selection for specific purposes in the end-use application.

ACKNOWLEDGMENTS

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ID 141
TO STUDY THE SUGAR CANE BAGASSE AND WASTE SHORT BANANA FIBRE MATERIAL PRODUCE OF REINFORCED NATURAL COMPOSITES SHEET

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ABSTRACT
Nowadays, natural fibre reinforced polymer composites are employed in a variety of technical applications to improve the product's strength, weight, and price. A coarse and powerful natural fibre, sugar cane bagasse and waste short banana fibre is utilized more and more in composite materials for reinforcement in the automotive, textile, furniture, and construction industries as well as in plastics and paper products. In this study, an epoxy resin mixture hand lay-up process was used to produce a fiber-reinforced polymer composite made of discarded short bananas and sugar cane bagasse. The investigation found that all of the metrics are considerably impacted. The overall findings show that composite sheets with a 40% fibre pulp and a 60% epoxy resin composition have the maximum tensile strength, whereas composite sheets with a 60% pulp ratio and 40% epoxy resin composition have better impact strength.

INTRODUCTION
The majority of developed nations now give environmental issues significant consideration. The best utilization of natural resources is a key component of some of the most significant environmental protection measures. Polymeric-based composite materials are now employed in a wide range of industries, including construction, electrical, automotive, sporting goods, marine, and home appliances. Great strength and stiffness, light weight, and high corrosion resistance are all characteristics of polymeric composites (S.M. Sapuan at al. 2015, Srinivas, K et al. 2017, Andrzej K et al. 2001). In order to create robust and lightweight materials, polymers can be reinforced with natural fibres, which are abundant in nature. A sugar cane bagasse and waste short banana fibre pulp reinforced epoxy composite material has been created in the current research effort with a mixture of 60%, 50%, and 40% epoxy resin and 40%, 50%, and 60% banana fibre pulp. To investigate the attributes of the composite sheet's thickness, density, impact strength, and tensile strength, experimental work was conducted. In the current research, an epoxy composite material sheet reinforced with waste short banana pulp and sugar cane bagasse has been used in percentages of 40%, 50%, and 60%. However, prior research, as seen in studies, reportedly used 15% to 35% banana pulp fibre composite sheet. Boat hulls, surfboards, athletic items, and construction panels are all made of epoxy matrix, which not only lowers costs but also prevents environmental pollution. The aim of this study is utilized agricultural waste which may be profitable, pollution free and economically viable for the farmer

and industries. These composites are also used in panel for partition and false ceiling, wall sheet, floor, window and door frame.

Table 1 Specimen composition

S. No	Thickness (mm)	Fiber pulp ratio (%)	Epoxy (%)	Hardener (%)
1.	4	40	60	10
2.	4	50	50	10
3.	4	60	40	10
4.	8	40	60	10
5.	8	50	50	10
6.	8	60	40	10

Figure 1: (a) Banana fibre **(b)** sugar cane bagasse fibre **(c)** Composite sheet

RESULTS AND CONCLUSIONS

The study's findings demonstrate that by combining waste short banana fibre pulp and sugar cane bagasse as reinforcing agents for the polymer of the composites' tensile strength and impact strength, a practical composite with good qualities can be effectively developed. Finding the ideal composition and manufacturing procedure for such a composite material is the result of this approach. This study examines the mechanical characteristics of epoxy resin composites made from waste short banana fibre and sugar cane bagasse. The following results are from experimental and analytical examination of pulp or resin composites.

Higher density of composite sheets is produced by a reduction in the pulp proportion. As we can see from the pulp to resin ratio, the tensile strength decreases as the percentage of pulp rises. Lower pulp content increases the impact resistance of composite sheets. The purpose of this study is to find ways to use agricultural waste that are profitable, clean, and practical for farmers and businesses. These composite materials are also used to make window and door frames, wall sheets, floor coverings, and partition and false ceiling panels.

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ID 142
ROBOTIZED 4-DOF DIC SYSTEM FOR STUDYING THE MECHANICAL BEHAVIOUR OF NATURAL FIBRES AT THE MICRO SCALE

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ABSTRACT
The present study presents the evaluation of a 4-DOF (Four Degrees of Freedom) DIC system for the characterization of the mechanical behaviour of natural fibres at the microscale, specifically for studying two hierarchies (technical and elementary). The developed system contributes to deeply understand the failure mechanisms of natural fibres at different microscales through digital image correlation analysis (DIC) during tensile testing, particularly for flax fibres in the present study. The equipment allows a precise characterization with clear strain fields ranging from 100 μm x 100 μm (technical) to 10 μm x 10 μm (elementary) with a maximum resolution of 50 nm/pixel. The obtained strain maps permitted to track the damage states until the end of the fracture propagation. This study opens a gate for future development of machine learning algorithms based on images for bioinspired designs of fibres and the development physics informed models.

INTRODUCTION
Natural fibers such as bamboo, flax and hemp are being incorporated in sustainable polymer composites manufacturing due to its contributions in the lightweight design, mechanical properties, and circular economy. However, their mechanical behavior has not been accurately understood at two hierarchies (technical and elementary fiber level). Remarkable differences are evidenced in the tensile strength of natural fibers; bamboo (391-700 MPa), flax (345-1035 MPa), since these have been estimated from a global approach [1]. However, using a more complex DIC analysis in hemp fibers in tensile loading allowed the direct association of the middle lamella composition with the shear strength between elementary fibers, which determines the strength of the fiber at the technical level [2].
The present work exhibits a novel 4DOF robotized camera system with long distance magnification capacity to apply the digital image correlation technique in natural fibers at the microscale during tensile testing. A tensile testing machine (INSTRON 5967) was implemented for applying the force. A Motion system conformed by 3 axis motorized stages coupled to a rotation stage (Zaber High precision XYZ motion system) was implemented to control the position of a CCD monochrome camera (Imperx B3440 9Mp) coupled with a high magnification zoom lens (12x zoom, Navitar Inc, rear converter: 2X, magnification: 3.2-28x; working distance: 86 mm). Linear micro step size 0.05 μm for the XYZ directions and rotation step size of 8 μradians permitted to position the camera on initial position for capturing the images. Using the camera coupled to the stages motion control a resolution of 50 nm/pixel was achieved. The present work permits

equipment capacities not available in the market in terms of control, manipulation, and time efficiency to measure samples such as natural fibers at the microscale during mechanical testing.

RESULTS AND CONCLUSIONS

Two groups of ten technical and ten elementary flax fibers were selected for our study with a mean diameter of 20 and 110 μm for the elementary and technical, respectively. A gauge length of 5 mm and 0.1 mm/min were configured for the experiment. A young modulus of 55.2and 65.7 GPa was determined for the technical and elementary level with tensile strength values of 807 and 1011 MPa, respectively. DIC results showed 2.1% of strain before fracture in a technical fiber and 3.9% for the elementary according with the localized strain analysis in both fibers in the direction of the applied force.

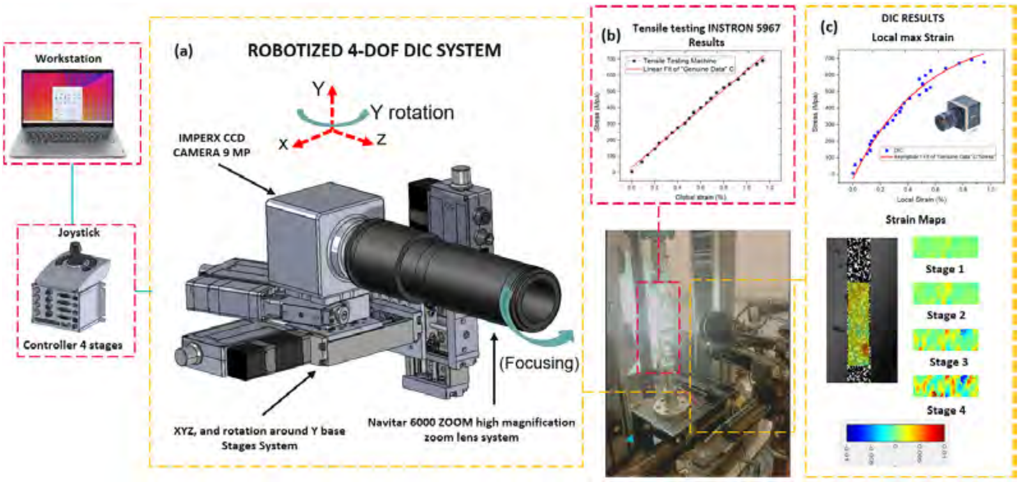

Figure 1. Experimental setup of the fibre strain monitoring robotized 4-DOF DIC system.

The proposed system allowed the application of DIC technique at the microscale allowing the analysis of a clear strain field ranging from technical to elementary size level. Parameters such as longitudinal and lateral strain rate, as well as stress concentrations were tracked and associated to the failure mechanisms in the studied stages. Future works will be developed using the robotized 4DOF DIC system applying simultaneous machine learning methods based on image databases for bioinspired and optimized designs of new type of fiber architectures [3].

ACKNOWLEDGMENTS

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ID 143

CARDOON (CYNARA CARDUNCULUS L.): A RENEWABLE RAW MATERIAL FOR CONSTRUCTION INDUSTRY

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ABSTRACT

The present works evaluates the use of lightweight particleboards of cardoon (here labelled as ADAPTIVE) as alternative material to construction industry. The density of composite materials evidenced a determinant role in their compression resistance. Additionally, thermal insulation properties of ADAPTIVE panels indicate that lightweight cardoon particleboards might be applied in construction industry, in particular for the retrofit of building roofs.

INTRODUCTION

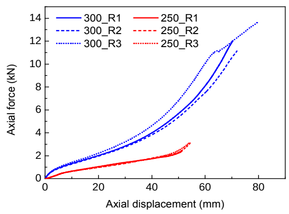
Cardoon (*Cynara cardunculus L.*) is a native plant of the Mediterranean region, with great application in the manufacturing of ewes' milk cheese in Portugal due to the high content of proteases and coagulant factors present in the aqueous extract of cardoon flowers (Monteiro, 2020). However, the remain parts of the plant, as branches and stalks, have no particular use yet, being considered residues. The lignocellulosic character of this type of material associated to its lower density (70 - 220 kg m^{-3}) turns them an excellent candidate to apply in lightweight composite materials, capable of being used in several fields, as construction industry.

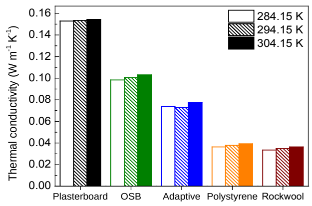
The project ADAPTIVE aims to develop a new advanced production system for the retrofit of building roofing, which will enable the reduction and potential elimination of the accumulated environmental impact of its production chain, substantially increasing productivity for the construction industry. This prefabricated lightweight particleboard, based on cardoon, will be used as a component for the retrofit of flat roofs in current buildings. The purpose is to increase the building's energy performance, collect and store rainwater, and provide for the use of the roofing area as a gardened leisure space.

Lightweight particleboard panels (labelled as ADAPTIVE), with 500x500x90 mm, were produced using cardoon particles (2 mm - 500 µm) and PU resin. The selected ratio between cardoon and resin was 0.45:0.55 (w/w). The panel production followed a simple procedure of blending wood-based raw material with the respective resin and placing the mix into a handmade mold, to reach the desired densities. The resistance to compression (EN 789) and insulation properties (EN 12667) of obtained materials were determined according to the standard procedures.

RESULTS AND CONCLUSIONS

ADAPTIVE panels were subjected to mechanical test to access the impact of density in the axial compression resistance (Fig. 1a). It was observed that panels with 300 kg m^{-3} can support about 11 - 14 kN of axial force, while panels with 250 kg m^{-3} of density only achieved 3 kN (c.a. three times lower).





a) b)

Fig. 1. a) Axial compression test of ADAPTIVE panels with 250 kg m^{-3} and 300 kg m^{-3}. Three specimens (R1, R2 and R3) of each material were tested. **b)** Thermal conductivity of ADAPTIVE panel (300 kg m^{-3}) and other conventional construction materials at 284.15 K, 294.15 K and 304.15 K.

Higher quantity of the mixture cardoon/PU resin increase the stiffness of the composite panel, which may improve its resistance to compression. Additionally, the best performing material (300 kg m^{-3}) was selected for further studies on thermal conductivity and it was compared to some conventional construction materials (Fig. 1b). ADAPTIVE panel present interesting results, with better insulation capacity than plasterboard and oriented strand board (OSB). However, when compared to the most common insulation materials (rockwool and polystyrene), ADAPTIVE panel has lower performance. As concusion, ADAPTIVE panels might be a valuable alternative to incorporate in the retrofit of building roof (Fig. 2).

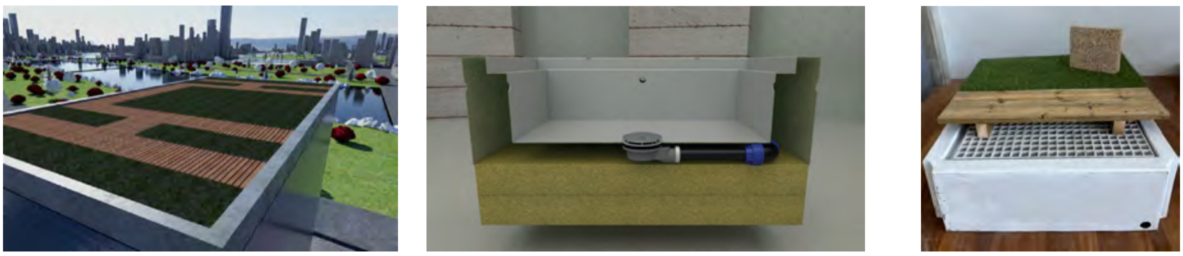


Fig. 2 ADAPTIVE: Retrofit of a building roofing with a gardened leisure space using the lightweight composite

ACKNOWLEDGMENTS

This work was financially supported by: LA/P/0045/2020 (ALiCE), UIDB/00511/2020 and UIDP/00511/2020 (LEPABE), funded by national funds through FCT/MCTES (PIDDAC) and Project ADAPTIVE – Projects in co-promotion ref. POCI-01-0247-FEDER-050191) in the scope of Portugal 2020 co-funded by FEDER (Fundo Europeu de Desenvolvimento Regional) under the framework of POCI (Programa Operacional Competitividade e Internacionalização).

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ID 148

ENHANCING TO REINFORCEMENT OF GREEN COMPOSITE AEROGELS USING WASTE-BASED NANOCELLULOSE

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ABSTRACT

This work investigated the reinforcing effect of cellulose nanostructures (CNS) obtained from lignocellulosic wastes on corn starch aerogels. A highly porous aerogel structure wasreceived, with densification of the aerogel structure. This effect contributed to an increase of 48, 39, and 127 times in compressive strength compared to aerogel without CNS. However, compressive modulus decreased due to voids and pores on the internal aerogel structure.

INTRODUCTION

Aerogels and high-performance materials development have recently seen increasing interest and research. Hybrid aerogels have organic and inorganic precursors in their constitution;they are multicomponent materials with improved performance and multifunction due to the synergistic effect between the organic and inorganic structures that compose them. Within the class of hybrid aerogels, composite aerogels included polymeric systems and/or inorganic polymeric systems, obtaining greater efficiency than the counterpart of their raw materials (Liu, 2020). The nanocellulose is the promising dispersive phase to develop composites. However, nanocellulose dispersion on the polymeric matrix is a broad challenge due to the high density of hydroxyl groups. It can favor the agglomeration of nanostructures (Chen, 2021). If nanocellulose is well-dispersed, a synergistic effect between two structures can be generated, expanding the application areas. This work investigated the development of agreen composite aerogel of corn starch-containing cellulose nanostructures derived from eucalyptus wastes and the reinforcement effect of CNS in the composite aerogel.

RESULTS AND CONCLUSIONS

The addition of CNSs became the starch aerogel less rigid and softer to the touch, like polyurethane foam. The apparent density and porosity (Table 1) are consistent with the existing literature on the subject, whose density varies between 0.1 and 0.25 g/cm3; however, the porosity should reach the range of 85% (Ganesan, 2018). Besides, they obtained shrinkage rate values between 11 and 13%, resulting in a stable structure without the collapse of the 3-D structure (Vareda, 2018).

Table1. Density, Porosity, and Mechanical Compressive Performance Values of composite aerogels without and containing CNS.

Samples	Density(g/cm^3)	Porosity(%)	Compressivemodulus(kPa)	Compressivestrength(kPa)
AeroCNS0%	0.08± 0.02	90±3	100.5± 0.7	33.4± 0.7
AeroCNS1%	0.26± 0.03	67±3	53.0± 2.0	1603± 430
AeroCNS3%	0.19± 0.03	76±4	51.5± 6.6	1304± 321
AeroCNS5%	0.19± 0.04	75±5	71.0± 5.5	4574± 175

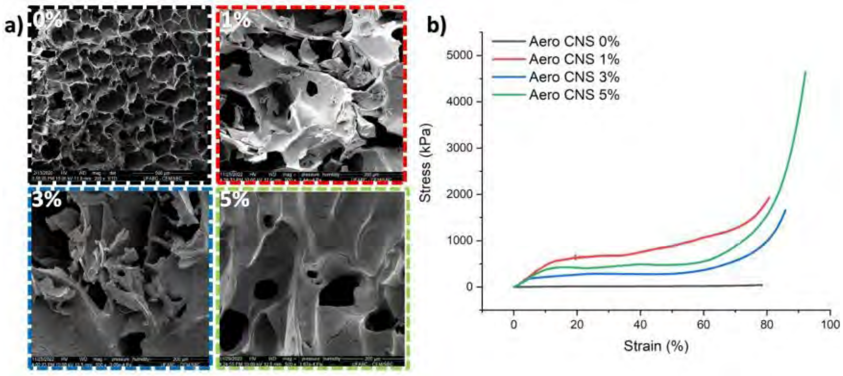

Fig.1 a) Photomicrographs, and **b)** stress-strain curves for all composite aerogels.

Their morphology resulted in interconnected 3-D structures composed of visible thin lamellae. Furthermore, the presence of pores is noted, varying their size, which is responsible for the low density previously discussed. Besides, particles of CNSs embedded in the structure of the starch aerogel are verified. A reduction of 53, 51, and 30 % in the compressive modulus of composite aerogels were observed compared to Aero NEC 0%. It is due to the increase in voids and pores in the material, which reduces the compressive modulus of composite aerogels (Zhao, 2020). However, an increase of 48, 39, and 127 times in compressive strength were verified compared to Aero CNS 0%. It is possible due to the reinforcing effect of the CNS and its good adhesion to the aerogel pore structure (Sorriaux, 2021).

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ID 153

MILKWEED FIBERS: BETTER EFFICIENCY FOR INSULATION UNDER COMPRESSION.

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Asclepias syriaca, or common milkweed (Milkweed), is a plant native to North America. At the end of the summer, when pollinated, its flowers transform into pods packed with hollow fibers. Septemberwinds,split them opentoreleasetheirseedsindividuallyattachedto afloss.This fiber raised our interest. Like cotton, milkweed fibers are single-cell fibers. Its hollow nature is responsible for good thermal insulation. Its smooth surface along with its low strength against externalloadscause difficultiesduringspinninghencethepreferencefor a nonwovenprocess. In additiontomilkweed'sfiberinsulation,factorequivalentto900fillpowergoosedown,itprovides amazing superiority when subjected to compression. Thanks to the hollow structure, air gets trapped between fibers, like conventional insulation, but also within the fiber itself.

Ekoterre inc., in partnership with the CTT Group has set up a manufacturing process for a 100% plant-based insulation using milkweed floss. This insulation can be manufactured to different weightsdependingontheapplication.Testshavedemonstratedaperformanceundercompression 3x higher than conventional synthetic insulation. This paper will be devoted to presenting milkweed fibers, their properties, insulation manufacturing and performance with and without compression.

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ID 155
STUDY OF THE PERFORMANCE OF ALTERNATIVE BIO-CONCRETE TO HEMP CONCRETE

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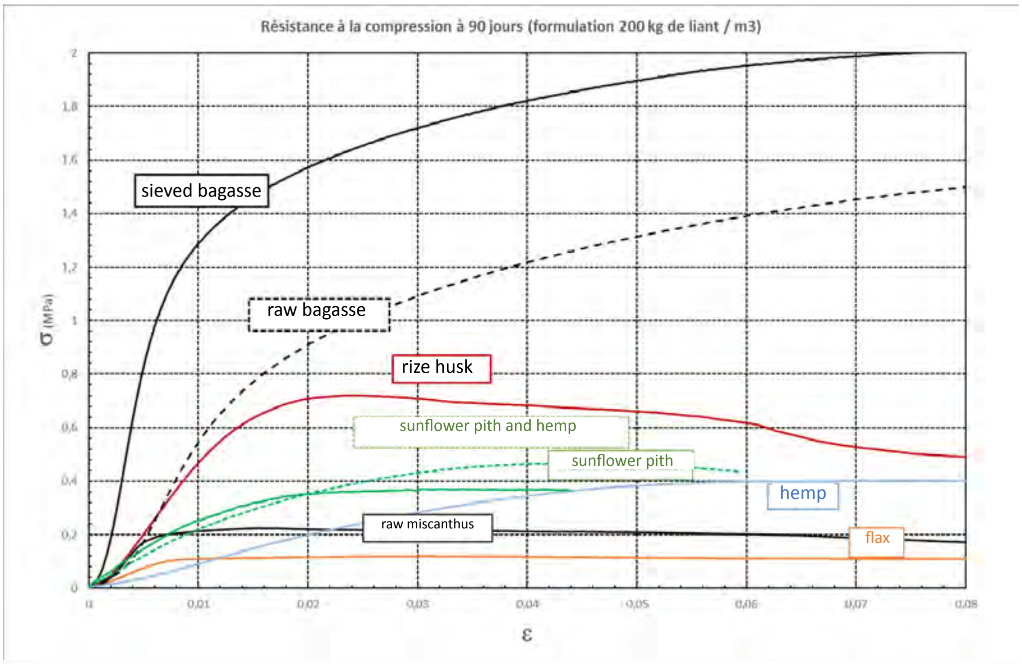
ABSTRACT
The hemp resource for concrete may be limited, due to the increasing needs of other applications such as plastics, so the use of other types of plants may become essential. This article presents a set of results obtained on the study of bio-concretes based on particles other than hemp, such as flax, rice husk, sunflower pith, miscanthus … The characteristics of the particles (absorption, leaching…) and the mechanical, thermal and hygrothermal performances of the concretes are presented, showing the potential of these concretes for a use similar to hemp concrete.

INTRODUCTION
Biosourced concretes are lightweight, self-supporting concretes that have thermal and acoustic qualities, while storing carbon and reducing the environmental impact of construction and the energy consumption of housing. Recently, during 2021, hemp concrete construction in France has been faced with a shortage of hemp raw material, due to an increased demand from the plastics industry, which also uses hemp as a filler, notably for the automotive industry. It is therefore essential to diversify the sources of supply in order to secure construction sites. However, the use of plants other than hemp poses regulatory problems, as hemp concrete is the only one in France to benefit from a regulation that allows it to be used in an insurable manner in the event of a disaster.
This question of using resources other than hemp in concrete is still relatively recent, but there have been a few studies on concrete made from rice husks, flax or miscanthus [Magniont, 2016] [Ratsimbazafy, 2022]. This article presents a set of results obtained on concretes based on different particles [Diaz, 2018][Mena, 2018][Recher, 2017][Nguyen, 2017], namely: flax shives, rice husks, bagasse (sugar cane), miscanthus, typha, sunflower pith. Each plant was characterized from the point of view of its use in concrete (density, water absorption capacity, solubility, granulometry) and the mechanical and thermal performances of the concretes were determined. The binders used in this study are those typically used in the manufacture of hemp concrete.

RESULTS AND CONCLUSIONS
The study highlights several points:
- the influence of grain size on mechanical performance,
- some possibilities of incompatibility with the setting of certain types of binders,
- very interesting mechanical performances, superior to hemp for several plant resources, for formulations with relatively low binder content,
- good thermal and hygrothermal properties, as well as very effective phase shifts for summer comfort.

Fig.1 Curves of Compression tests of bio-concretes – formulation 200kg/m³ – curing time 3 months



This study shows the potential of plant co-products for the production of high-performance concrete. This could help avoid supply disruptions or the need to import hemp over long distances, while at the same time relocating the deposits from diversified resources.

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ID 157
EXPERIMENTAL AND NUMERICAL ANALYSIS ON CARBON-FLAX / EPOXY LAMINATES: DELAMINATION AND IN-PLANE CRASHWORTHINESS

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ABSTRACT
This work presents an experimental and numerical study to characterize the delamination and in-plane crashworthiness behaviour of carbon, flax, and carbon-flax (hybrid)/epoxy composite laminates. DCB, 4ENF, and ILSS tests were carried out on carbon/epoxy and flax/epoxy laminates to determine the values of the interlaminar fracture modes required to model fracture behaviour. In-plane crashworthiness tests were then performed to investigate the delamination phenomenon and energy absorption capacity considering a carbon-flax (hybrid) configuration and two different specimens' geometries: flat and corrugated. These data are then used to build numerical models in LS-DYNA able to reproduce the specific experimental trends and to design composite structures.

INTRODUCTION
In recent years, the research of new green composite solutions in terms of sustainability and recyclability is the main challenge for researchers and industry. In this context, natural fibres such as flax and hemp are widely used due to their low-density, low-cost, high strength-to-weight ratio and interesting energy absorption properties. Their mechanical properties in terms of strength and stiffness are, of course, not comparable to those of synthetic fibres such as carbon. Therefore, the study of hybrid solutions containing both natural and synthetic fibres is carried out. In this work, the delamination phenomenon of carbon/epoxy and flax/epoxy laminates is studied from both experimental and numerical points of view. A complete experimental campaign including tensile, compression, four-point bending, and low-velocity impact (LVI) tests has already been performed on the same material configurations. Therefore, the DCB, 4ENF, ILSS, and in-plane crashworthiness tests are discussed here. The data obtained from these tests were then used as material parameters to build the corresponding finite element models in LS-DYNA and to reproduce the different physical phenomena.

RESULTS AND CONCLUSIONS
The results of the DCB and 4ENF tests are used to compute the mode I (G_{IC}) and the mode II (G_{IIC}) interlaminar fracture toughness of the toughened epoxy resin and the corresponding numerical models have been shown to reproduce the experimental trends very well. On the other hand, only the hybrid configuration is considered for the in-plane crashworthiness tests since it can achieve higher force values by combining

the potential of both fibre types. However, in this case, two different geometric configurations are analysed under dynamic conditions: flat (rectangular) and corrugated (S-shape). In the former case, the specimen is easier to manufacture but requires a complex anti-buckling fixture and a sawtooth-shaped release feature in the shorter side opposite to the impactor to avoid undesirable failure modes like tearing or buckling. In the latter case, instead, the corrugated shape does not require a specific fixture. Therefore, starting from the analysis of flat samples, an impact energy of 300 J for three orientations (0°, 90°, and isotropic) was considered. Then, considering the same orientations, but the corrugated geometry, a higher resistance was noticed and therefore higher energy levels (400 J for 0° and 500 J for 90° and isotropic cases) were considered. The experimental load-displacement results for both cases are shown in Fig. 1. From this comparison, it is found that the latter geometry is also the best in terms of specific energy absorption (SEA).

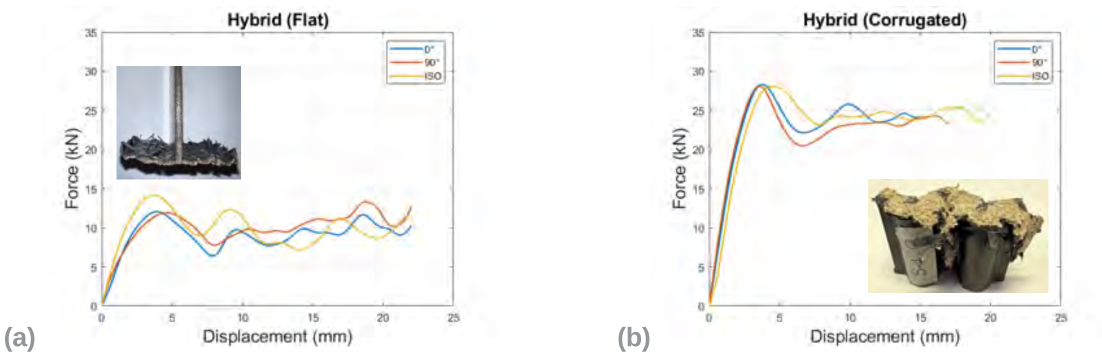

Fig.1 Experimental load-displacement plots for hybrid **(a)** flat and **(b)** corrugated samples under in-plane crashworthiness.

All these experimental results are then compared with the results of the corresponding numerical models. In particular, in order to correctly reproduce the splaying phenomenon, a model with two shell layers connected by a tie-break contact is used. After a suitable tuning of the material parameters, the results of the numerical simulations can reproduce the experimental trends and to capture the phenomena of fracture and delamination. One way to further improve the results is to apply an optimization strategy to identify non-physical parameters required by the material cards. A well-calibrated model could become a very powerful tool for predicting the behavior of more complex structures under crushing before they are manufactured.

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ID 158
OPTIMIZATION OF COTTON KNIT DYEING WITH YERBA MATE (ILEX PARAGUARIENSIS)

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ABSTRACT

The use of natural dyes obtained from natural sources is a good alternative to reduce the environmental impact of the textile industry. Furthermore, it could be interesting that these natural sources are industrial processes by-products. However, the industry requires reproducible processes that can be easily scaled-up. This work aims to compare the influence of the extraction conditions of yerba mate (*Ilex paraguariensis*) on the properties of its extracts. In order to scale-up the process, data from yerba mate extraction and dyeing stages have to be collected, which can be achieved monitoring the lab scale process by UV-Vis spectroscopy. In addition, the influence of dyeing time on the color of the dyed cotton fabric was evaluated.

INTRODUCTION

The yerba mate (*Ilex paraguariensis*) has been commonly used to prepare tea-like beverages in Argentina, Paraguay, Uruguay, and southern Brazil. Yerba mate extracts are composed mainly of polyphenolic compounds (chlorogenic acid) and xanthines (caffeine and theo-bromine) (Gullón, 2018). Due to its properties, the leaves and branches of yerba mate are also used industrially in the production of energy drinks, beers, soft drinks, and liqueurs. In addition, they also have interesting applications in other industries, like the production of functional foods, or natural antioxidant additives, or in the cosmetic or pharmaceutical industries (Croge, 2020). During the industrial production process of yerba mate-based products, large quantities of by-products are generated (Gullón, 2018), and though they create an opportunity to investigate their valorization routes. Other authors investigated the dyeing properties of a mate plant in silk, wool, linen, and cotton fabrics (Yoo H-J, 2012).

In this study, the traditional extraction conditions (hot water) as well as an alternative alkali extraction methodology were studied. The impact of extraction conditions on extract properties was assessed the viability of monitoring the dyeing process by UV-Vis spectroscopy was proved. In addition, the influence of the dyeing time on the color of the cotton knit dyed was evaluated.

RESULTS AND CONCLUSIONS

The impact of the extraction conditions on the properties of the extract was evaluated (Table 1). As observed, the alkali extraction allowed to achieve higher extraction yield but the extract exhibit lower phenol content and antioxidant activity than the one obtained only with water.

Table 1 – Extraction Yield and characterization of yerba mate extract

Sample	Extraction conditions	Extraction Yield EY (%)	Total phenols content (g GAE/100 g extract)	Antioxidant activity (nmol AAE/mg EM dry extract)
EM0	--	28.4 ± 0.5	48.1 ± 2.7	915.0 ± 69.0
EM1	NaOH	34.2 ± 0.6	41.1 ± 2.1	667.0 ± 36.7

AAE: ascorbic acid equivalent; EM: Yerba mate extract. S/L=1/10, temperature 90 ºC, extraction time 60 min.

Subsequently, the yerba mate alkali extraction and the dyeing process with the extract were monitored by a UV-VIS spectrometer equipped with a transmission probe (Fig.1)

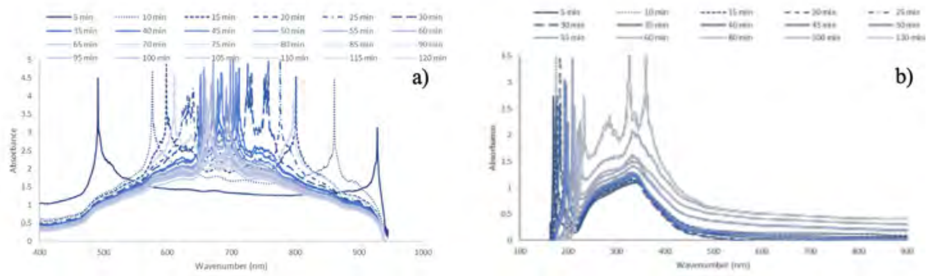

Fig.1 Monitoring by UV-Vis of a) the alkali extraction process of yerba mate and b) the dyeing process with the yerba mate extract (EM1)

The impact of dyeing time on the color of the cotton knit dyed was evaluated (Fig.2).

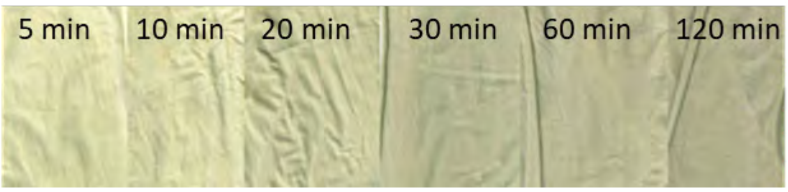

Fig.2 Cotton textile samples dyed with Yerba Mate extract (EM1): effect of dyeing time

In the case of yerba mate, the control of both extraction and dyeing processes, based on UV-Vis spectroscopy monitoring, has proven to be an important tool to achieve the requirements of the textile industry.

ACKNOWLEDGMENTS

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ID 159
INVESTIGATIONS ON ULTRASTRUCTURAL PROPERTIES OF DIFFERENT TYPES OF SPIDER SILK BY USING NANOBEAM X-RAY DIFFRACTION

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ABSTRACT
This work shows investigations on the ultrastructure of spider silk. By using ESRF nano-X- ray beam to perform wide angle x-ray scattering (WAXS) and small angle X-ray scattering (SAXS), we could obtain information on the inner structure of one of the most fascinating natural materials. Besides gaining a more detailed understanding about underlying structure- function relationships, we want to understand, why some spider silks react differently toothers when used as a support forcells to treat peripheral nerve injuries. Apart from lookingat single strands of spider silk we investigated µm thin transverse and longitudinal sections derived by cryo-microtomy. Not only one type of spider silk but a variety of different species was looked at. Finally, the same type of silk but pre-treated in different ways was tested.

INTRODUCTION
Spider silk is a very fascinating material, which has been engineered by nature since almost 400 million years (Shear, 1989) and shows exceptional mechanical properties. In addition to its impressive mechanical performance, a variety of possible medical applications have emerged due to its excellent biocompatibility. Specifically, spider silk is a promising material for nerve regeneration and has successfully worked as center piece of nerve guidanceconduits. The silk supports the adhesion and movement of Schwann cells which are responsible for the regeneration of peripheral nerves after injury (Kornfeld, 2021, Semmler, 2023). In cell culture experiments Schwann cells were found to prefer certain types of spider silks to others. It is still largely unknown why this is the case. In this work we investigate the ultrastructure of silk, which could be a possible parameter influencing the acceptance by Schwann cells.
Scanning nanobeam X-ray diffraction (nanoXRD) (Riekel, 2019) was used as a method of choice for ultrastructural characterization. Because of the small dimensions of the samples a sub-micron synchrotron X-ray beam was employed to get information about the silk ultrastructurein aspatially resolved manner. Nano-diffraction experiments werecarried outat the European Synchrotron Radiation Facility (ESRF) ID13 beamline in Grenoble.

RESULTS AND CONCLUSIONS

Findings show differences in crystallinity and crystal domain size for differently pretreated (sterilized) silk from the spider Nephila edulis. Pretreatments included immersion in ethanol, autoclavation and UV irradiation (Figure 1). Another interesting result is the ultrastructure of the tubuliform silk, which can be found in the egg sac of spiders. This kind of silk was found to show an additional not yet reported Bragg peak in the WAXS pattern compared to dragline silk (Figure 2). Data obtained from scanning the transverse and longitudinal sections is currently analyzed and results can be expected soon. In conclusion nanobeam X-ray diffraction is a very suitable method to get detailed structural information on different spider silk morphologies (single fibers as well as transverse and longitudinal sections).

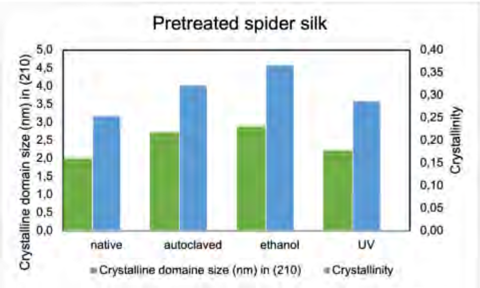

Figure 1. Crystalline domain size in (210) direction and Crystallinity parameter for differently pretreated (sterilized spider silks from Nephila edulis derived by nanobeam XRD.

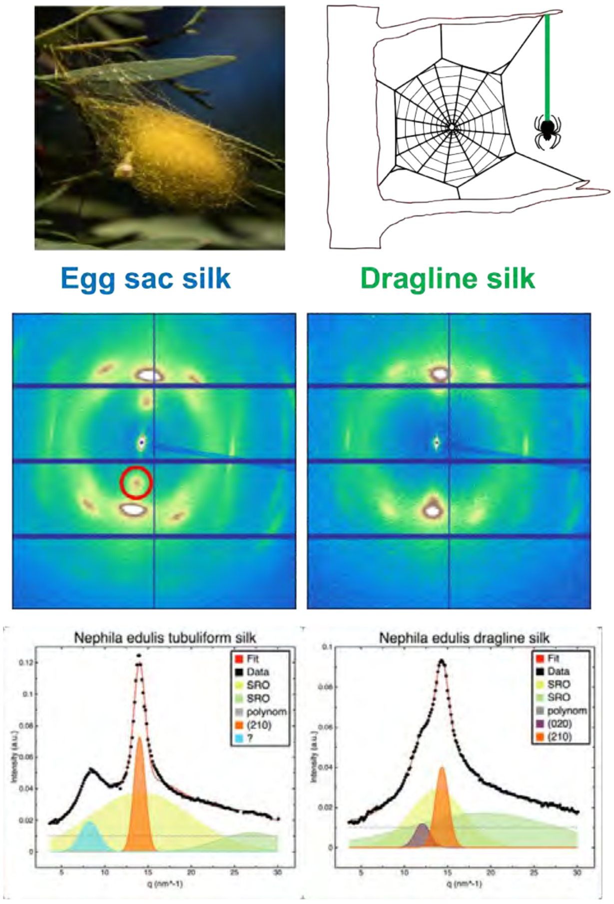

Figure 2. On the left: Illustration, WAXS pattern and 1D integration for of Nephila edulis egg sac silk (tubuliform silk). Ontheright:Illustration, WAXSpatternand 1D integration for of Nephila edulis dragline silk. The red circle on the left WAXS pattern indicates the additional not yet reported Bragg peak for tubuliform spider silk.

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ID 161
A STUDY OF MECHANICAL AND IR PROPERTIES OF WOOL REINFORCED COMPOSITES

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ABSTRACT
Pakistan is blessed with an abundance of natural resources. The wool industry has depleted in Pakistan due to different economic factors like the import of fine-grade wool and semi-processed fibres from other countries. This work package covers the revival of coarse-grade wool in Pakistan, which mostly remains unattended. The research aims to develop optimized processing of local wool for the fabrication of sheets utilizing the latest techniques according to the required dimensions. These sheets would be proceeded for the development of composites to acquire the shape of different valuable products having features like thermal insulation, moisture retention, sound absorption, filtration & fire retardancy. Different composite samples with different amounts of wool fibre, resin, and filler were prepared. Wool fibres of volume fractions 0.05%, 0.10%, 0.15%, 0.20%, 0.25%, and 0.30% and fillers of Aluminium powder, Oxides of metals like Aluminium oxide, Zinc oxides, and Iron oxide with volume fraction 0.06%, have been exercised. Each sample was tested for IR absorption, IR Transmission, and Tensile strength. With the increase of wool volume fraction, IR absorption increases and reduces IR transmission. Fillers increase the IR absorption property of composites. Zinc oxide, Aluminium Oxide, Titanium oxide, and Aluminium particles introduced good IR absorption properties and good tensile strength. The samples tested showed a significant impact on performance parameters.

INTRODUCTION
In history, wool is considered as a precious natural fiber, used for a long time as a textile raw material (J. Gordon, 1984). Sheep wool is curled and having scales which is heavier and keep the body warm from wind. It helps to protect the body from cold and heat from the outside. Wool has Natural antibacterial, antimicrobial, UV-blocking, and insulating properties (B. Rohrig, 2022). Pakistan's economy majorly depends on the production of livestock and crop farming (J. G. Nagy, 1998). Globally, 995 sheep breeds have been reported, where Asia is sheltering 265 (27%) sheep breeds. According to the 2014-2015 Pakistan economic survey, The Sheep population in Pakistan was 29.4 million which yields 44.1 million wool fibers. More than 14.1 million which is 48% of the total sheep population found in Balochistan (Abdul Sattar Safi, 2017). Balochi wool is used as raw material in carpet making but mostly wool from these sheep goes to waste because it is coarse and short. The main objective of this study is the conversion of this Balochi wool into value-added products like functional composites.

RESULTS AND CONCLUSIONS
All the data related to the tensile strength values have been shown in the following Figure 1. It is clear from the figure that as we increase the content of wool fibre in the samples (1 to 6), the value of tensile strength increases considerably and after a considerable increment it tends to go downwards. It also means that the

optimized values of tensile strength may be achieved till the wool fibre content of 0.25 %.

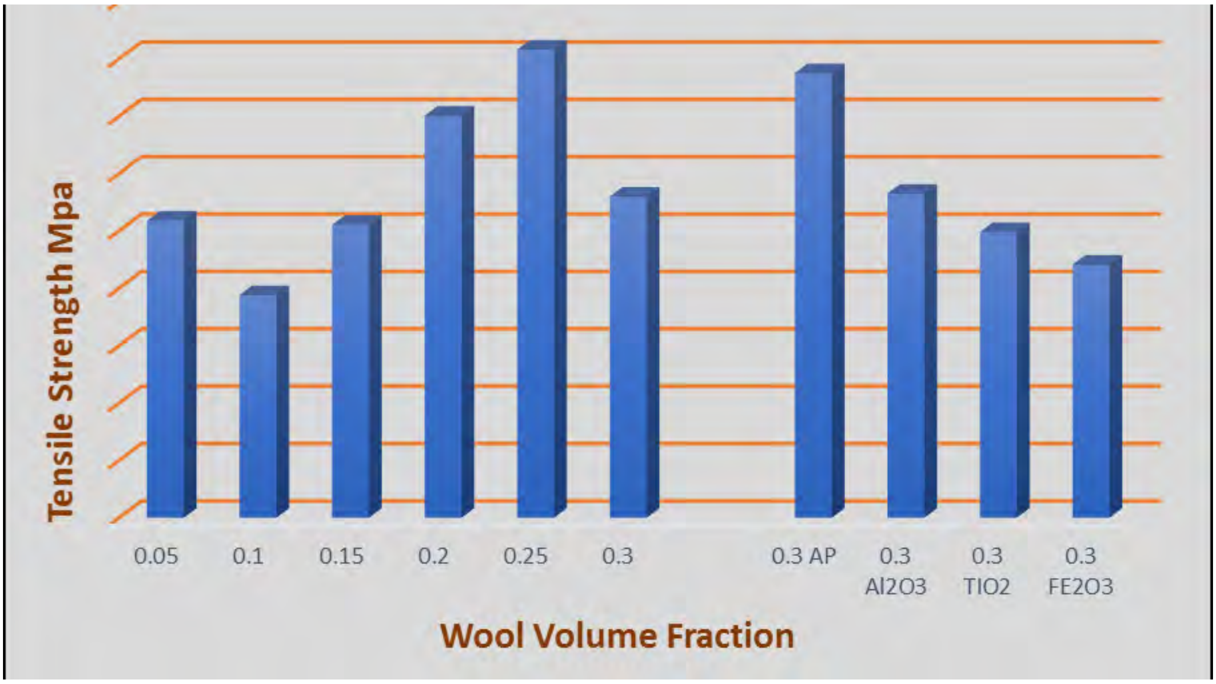

Fig.1 Tensile test summary

In the case of using fillers, we see a different behavior with regards to the intensity but there exists quite a significant impact of different fillers on the tensile strength.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the funding by HEC (Pakistan), under Great Challenge Fund (GCF).

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ID 166
DEEP CHARACTERISATION OF THE POROSITIES INDUCED BY DEFECTS IN FLAX FIBRES BY EXPLOITING X-RAY TOMOGRAPHY

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ABSTRACT
Flax fibres are valuable reinforcements for tomorrow's composites. However, defects such as kink-bands might affect their mechanical properties due to the porosities they contain. In this work, phase-contrast X-ray microtomography (Anatomix beamline, Synchrotron SOLEIL) was used to display a sharp 3D representation of these porosities. The study highlights the relationship between kink-bands and secondary cell wall ultrastructure, by analysing the organisation (shape, orientation) and morphology (thickness, volumes) of the pores. Stress concentration in kink-band was investigated using finite element (FE) modelling.

INTRODUCTION
Flax fibres present ecological advantages while ensuring good mechanical properties. However, these fibres are affected by defects in their internal structure called kink-bands or dislocations, which can lead to the appearance of weakness areas and cracking initiation and debonding in composites. The origin of defects is not yet certain, but the literature agrees that the extraction of fibres has an impact on their occurrence (Bourmaud et al., 2022).

Elementary flax fibre is composed of several hierarchically arranged layers, which have different compositions and thicknesses (Baley et al., 2018). The presence of kink-bands affects the structural organisation in all these layers, but especially the S2-G layer, located in the secondary cell wall, which is the thickest and is mainly responsible for the longitudinal mechanical properties of the fibres. Cellulose microfibrils contained in this layer are oriented at a microfibrillar angle (MFA) which is closely correlated to the mechanical properties of the fibres. Previous work has shown that the presence of a kink-band affects the MFA (misorientation from 5-8° up to 30-40°) and promotes the local appearance of porosities (Melelli et al., 2021). The following results aim to investigate these porosities.

RESULTS AND CONCLUSIONS
X-ray microtomography complements other imaging techniques by showing a detailed 3D representation

of the voids in the defect regions (Fig. 1a). It appears that pores are organised concentrically around the lumen cavity (Fig. 1b) and are slightly misaligned with lumen axis (Fig 1c), which reminds the typical disorientation of cellulose microfibrils in defected areas. The thickness of S2-G sub-layers between pores was estimated to be about 0.4 µm, which is very thin. These findings corroborate the idea that voids appear at cellulose layer interfaces in the gelatinous bulk in the secondary cell wall.
Finally, the impact of kink-bands on stress concentration was evaluated. 1% strain was applied on a flax fibre in order to assess stress distribution using FE modelling. It appears that defected areas show an increase of stress concentration at the pore surfaces (Fig. 1d).

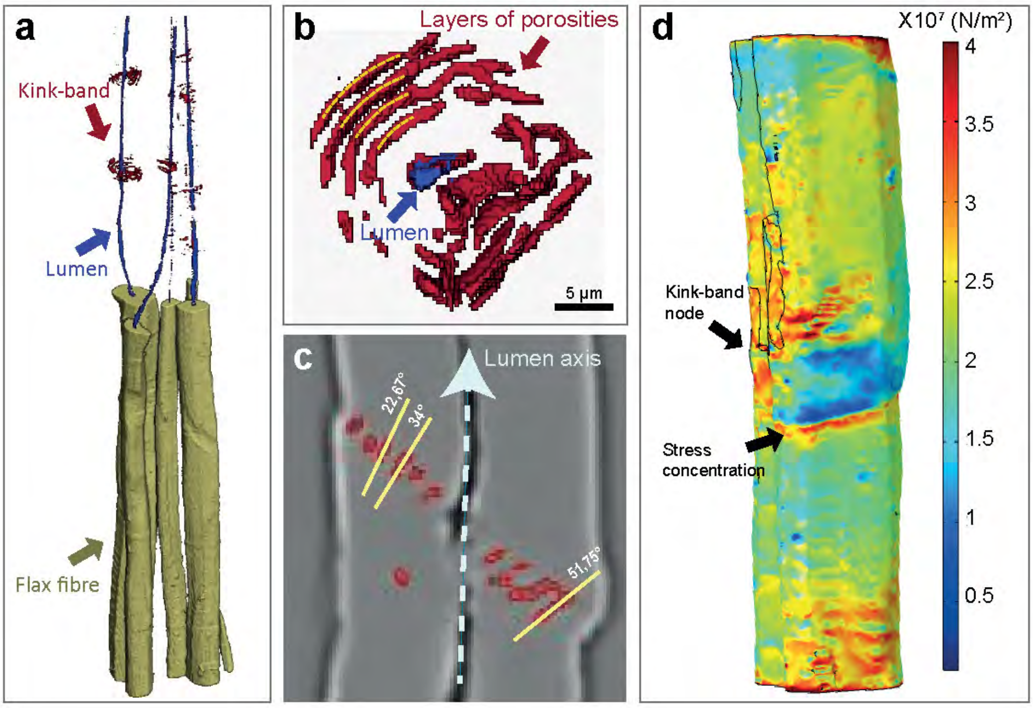

Fig. 1 (a) Flax fibres reconstruction on the lower part, porosities segmentation on the upper part, **(b)** kink-band pores view from above, concentric organisation highlighted in yellow, **(c)** kink-band pores misorientation, **(d)** stress distribution in flax fibre under 1% strain

ACKNOWLEDGMENTS

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ID 168
MACHINE LEARNING FOR NATURAL FIBRE-REINFORCED COMPRESSED EARTH BLOCKS

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ABSTRACT
For centuries, natural fibres have found wide application in traditional natural building materials, such as earth. However, the great variety of fibres in nature and the extreme variability between them make the design of modern blends difficult. The aim of this study is tocontinuetoinvestigatethepossibilityofusingarti-ficialneuralnetworkstodeveloppredictive modelsthatsupportthedesignofcompressedearthblocksreinforced-withrandomlydistributed naturalfibres.Realandsyntheticdatafromliteratureminingformthedatabaseused-totrainthe network. The overall results show a very high prediction accuracy. For example, a correlation coefficient (R-value) of 0.97 was found for the prediction of the compressive strength value.

INTRODUCTION
Natural materials are gaining increasing interest due to the sustainability issues in the construction sector. Exploring new material design spaces is indispensable to promote innovation in this research field, but the great variability of natural elements makes the task hard to execute. Compressed earth blocks are a modern application of earth as a construction material. It is well known that earth suffers from certain problems such as durability, dimensional instability, brittleness, low mechanical strength, etc. (Turco et al., 2021b). For thesereasons,inadditiontovariouschemicalstabilisers(lime,cement),theadditionofnatural or synthetic fibres in the mixture design is being increasingly experimented. However, the literature underlines the effort that the experimental determination of the physical-mechanical characteristics of this construction material requires (Fabbri et al., 2022). In order to obtain reliable and statistically relevant knowledge of the information sought for the development of each product, extensive experimental campaigns employing large amounts of resources (materials, energy, instruments) are required. Artificial intelligence is a valuable tool in supportingmaterialscienceproblems.Themainadvantage initsapplicationisthatknowledge ofthep-hysicsgoverningthebehaviourofthematerialtobedeveloped,improvedoroptimised isnotrequired formachine training.Infact, machine learning models are trainedon databases andtherelationshipbetweentheinputvari-ablesandtheoutputissought.Basedontheprevious work (Turco et al., 2021a), the enhanced Artificial Neural Networks (ANN) model for the prediction of natural fibre-reinforced compressed earth blocks' mechanical properties is proposed in this paper.

RESULTS AND DISCUSSION
Data from literature mining and synthetic data are used to train the model. Features include mix design proportion (soil, binder, natural fibers and water content), curing time, fibers' properties (aspect ratio, tensile strength). Due to the lack of comprehensive data available in the literature, predictions are limited to blocks' mechanical characteristics. The available database was randomly divided as follows: 70% is used for training the network,

15% is used for validating the model and the remaining 15% is finally used for testing the model.
The graph in Figure 1 (left side) shows the result of the regression model obtained from the neural networks. The dotted line represents the ideal scenario in which the predicted compressive strength value coincides with the objective, or actual, value. The overall results show very high prediction accuracy. For example, a correlation coefficient (R-value) of 0.97 was found for the prediction of the compressive strength.randomly divided as follows:70% is used for training thenetwork, 15% is used for validating the model and the remaining 15% is finally used for testing the model.
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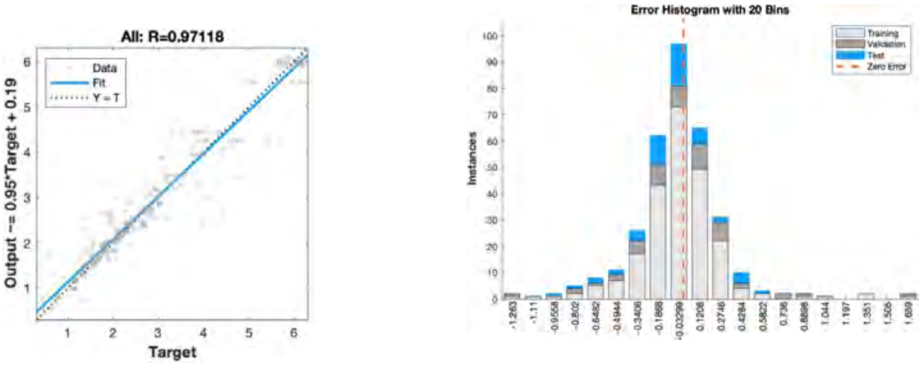

Fig.1 Accuracy of the regression model (left) and error distribution (right).

The developed computational tool allows to:
• Switch from systematic or random (trial-and-error) exploration approach to a more performing targeted mixture design approach.
• Perform several computer simulations and improve the knowledge of the problem, transforming some variables in design priority.
This study represents one of the first attempts to apply machine learning in the field of an all-natural building material (earth + natural fibers) for modern applications. The authors are aware of the small size of the database compared to other fields of application; however, it also represents an attempt to raise awareness of sharing experimental data and information relevant to the development of a robust model.

ACKNOWLEDGMENTS

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ID 169
APPLICABILITY OF COIR FIBRE-REINFORCED COMPOSITES IN BUILDING AND CONSTRUCTION ENGINEERING: A REVIEW

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ABSTRACT
Coir fiber is a natural and biodegradable material, making it an environmentally friendly alternative to synthetic fibres. Due to its high water-holding capacity, coir fibres have been used in different studies to design composites. The present article discusses a review of recent developments regarding coir-based composites. The coir fibre reinforced composites (CRFP) find their advantages and disadvantages for various reasons. The article mentions recent developments in the field of CRFP. The applicability of these composites is common in civil and construction industries for reinforcing concrete and masonry structures. CFRP composites have several advantages over traditional materials, such as high strength, stiffness, and durability, as well as low weight and corrosion resistance.

INTRODUCTION
Coir fiber reinforced polymer (CFRP) composites are materials made by combining coir fiber with a polymer matrix. The polymer matrix can be made from various materials, including epoxy, polyester, and polypropylene. The coir fiber provides strength and stiffness to the composite, while the polymer matrix acts as a binding agent and protects the fiber from damage. CFRP composites are used in various applications due to their high strength-to-weight ratio and durability. Additionally, coir fibre is an environmentally friendly and renewable resource, making CFRP composites a sustainable alternative to traditional materials (Haydaruzzaman et al. 2010; Wang and Hu 2016; Singh et al. 2021).
The use of coir fibre-reinforced polymer (CFRP) composites in civil engineering has been the subject of many studies, and the results suggest that they have great potential in this field. Studies have shown that CFRP composites have high strength, stiffness, and durability, making them well-suited for the reinforcement of concrete and masonry structures, such as bridges, buildings, and retaining walls. They are particularly effective in retrofitting existing structures, improving their strength and stability and making them more resistant to seismic events (Rafiquzzaman et al. 2017).
One of these studies' major results is that using CFRP composites can significantly improve structures' load-carrying capacity (Wang and Hu 2016). For example, a study by Lim, Lindberg, and Bates showed that using CFRP composites increased the load-carrying capacity of reinforced concrete beams by up to 50%. Another study claims that using CFRP composites improved the seismic performance of reinforced concrete beams, reducing damage and cracking (Tran et al. 2019). Another significant result from these studies is that CFRP composites are a sustainable and cost-effective alternative to traditional materials.

They are made from renewable resources, such as coconut fibres, and have a lower carbon footprint than conventional materials like steel (Saxena and Chawla 2020). In addition, they are lightweight and easy to install, making them a convenient and efficient choice for structural reinforcement and retrofitting.
Overall, the results of these studies suggest that CFRP composites have great potential in civil engineering. They have high strength and durability, are effective in retrofitting existing structures, and are a sustainable and cost-effective alternative to traditional materials. As a result, the use of CFRP composites is expected to increase in the coming years, contributing to developing more sustainable and resilient infrastructure.

RESULTS AND CONCLUSIONS

The studies above provide evidence of the potential of coir fibre-reinforced polymer (CFRP) composites in civil engineering. The results suggest that CFRP composites have high strength, stiffness, and durability and can effectively reinforce concrete and masonry structures. CFRP composites effectively retrofit existing structures, improving their strength and stability and making them more resistant to seismic events. The studies also indicate that CFRP composites are a sustainable and cost-effective alternative to traditional materials and can significantly reduce the construction industry's carbon footprint. Overall, the results of these studies support the increased use of CFRP composites in civil engineering.

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ID 171
FUNCTIONAL SILK FIBERS BY THE MOLECULAR IMPRINTING OF SILK NANOPARTICLES AND A HIERARCHICAL APPROACH

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ABSTRACT
With the present work we propose a versatile and hierarchical strategy to the preparation of functional fibers, based on the molecular imprinting technology applied for the first time to natural polymers. As a proof of concept, silk fibroin molecular nanotraps, selective for a serum protein, were synthesized and used to decorate silk nano- and micro-fibers, outlining a general route to form biocompatible functional fibrous materials.

INTRODUCTION
The molecular imprinting of polymers (MIPs) is an established strategy to prepare molecular recognition materials (Arshady and Mosbach 1981). It consists of a template assistedsynthesis that enables to stamp binding sites in the growing polymeric material (Figure 1), in fact,thetargetedmoleculeactsasatemplateandis placedinsolutiontogetherwithfunctional cross-linkable monomers; the weak-bond interactions between the monomers and thetemplate are next stabilized by polymerization. As a result, molecularimprints of the template are left on the formed MIP, which possesses high affinity, specificity and selectivity for the target. MIPs are robust to harsh conditions and cheap to synthesize. Additionally, MIPs canbe prepared of nanometer dimensions (nanoMIPs) (Pilestky et al.2020), rivalling with natural monoclonal antibodies (Bossi AM 2020) and holding promises in the analytical domain andin medicine (Dong et al. 2019).
Typically to date, MIPs have been prepared starting from acrylamides, acrylates, or methacrylates monomers, while the use of natural materials as building blocks has gone largely unexplored. Here we demonstrated the feasibility of imprinting silk fibroin, and we named these natural MIPs as bioMIPs. Further, these highly selective bioMIPs were used to decorate silk fibers, tracing a general route to the formation of a class of hierarchical functional materials.

RESULTS AND CONCLUSIONS
Molecularly imprinted silk fibroin nanoparticles, called bioMIPs, were synthesized starting from methacrylated silk fibroin (SilMA) building blocks, placed in solution with the chosen template, that was the protein human serum albumin (Figure 1). Photopolymerization was used to stabilize the supramolecular silk fibroin nano-assemblies, thus forming the bioMIPs. The optimization of the synthetic conditions was performed by applying a response surface method, in which the input variables were: the concentration of silk fibroin, the pH, the photoinitiator concentration, whereas the output variables were the hydrodynamic size of the bioMIPs and the polydispersity index (PDI). As a result, bioMIPs with hydrodynamic size (Z_{ave}) of 95.6±1.2

nm and PDIof 0.358 were obtained, hinting to a homogeneous population of functional silk nanoparticles. Selective recognition towards serum albumin was demonstrated by spectrophotometric and nanocalorimetric experiments. The dissociation constant of the bioMIPs for albumin was in the nanomolar range, accounting for high affinity imprinted cavities and selectivity was demonstrated (Bossi et al 2021). Additionally, the bioMIPs showed non-toxicity and cells compatibility (Maniglio et al. 2023). Hence, the bioMIPs met the expected role of functional nanotraps for albumin.

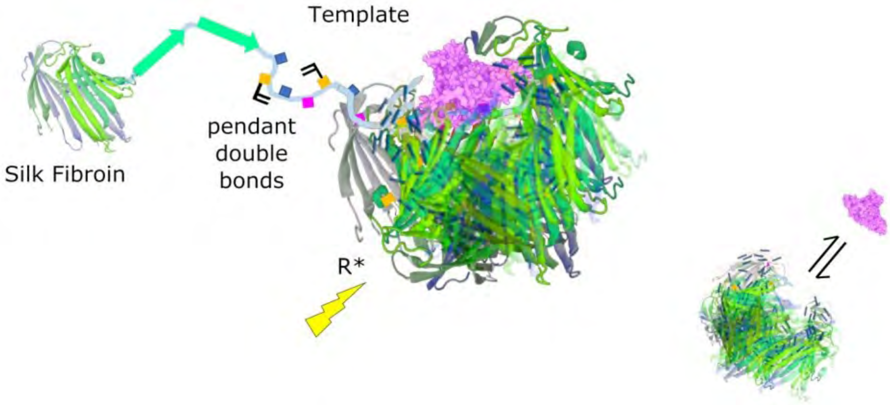

Fig.1 The principle of formation of molecular imprinting silk nanoparticles (Adapted from Maniglio et al. 2023). Silk fibroin structure is depicted in green, the human serum albumin 3D structure is purple.

The bioMIPs were further integrated to natural silk micro- and to electrospun silk nano-fibers, by exploiting bioconjugation. As a result, the bioMIP-decorated fibers acquired selective recognition for the protein human serum albumin, demonstrating the successful entailing of recognition to the fibers.

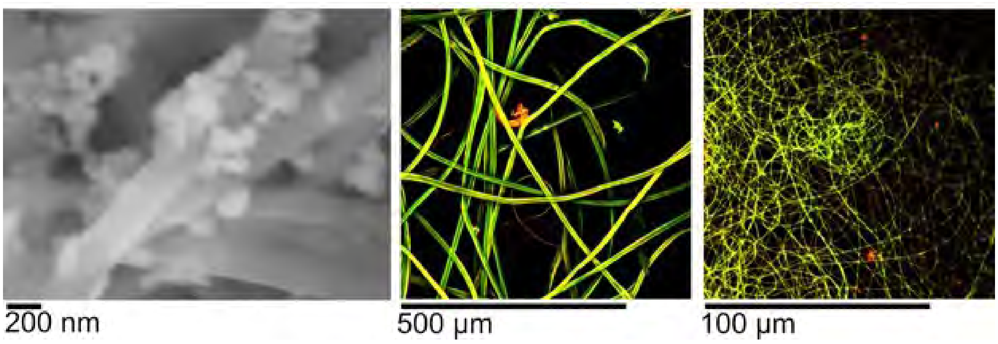

Fig. 2 Functional silks prepared by decorating micro- or nanofibers with silk molecularly imprinted nanoparticles (i.e. bioMIPs). Left panel: SEM image showing the bioMIPs immobilizedontothesilknanofibers. Centralandrightpanels:confocalmicroscopyimages proving the recognition of the bioMIPs/silk functional fibers. Green: emission of human serum albumin labelled with fluorescein; Red: bioMIPs labelled with rhodamine; Yellow: colocalization of signals of human serum albumin and bioMIPs, supporting the albumin binding to bioMIPs. (Adapted from Bossi et al. 2021).

This study demonstrated the feasibility of forming functional materials adopting a hierarchical strategy, that proceeds through the formation of discrete silk nanotraps, able to selectively bind a designed target molecule and obtained by the molecular imprinting of silk fibroin, followed by the coupling of the specialized bioMIPs to natural fibers. The approach can be generalized to any target molecule, allowing the design of green functional (bio)materials.

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ID 172
HOW THE MEASUREMENT TECHNIQUE DRAMATICALLY IMPACTS THE MECHANICAL BEHAVIOR EVALUATION FOR NATURAL FIBERS

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ABSTRACT
In this research work, we compare two measurement techniques for evaluating the mechanical properties of natural elementary fibres in tension. Due to the non-circularity of the fibres sections generally, the classical technique introduces bias in the fibre cross section measurement. This study offers to evaluate the systematic error introduced in the fibre cross section area evaluation and in the strength and elastic modulus using an alternative experimental approach. Using a simple finite element approach, the fibre strength potential is evaluated and compared to experimental measurements conventionally evaluated.

INTRODUCTION
Tensile tests are classically performed to measure the mechanical performances of natural elementary fibres in tension. Once the test is performed, a tension displacement curve can be obtained. Material behaviour is often obtained on a stress-strain curve. The strain in finite deformations is straightforwardly obtained knowing the length of the tested fibre and its elongation. Considering synthetic fibres such as glass or carbon fibres, depending on the process repeatability, fibre possesses globally well-circular cross sections. Evaluating their surface is then easy and is done without a major error on a scanner or a microscope. The calculated stress is representative of what the fibre effectively handles.
Natural fibres, such as flax, hemp, nettles, on the other hand, possess noncircular sections. Determining the surface cross-section of the fibre in this case necessitate hypothesis. Until recently, the common practice was to consider a mean diameter as specified in the Standard test method (ASTM D3822 / D3822M—14). Technically, the fibre is mounted on a paper frame and observed under a microscope (five measurements are recommended to average the variation of the apparent diameter along the fiber). The term diameter makes sense here since a circular approximation of the cross-section is assumed.
Non-circularity of natural fibres is commonly observed specifically for flax and hemp bast fibres quantified by the circularity index which, for this type of fibre is typically between 0.3 and 0.6, indicating a pronounced ellipticity of the sections. Similar observations were made previously by (Garat, 2018) on technical fibres (fiber bundles).
Elliptic approximation should be preferred to approximate with fewer errors the cross-sections of elementary fibers. This is made possible using a FDAS scanning system (from Dia-Stron Ltd.) which measures the projected diameters of a fibre during a complete revolution along its axis; this operation can be repeated along the fibre length to quantify the evolution of the cross-section along the fibre. This evolution can be observed in terms of maximum and minimum projected diameters and in terms of section.

RESULTS AND CONCLUSIONS

One could argue that statistically, measuring 5 projected diameters along the fibre length as recommend by the standard procedure allows capturing a mean diameter for the circular model. It turns out that 80% of the time, the projected diameter measured with the microscope technique is higher than the projected diameter measured after a rotation of 90°. A bias is clearly introduced while mounting the fibre in the paper frame which leads to an overestimated fibre cross-section and then to underestimate tensile properties of the fibre (modulus and strength).

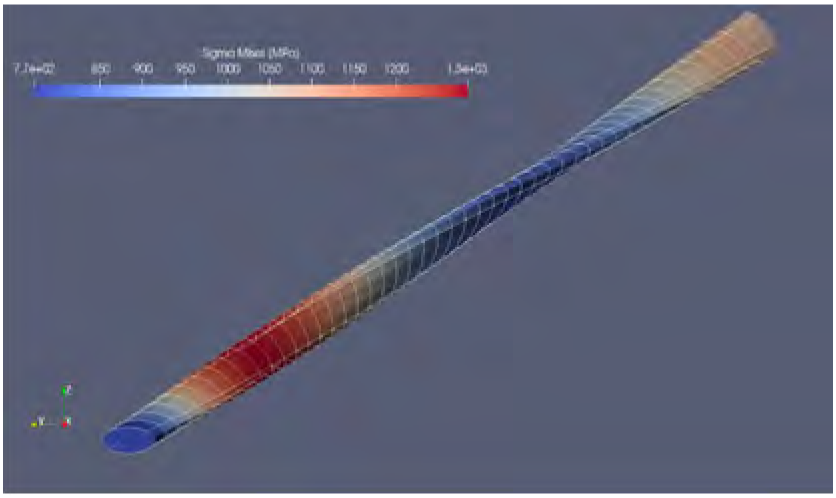

Fig.1 Strain distribution at failure in a hemp elementary fiber.

Additionally, as the section varies along the fibre, for a proper evaluation of the fiber stiffness, this evolution should be considered. This can be done analytically, if one knows the evolution of the radius along the fibre for a circular approximation (Qiu, 2000). In our case, we used a numerical simulation based on an elliptic approximation of the section. Based on the dimensions measured on the FDAS system using 70 sections along the fibre, the evolution of the maximum and minimum diameter together with the surface areas were determined. An isogeometric (Hughes 2005) mesh was constructed using quadratic functions in the cross section according to the real geometrical description. From the experimental load displacement curve, the elastic modulus is identified and the local stress at failure allows reading the ultimate stress the fibre handled at failure Fig. 1.

The results obtained using the elliptic approximation will be presented and compared to the ones obtained using the conventional standard test method. Differences observed between different bast fibres (textile flax, linseed flax, hemp and nettle) will also be presented.

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ID 173
INVINOTEX - FROM WINE HARVEST WASTE TO A TEXTILE-BASED ALTERNATIVE TO LEATHER

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ABSTRACT
Across society there is a lively moto inciting the creation of products embedded in zero waste principles (Hussain, 2022), building-up a sustainable future towards the establishment of areal balance in the way ofliving and exploring the available planet resources (United Nations, 2015). On the basis of this theory stands the circular and bio economy concepts, which envisage an enlightenment of any nature of waste as a viable raw material (European Environment Agency, 2017 and Schoenmakere, 2018). Hence, Tintex explored all these visions in the form of a composite textile-based material that presents a sophisticated unification of eco-borne materials & substances, present both in the textile substrate and inthe dual-layer coating assembly, which embodies c.a. 12% of grape marc powder. As a result, the material obtained is intuitively perceived as a fashionable proven concealment of high- sustainable guidelines, namely, the incorporation of Biosubstances, being naturally dyed (colour delivered by the combination of both the addition of grape marc fragments and the intrinsic colour of the bio-based polymers), having a cross Eco-Responsible processing (Low Temperature-Pressure treatments, Clean Gas Emissions). All these bioeconomic traces highlight the birth of a new leather concept, boosting the design, sustainability, circular economy and industrial symbiosis - granting long lasting nature-based aesthetics to the surrounding environment. Following the impetus revealed by this materialization, InVinoTex trademark arises as a new generation of products, merging two different Portuguesetrademark economic sectors, wine and textile, which aims to revolutionize the traditional leather-goods market trends.

INTRODUCTION
At Tintex, the eco-responsible sense/ vision exists behind each material/ product conceptualization, transformation, and conversion processes. Tintex value proposition is based on differentiation and specialization combined with the minimum environmental impact processing, materialized through advanced performance, and look enhancements on innovative knits.

Nowadays, Tintex is paving the way to create an opportunity to yield the uniqueness and sophistication aspects in new products, by intertwining knowledge and traditions from different industries. As a result, the proposed innovation addresses the incorporation of grape marc powder from Wine harvesting, usually treated as a waste after the grape pressing operation, in our biobased coatings.

Sustained by our deep knowledge in coating technology, it was possible to envisage how to build-up a variety of composite multilayered systems, which perfectly unifies all the elements - the bio/ green water borne polymers, waste and textile woven structures (knit). Taking advantage of the knit as the backing and support material for the coating layer deposition and utilizing Knife over roll methodologies a solid polymeric layer is obtained (Sen, 2008). Firstly the process starts, with a liquid, blending the polymer dispersions with the grape marc powder particles, along with the adequate rheology levelling and controlling additives, that is then transformed into the solid polymeric layer, under controlled temperature and pressure processing, by drying the water (conducted around 100 °C) and promoting both, polymer coalescence and curing (at 150 °C). This layer can be optimized/adapted by craving the surface profile by applying paper embossing technologies, under the conditions of 195 °C and 4 bar of pressure. This sequential arrange of transformations comprises a harmonious unification of all eco-borne materials & substances, shaping what it is meant to be a multi-layered composite material.

This ideation, leveraged by the development of an interindustry partnership with a winemaker company, Quinta do Soalheiro, lead to the dawn of new products, both at a lab and industrial scale, aiming to be presented as high value alternatives/ solutions to the current leather-based material/ product status quo, embedding the in vino veritas soul into the aesthetics essence of these composite textile materials. This is the story of the InVinoTex, a brand and a design movement of sustainability of the alternatives to leather-based materials.

RESULTS AND CONCLUSIONS

From all the combination trials conducted, the synergy was identified as optimal for amaterial that comprises three layers, the first one being the textile substrate, a 100% Cotton Interlock Knit with a mass per unit area of 410 g/m^2, and two overlayed coating layers. These layers are obtained by processing, a 350 g/L stable foam with c.a. 20% content of green and biobased polyurethane (considering the nature of its source, vegetal instead of petroleum based and/or its biodegradability), c.a. 68% of synthetic waterborne polyurethane and c.a.12% of grape marc powder (0.05 mm to 0.5 mm of particle size distribution, on both layers). As a result, an elegant textile-based alternative to leather emerges, as highlighted in Figure 1, with a weight of 590 g/m2 and 0.8 mm thick, keeping the original traces of the incorporated wine industry waste, prompting an immediate immersion into its growing bucolic ecosystem, and/or triggering an association to other products derived from this raw material.

Figure1– Photographic example of the aesthetics of an InVinoTex material

In terms of the technical performance criteria, this product excels for its inherent crocking and abrasion resistances (level 4/5, under the test conditions of ISO 105-X12 for 20 cycles and level 1,under the test conditions of ISO 5470-2 for 25600 cycles, respectively), as well as its high, 3-4, colour fastness classifications to water spotting (for all the criteria evaluated under the test conditions of ISO 15700), being in compliance with all the flammability behaviour performance of FMVSS 302 standard. Noting that all these properties are carefully imparted and intertwined with the perfect balance of elasticity and flexibility.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the partnership with Vinusoalleirus, for all the *grape marc* supplied and all the shared encouragement.

The basis for this new material was established as a result of a joint partnership assembled in the TEXBOOST project, in which Tintex teamed-up with CTIC, CITEVE and CENTI, the entities ofthetechnological and scientificnational system, along with the companies, Sedacor and Têxteis Penedo.

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ID 174
POTENTIAL OF HEMP FOR SUSTAINABLE FUTURE

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ABSTRACT
This paper presents holistic approach hemp potential in terms of sustainability of the fibres and other raw materials delivered during hemp processing as well as sustainability of technologies applied for the whole value chain. Analysis of world hemp market allows to forecast of direction of hemp sector development up to 2027.

INTRODUCTION
Currently *Cannabis sativa* L. is one of the most important plant delivering raw materials for manufacture of sustainable bio-products. Multi-perspective potential of the plants and possibilities of use them in different sectors of economy makes the industrial hemp attractive for agriculture, textile, construction, composite, transport, energy, medicine, food, and other industries.
Hemp has got an appreciation due to its appropriateness to sustainability requirements, e.g. environmental, economic and social issues together, which are included in European Green Deal addressing: "no net emissions of greenhouse gases by 2050", "economic growth decoupled from resource use" and "no person and no place left behind". Sustainability is a high priority not only for Europe, also United Nations established Sustainable Development Goals SDGs. Hemp well corresponds with eight from the 17th SDGs: (2) end hunger, sustainable agriculture, (3) good health and well-being, (7) clean energy, (8) sustainable economic growth and decent work, (9) industry, innovation, infrastructure (12) sustainable consumption and production patterns, (13) action to combat climate change, (15) life on halt and reverse land degradation and halt biodiversity. This paper discusses aspects of hemp sustainability and world hemp market changes.

RESULTS AND CONCLUSIONS
The all stages of total value chain of hemp e.g. growing, processing, use and finally recycle/reuse/ biorefination/waste management, fulfil principles of mention above strategies and can contribute to the fight against greenhouse emission. Aware consumers are looking for bio-based products to be sure that they make their small contribution to protect the Earth against climate change. From this reason, the hemp-based products market grows quickly at the whole world, Fig.1.

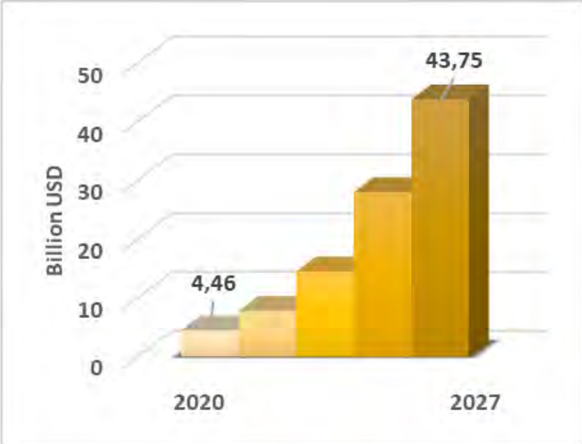

Fig. 1. Forecasted size of global hemp fiber market up to 2027

Conducted analysis of different methods of hemp processing, technologies, raw materials, by-product and waste use according to sustainability principles concerning development of European and World hemp marked allows to conclude, that hemp has great potential for sustainable future.

ACKNOWLEDGMENTS

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ID 175
OBTAIN CELLULOSE FIBRES FROM RICE STRAW THROUGH AN INTEGRATED SUBCRITICAL WATER HYDROLYSIS-BLEACHING PROCESS.

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ABSTRACT
This work analyses the capacity of subcritical water extraction (SWE) to modify the lignocellulosic complex of rice straw (RS) in order to facilitate purification of cellulose fibres with greener methods. Two extraction conditions (160 and 180 ºC, under pressure) were applied for 30 min in an integrated SWE-bleaching process and the obtained fibres were compared in their compositional and structural properties before and after bleaching. SWE at 180 ºC was more effective at removing hemicellulose from RS whereas lining and ash contents were similar in fibres obtained for both temperatures.

INTRODUCTION
Rice straw (RS) is one of the most abundant lignocellulosic waste materials in the world that could be used as a source of cellulose [1]. Pre-treatment is a crucial step in the biomass conversion process to break the lignin linkages with the polysaccharides, remove the hemicelluloses and expose the cellulose fibres [2]. Subcritical water extraction (SWE) is gaining attention for its eco-friendly solvent and selective extraction capacity near to lower polar solvents, such as ethanol or methanol [3]. Due to the mildly acidic nature of SWE, this also selectively hydrolyses hemicelluloses without degrading the cellulosic moieties, which is highly desirable for cellulose recovery [3]. Applying SWE treatment, the alkaline pulping and bleaching treatments, usually applied in the cellulose recovery, may be reduced, thus limiting the use of acidic or alkaline solutions that add to the cost of material resistance to corrosion and post-processing technologies to dispose of the contaminant chemicals.
SWE at two temperatures (160 and 180 ºC) was applied to ground and sieved (0.5 mm) RS, using a water to RS ratio of 10:1, prior to the bleaching process, in order to replace the usual alkaline pulping process. The obtained cellulosic residues were characterised at the different process steps, as to their composition and structural properties and the efficiency of the different steps was evaluated.

RESULTS AND CONCLUSIONS
Compositional and thermal analysis revealed the efficiency of SWE at removing different components from the lignocellulosic complex of RS while weakening the plant tissue, thus favouring the subsequent bleaching step. Crystallinity index of the material rose from 40 % in the raw RS to 53 and 55 %, respectively, after the SWE step at 160 and 180, and to 61 and 66 % after the respective bleaching treatment, according to the cellulose enrichment.

Table 1. Yield (with respect to the initial mass of RS), water extractables, and composition of the untreated RS and the cellulosic fractions before and after the bleaching (B) step (mean values ± standard deviation).

Sample	Yield (% wt.)	Water extractables (% wt.)	Cellulose (% wt.)	Hemicellulose (% wt.)	Klason Lignin (% wt.)	Ashes* (% wt.)
RS	-	9.3 ± 0.8	36.7 ± 0.4	19.3 ± 0.1	21.2 ± 0.5	17 ± 2
SWE-160	79.5	15.5 ± 0.4	36.0 ± 0.2	14.2 ± 2.3	22.6 ± 0.2	15 ± 1
SWE-180	75.2	12.3 ± 0.1	38.1 ± 2.0	2.7 ± 0.1	23.7 ± 1.5	13 ± 5
SWE-160-B	35.0	n/a	61.6 ± 0.1	9.7 ± 1.3	4.7 ± 0.6	15 ± 1
SWE-180-B	39.2	n/a	68.7 ± 1.4	3.4 ± 0.4	5.1 ± 1.1	15 ± 3

*Determined from the TGA residual mass.

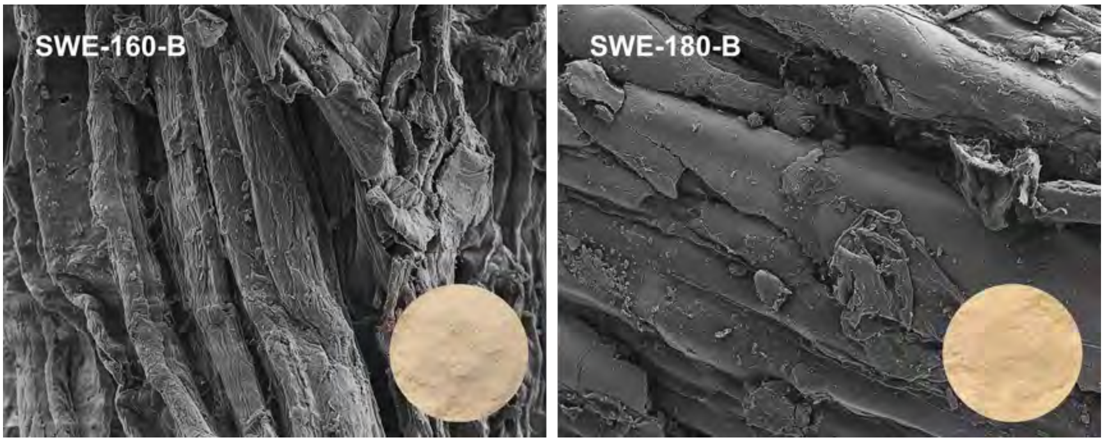

Fig.1 FESEM images of the bleached fibres (X 2000) and visual image.

This study shows that SWE could substitute the alkaline treatment during the cellulose recovery from RS producing an efficient separation of hemicellulose, especially at 180 ºC. Further bleaching step eliminated the residual lignin, giving rise to cellulose fibres with about 70 % of cellulose. Nevertheless, the high silica content in RS was not released from the fibres, and their role in the fibre functionality must be analysed.

ACKNOWLEDGMENTS

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ID 176
COTTON FIBRE-REINFORCED COMPOSITES IN THE AUTOMOTIVE SECTOR USING THE EXAMPLE OF THE TRABANT SMALL CAR SERIES – AHISTORICAL AS WELL AS TECHNICAL ASSESSMENT AND EVALUATION –

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ABSTRACT
The Trabant P601, with 10 outer skin components made of cotton fibre-reinforced phenolic resin, was produced from 1963 to 1990. More than 2.8 million vehicles of the P601 (1963 to 1990) and the Trabant 1.1 (1988 to 1991) were manufactured. The development of the outer skin components made of cotton fibre-reinforced phenolic resin not only compensated for the steel sheet shortages in the GDR but also represents, in many respects, a prime example of innovative material development in connection with innovation pushes:

● Development of a composite material against the background of replacing materials that were difficult to procure.
● Development of new and adapted process techniques, from textile processing of the fibres and pressing technology to assembly and lacquering.
● Development of a natural fibre composite material that meets the high demands of car body construction.
● Production of a natural fibre-reinforced composite material for permanent outdoor use.
● First large-scale use of (natural) fibre-reinforced composites for exterior applications in vehicle construction.
● Implementation of a vehicle concept that also meets the requirements of lightweight construction.

INTRODUCTION
Many people think of cotton as fibres traditionally used in denim clothing. However, cotton is also used in technical products. The development and production of the P 601 Trabant (Figure 1) from the 1950s to 1991 is an inspiring story about the use of cotton fibres in technical products (fibre-reinforced composites). Dr Werner Reichelt's contributions (Reichelt, 2021a &b) to the development and production of composite car bodies led to the success of cotton fibre composites in the Trabant small car series are extremely exciting. Reichelt reports on these developments and provides deep insights into developing new materials and the necessity of designing and realising adapted and completely new manufacturing processes (Müssig, 2022). This talk will present the potential for the technical use of cotton in fibre-reinforced plastics and the associated material innovations from a historical perspective.

RESULTS AND CONCLUSIONS

The fabrication and the exterior components of the Trabant P 601 are shown in Figure 1. The thermoset material with cotton fibres showed high flexural and tensile strengths, high flexural rigidity and particularly high impact strength, exhibiting very low-notch sensitivity. Particularly noteworthy is the high long-term resistance: after 15 years of weathering, the flexural strength decreased by only 7 - 10 %, while the impact strength even showed an increase of 5 to 9 %. In addition, the body components impressed with good thermal insulation, soundproofing, and low mass.

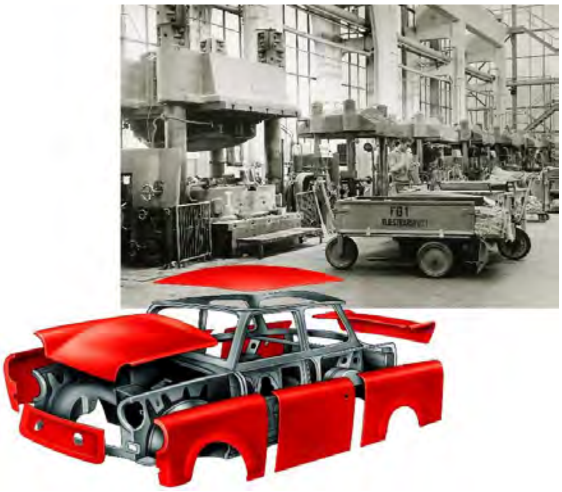

Fig.1: Thermoset press, lower-piston column design, driven by central water hydraulics (the 1970s) and exploded view of Trabant P601 with ten outer skin components made of cotton fibre-reinforced phenolic resin.

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ID 177

INDUSTRIAL TEXTILE WASTES AS A POTENTIAL SOURCE OF NANOCELLULOSE: EXPLORATION OF ITS APPLICATION IN ADVANCED NANOCOMPOSITES

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ABSTRACT

The consumption of cotton-based textiles has been growing steadily over the past few decades. As result, the amount of cotton waste generated has significantly increased, being majority discarded in landfills. Considering these issues, the reuse and valorization of cotton waste is essential. In this way, cotton-based textile waste from middle chain industrial processes was gently provided by Tintex Textiles S.A. and was submitted to sulfuric acid hydrolysis to extract cellulose nanocrystals (CNCs). In an attempt to develop more sustainable extraction processes, sulfuric acid (H2SO4) hydrolysis was conducted with five different concentrations of acid (64, 55, 50, 45 and 35 wt.%). The optimal hydrolysis parameters were 55 wt.% of H2SO4 at 50 ºC with a sample (in grams) to acid (in mL) ratio of 0.6:20. A yield of CNC extraction of 67 % was achieved. Extracted CNCs were analyzed by Scanning Transmission Electron Microscope (STEM), Attenuated Total Reflection – Fourier Transform Infrared (ATR-FTIR) Spectroscopy, X-ray diffraction (XRD) and Thermogravimetric analysis (TGA). After, chitosan-based films reinforced with 5, 10 and 25 wt.% of these extracted CNCs were successfully produced via solvent casting. ATR-FTIR, TGA and uniaxial tensile tests evidenced the improvement of mechanical and thermal properties with the incorporation of CNCs in the matrix, demonstrating the potential of these CNCs to act as a filler in advanced nanocomposites.

INTRODUCTION

Cotton-based textile waste can contain up to 99 % of cellulose content, after being submitted to scouring and bleaching, which are industrial routine preparation procedures. Due to the absence of efficient recycling techniques, most cotton wastes end up in landfills or incinerators and only 25 % of these resources are recycled. This is not only a huge loss of excellent cellulose resources but also contributes to the raise of environmental concerns due to the cotton's anaerobic degradation and burning. Therefore, the application of cotton waste as a raw material for the generation of CNCs might be a promising recycling solution to reduce the disposal of cotton source materials (Shirvanimoghaddam, 2020). Furthermore, this strategy contributes to the circular economy, by converting textile waste into high-value applications. Acid hydrolysis, using sulfuric acid (H2SO4), has been the most used method to extract CNCs due to its efficacy, accessibility, and low price. In the last decade, CNCs have attracted a lot of attention due to their availability, renewability, biodegradability, and their good mechanical properties, turning them into promising candidates as a reinforcement phase in the development of advanced polymeric nanocomposites. In this context, chitosan a sustainable, biodegradable, non-toxic, antimicrobial, antioxidant, anti-inflammatory, hemo-compatible and hemostatic material with excellent film forming properties is a suitable candidate to be used as a polymeric matrix for developing CNCs-filled chitosan nanocomposite with required properties for advanced applications (Ferreira, 2016).

RESULTS AND CONCLUSIONS

Fig. 1 a) displays the STEM images of CNCs obtained with 55 wt.% H2SO4. All CNCs have a needle-like shape with a nanoscale dimension, with an average of 41 nm in diameter and 428 nm in length. ATR-FTIR technique allowed to confirm the absence of hemicellulose and lignin due to the non-appearance of a peak at around 1735 cm-1. The crystallinity index achieved by the isolated CNCs was 83.4 ± 0.86 %, indicating that the reaction parameters selected were ideal to avoid an excessive hydrolytic cleavage of the glycosidic bonds. The presence of sulfate groups from acid hydrolysis using H2SO4 can be evidenced by the appearance of an early shoulder between 135-180 ºC on TGA analyses.

(a) (b)

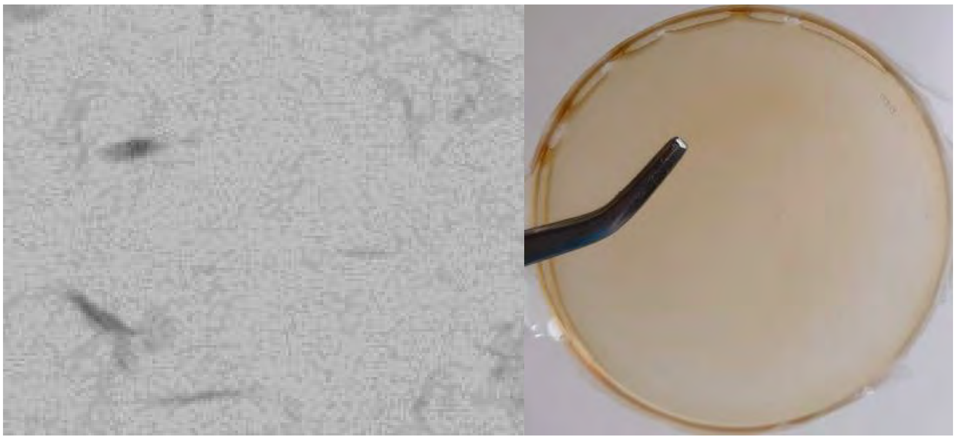

Fig.1 a) STEM micrograph of the extracted CNCs (50 000×). Scale bars represent 2 μm.
b) Chitosan-based films reinforced with 25 wt.% of CNCs extracted.

The chitosan-based films produced showed very homogeneous and smooth surface. The films were observed to have a slightly rougher surface with the incorporation of CNCs. The CNCs appear to be evenly distributed in the matrix, as no notable aggregation was observed (Fig. 1 b)). All chitosan-based films reinforced with 5, 10 and 25 wt.% of CNCs showed improved thermal stability concerning those prepared exclusively with chitosan, which can be ascribed to the strong percolation effect between CNC and the matrix. It was also possible to verify enhanced mechanical properties by the addition of CNCs, namely on Young's modulus and Ultimate tensile strength (UTS). The reduction of strain at failure can be due to the interruptions along the chitosan chains, as a result of the CNCs introduction. Chitosan-based films exhibited antibacterial activity against Staphylococcus aureus and Escherichia coli bacteria, being this effect superior on films reinforced with 25 wt.% of CNCs.

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ID 179
LOW COST COMPOSITE FILMS BASED ON THERMOPLASTIC STARCH AND FIBER-RICH TIGERNUT BYPRODUCT.

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ABSTRACT
In line with the circular economy and zero waste trends, the present study focused on the use of the tigernut milk by-product, rich in starch and fibres, in developing corn starch composite films. To this end, 0, 40, 50 and 60% of tigernut residue, previously dried, ground and sieved (<0.5 mm) was incorporated into glycerol plasticized starch films by melt blending. Moreover, 50:50 formulation was also obtained with previously gelatinized starch in the residue (50:50G). The water content, thickness, water vapour permeability, mechanical and optical properties, solubility, and heat-sealing capacity were characterized for each film formulation. The results showed that it is possible to substitute up to 60% of starch by the tigernut residue to obtain starch-based composite films, although these exhibited worsened physico-chemical properties, being mechanically less resistant and stretchable, with lower water vapour barrier capacity, gloss and water solubility. Of the films studied, the formulation 50:50G exhibited the optimal properties, due to the contribution of the pregelatinized starch in the residue to the film matrix. This pre-treatment gave rise to a better compatibility between the by-product components and starch, promoting adhesion between the hydrophilic matrix and the fibrous fraction, hence improving their mechanical performance.

INTRODUCTION
The use of vegetable by-products as part of food packaging is a great alternative to their valorization, while contributing to alleviate the problems they generate. These lignocellulose wastes from agroindustry contain carbohydrates, fibers or bioactive molecules, such as phenolic compounds or carotenoids that can be of interest to produce active packaging materials. Moreover, given the current concern for environmental impact, together with the large amount of plant residues produced, these are increasingly of interest for sustainable packaging (Dilucia et al., 2020). The solid residue obtained from the production of horchata, a traditional drink from the Valencian Community in Spain is highly rich in fiber and starch, with a remarkable content in total phenols, around 16-50 mg GAE/100 g dry matter (Roselló-Soto et al., 2018), mainly consisting of phenolic acids and flavonoids, which provide the product with antioxidant activity (Roselló-Soto et al., 2018).
The general objective of this work was to develop biodegradable films from thermoplastic starch that incorporate Cyperus esculentus residue from the production of horchata for its valorization in the framework of circular economy. To this end, glycerol plasticized corn starch composite films, containing 0, 40, 50 and 60% of tigernut milk by-product, were obtained and characterized. The effect of pregelatinizing of the residual starch in the by-product was analyzed in the 50:50 formulation.

RESULTS AND CONCLUSIONS

The results from the tensile tests are shown in Table 1. The stress and strain at the breaking point decreased significantly with increasing residue content, i.e., the films became less tough and deformable and thus more brittle. This points to a low compatibility between the residue particles and the matrix, which causes the mechanical properties of the matrix to worsen. However, the mechanical strength of the 50:50G films was the highest, even higher than that of the control film ($p<0.05$), which highlighted the better capacity of the gelatinized residue to be fully integrated into the starch matrix, promoting a reinforcing effect. In fact, the 50:50 and 50:50G formulations behaved mechanically very differently, as shown in Figure 1. The previous starch gelatinization in the residue provides the films with a more plastic mechanical behaviour, with no cracks or micro-cracks at low deformations, exhibiting higher stretchability than the 50:50 composite without the gelatinized residue.

Table 1. Tensile strength (σ), percentage of elongation at break (Ɛ%) and elastic modulus (EM) of composite films with different ratio of particles in the matrix.

Film	σ (MPa)	Ɛ(%)	EM(MPa)
0:100	$3{,}6 \pm 0{,}6^{b}$	22 ± 6^{b}	79 ± 31^{a}
40:60	$1{,}84 \pm 0{,}16^{a}$	$0{,}7 \pm 0{,}3^{a}$	569 ± 191^{b}
50:50	$1{,}5 \pm 0{,}3^{a}$	$0{,}35 \pm 0{,}06^{a}$	553 ± 69^{b}
50:50G	$4{,}3 \pm 0{,}7^{c}$	$1{,}1 \pm 0{,}2^{a}$	538 ± 134^{b}
60:40	$1{,}4 \pm 0{,}2^{a}$	$0{,}27 \pm 0{,}07^{a}$	632 ± 149^{b}

a, b..: different letters in the superscripts indicate significant differences ($p<0.05$) between films at 95%.

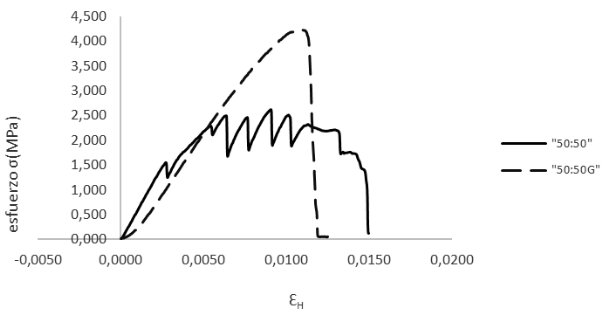

Fig. 1. Typical stress-strain curve at the breaking point of 50:50 and 50:50G formulations.

This study shows that the pregelatinizing of the residual starch in the by-product prior to its incorporation into composites allowed for better compatibilization between the fibrous particles and the starch matrix, by improving adhesion between the hydrophilic matrix and the incorporated fibers, thus increasing their mechanical resistance.

ACKNOWLEDGMENTS

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ID 180
SURFACE FREE ENERGY OF FLAX FIBRES

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ABSTRACT
Aim of the study was exploration of the surface free energy of flax fibers extracted from different varieties of flax: Modran, Nike, B14 after applied successive stages of fiber processing in relationship to their chemical structure. Results of the study proved, that surface free energy of flax fibers was related to their chemical composition, e.g. percentage content of cellulose, hemicellulose, lignin, waxes and pectin in the fiber. The presented work is continuation of research on the relationship between the properties of fibers and their chemical structure (Zimniewska, 2018).

INTRODUCTION
Bast fibers belong to hygroscopic bodies, which means that they are able to bind a certain amount of water or to transfer liquids as a result of surface tension. This phenomenon is directly proportional to the value of the surface free energy of the bast fibres. The free surface energy of bast fibres is ambiguous and depends on the fiber chemical composition e.g.: cellulose, hemicellulose, pectin, lignin, waxes and fats (Gassan, 2000). On the over hand, the amount of individual components in the fiber depends on the plant variety, as well as the method of the fiber extraction from the straw (Morison, 2000).
The surface free energy of natural fibres, including bast fibres, can be tested with use different methods. However, the specific fiber morphology and heterogeneity of its surface make it difficult to obtain repeatability of the test results (Heng, 2007). In the tests conducted within current study, the *Sessile Drop* method was used - a method consisting in “to sessile” a drop on the surface of the sample and based on analysis of the drop shape, the contact angle is determined. Contact angles were tested with use of two measuring liquids: polar with use of HPLC water and non-polar with diiodomethane, are then the data are converted to surface free energy.
The analysis of the surface energy of bast fibres allows to design of fiber processing and modification to ensure fiber properties suitable to further needs.

RESULTS AND CONCLUSIONS
Results of test of chemical composition of fibres extracted from different flax varieties after each technological stages are presented in Table 1. Successive technological stage contribute to reducing the value of surface free energy, regardless of the variety of flax fibres. This is due to the increase in the percentage content of cellulose in flax fibres in subsequent technological stages (Table 1).

Table 1 Chemical composition of flax fibres, according to variety and technological processes

Fiber		Waxes and fats [%]	SD	Pectin [%]	SD	Lignin [%]	SD	Cellulose [%]	SD	Hemicelluloses [%]	SD
MODRAN	Decortication	1.26	0.00	4.62	0.16	4.00	0.16	68.89	1.91	29.35	0.16
	Wet degumming	0.69	0.07	4.41	0.50	4.20	0.16	75.54	1.18	19.62	0.15
	Cottonization	0.97	0.10	4.72	0.39	4.26	0.15	73.51	0.98	16.44	0.23
NIKE	Decortication	1.47	0.07	4.11	0.38	8.60	0.30	64.57	0.85	29.38	0.08
	Wet degumming	0.76	0.00	3.56	0.27	4.46	0.48	77.44	1.58	16.43	0.25
	Cottonization	0.95	0.05	2.39	0.22	4.87	0.51	74.25	0.20	13.84	0.06
B14	Decortication	0.88	0.01	4.16	0.12	5.25	0.03	68.21	1.06	31.02	0.25
	Wet degumming	1.33	0.01	5.43	0.28	6.69	0.48	75.04	0.46	23.92	0.02
	Cottonization	1.72	0.09	3.57	0.23	6.10	0.01	72.20	0.47	20.41	0.09

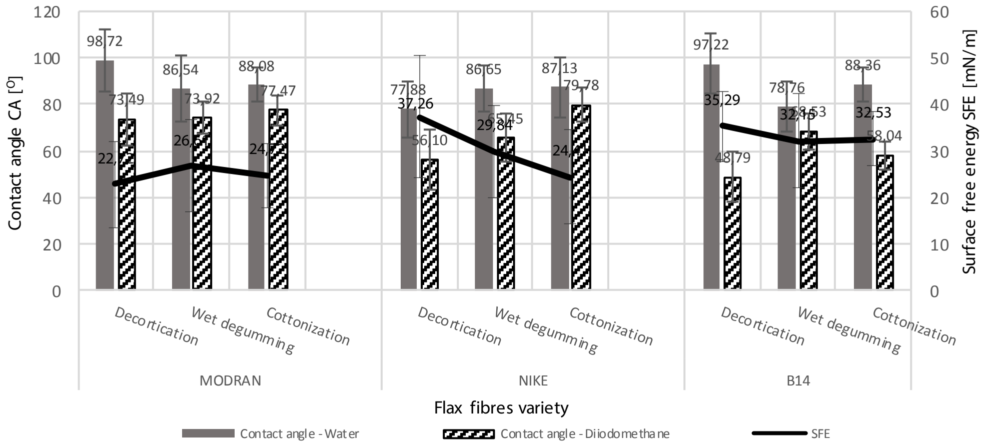

Fig.1 The results of the surface free energy determined by measuring the contact angle method of flax fibres varieties depending on the consecutive stage of fibres processing

The obtained results confirm that the values of the surface free energy of flax fibres depend on the content of individual components in the fibres.

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ID 182
MECHANICAL PROPERTIES OF SISAL FIBERS BY SINGLE FIBER TENSILE TESTS: DIGITAL IMAGE CORRELATION TECHNIQUE AS A STRAIN MEASUREMENT

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ABSTRACT
Sisal fiber demonstrates interesting properties to be used as a reinforcement in biocomposites. In this study, sisal fibers were extracted from the leaves of an agave sisalan plant by manually decorticating method. Water retting was the applied to the decorticated fibers for 30 days. Single fiber tensile test was performed to investigate the tensile properties of sisal fibers using digital image correlation method. The tensile strengths of water retted and unretted fibers were 679 and 718 MPa, respectively. The strain at failure and elastic modulus of water retted and unretted fibers were obtained as 2.0 and 34 GPa, as well as 1.5 and 48 GPa, respectively. Retting seems to predominantly influence the strain at failure of the fibers. Although even this effect is probably not statistically significant when the standard deviation is considered.

INTRODUCTION
Single fiber tensile testing is a technique to determine the mechanical properties of a fiber. Since only a limited amount of material is required, this test can be performed in an early stage of the material and product development. In contrast to synthetic fibers, datasheets on the mechanical properties of natural fibers are seldomly available, especially in the case of new and unexplored materials (Depuydt et al., 2017). The sisal fibers used in single fiber tensile tests are commonly technical fibers which consists several elementary fibers. The displacement is applied to the single technical fibers by gripping the fiber from end to end. There are two methods of displacement measurement in single fiber tensile testing. The first method is direct measurement of displacement in accordance to ASTM D3822, using a tensile test machine (Bermudo et al., 2019). In this method, the displacement is determined by the movement of clamps. This results in errors due to deformation of the test machine and fiber slippage in the grips. The second method is displacement measurement in accordance to ASTM C1557, using digital image correlation (DIC) (Sarasini, Tirillò and Seghini, 2018). DIC is used to determine a fiber's strain and modulus by considering a fiber length in between two optical flags to avoid deformation error due to fiber slippage (Mehdikhani et al., 2016). A total of 100 tests were performed on both water-retted and unretted fibers.

RESULTS AND CONCLUSIONS
The results from the single fiber tensile tests are shown in Fig. 1. Fiber diameters were obtained by optical microscopy measurement and calculation based on weight of the fibers. The mean tensile strengths for

the water-retted and unretted fibers were calculated. Density of the fibers were measured. The density of water-retted and unretted fibers was determined at different fiber lengths. Table 1 shows the results for the densities of the fibers.

The most significant difference of fiber densities appears between retted and unretted fibers was for higher fiber length (10 mm). Density of milled powder is more than all other types.

Table 1 Densities of the various sisal fiber types using helium gas pycnometer.

Fiber (type, length)	Weight, g	Pressure1, Pa	Pressure2, Pa	Density, g/cm3	StD
Retted, 10 mm	1.23	$124.24*10^3$	$36.75*10^3$	1.32	0.010
Unretted, 10 mm	1.40	$124.18*10^3$	$36.82*10^3$	1.37	0.004
Retted, 5 mm	2.24	$124.45*10^3$	$37.44*10^3$	1.41	0.005
Unretted, 5 mm	2.16	$125.07*10^3$	$37.58*10^3$	1.42	0.007
Milled powder	3.84	$124.18*10^3$	$38.47*10^3$	1.45	0.002

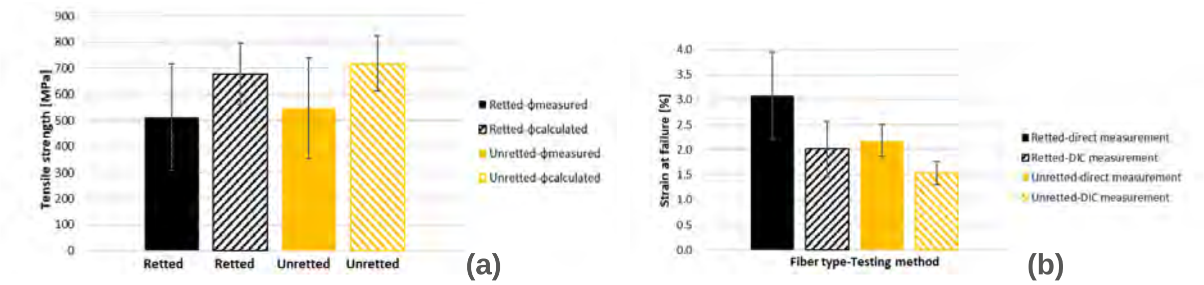

Fig.1 Single fiber tensile test results of retted and unretted fibers: (a) strength, (b) strain

This study shows that there is no substantial difference in tensile strength of water-retted and unretted fibers. But, the results reveal that retting has an influence on the strain of the fibers.

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ID 185
PLASMA AND DIGITAL INK JET - GREEN TECHNOLOGIES FOR IMPROVEMENT DURABILITY AND SUSTAINABILITY OF FUNCTIONALIZED COTTON KNITWEAR

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ABSTRACT
In order to improve the quality of digital inkjet printing, the influence of low-pressure plasma treatment on cotton fiber knitwear samples was tested. It is known that the application of plasma on the surface of textile material changes its physical and chemical properties, resulting in a change in surface morphology. In the work, surface activation with oxygen and argon plasma was performed to improve the functional properties of the surface, which was followed by processing with digital inkjet pigment printing. The durability of the digital printing was tested by wash care and abrasion resistance (using the Martindale instrument). The effectiveness of plasma pretreatment on the fastness of digital pigment inkjet printing was characterized by fiber surface analysis using a SEM microscope, and the quality and depth of the print was evaluated by spectroscopic analysis of color coordinates, color strength and total color change (dE).

INTRODUCTION
The use of plasma as an ecologically and economically acceptable medium for surface modification, with which new functional properties of the textile material are achieved, is becoming an extremely acceptable method of surface treatment of materials in comparison with various conventional processing methods, which include various chemical, mechanical and thermal processes with a very high degree of environmental impact. The advantages of plasma are mainly its applicability to all types of materials and the possibility of obtaining functional properties without affecting the basic characteristics of the textile material and with very low environmental impact. In addition to plasma, inkjet printing for surface printing is a relatively new printing technology that is experiencing exponential growth, and the success of the printing is reflected in the quality of the printer, which prints the surface with high resolution. Inkjet is now a printing technology that can be used to print on all types of substrates, including various textile materials with different structures and shapes. Due to the new demands on the market, the textile industry itself has to develop new solutions aimed at shortening the time for product manufacturing while increasing the quality level and reducing the environmental impact. Due to their efficiency, digital printing technologies are increasingly used in the textile and fashion industry [1-3].
In this work, the effect of low-pressure plasma pretreatment on knitwear samples made of cotton fibers was studied to improve the quality and durability of digital inkjet printing. The changes in the properties of cellulosic knitwear after pretreatment of the samples with oxygen and argon plasma at optimized parameters of

the surface activation process were investigated, which affect the durability and quality of digital pigment inkjet printing after washing and wearing processes.

RESULTS AND CONCLUSIONS

The pretreatment of the knitted fabric was carried out using the Nano LF -40kHz plasma system (Diener company) and printed using an Azon Tex Pro digital printer from Azonprinter with piezoelectric droplet formation.

Table 1 Total color difference (dE) of untreated and plasma pretreated fabric - expressed for the washing process

Sample label/Properties	dE (CMC) Cyan	Magenta	Red	Yellow	dE (CIE) Cyan	Magenta	Red	Yellow
CO_UN	**3.17**	**2.92**	**3.01**	**4.53**	**4.61**	**5.61**	**5.67**	**12.36**
CO_Ar / 500 W, 2 min	1.58	2.32	1.47	1.96	**2.83**	**4.71**	**2.03**	**4.52**
CO_1 / O_2, 500 W, 2 min	**3.31**	**3.36**	0.82	1.42	**5.22**	**6.99**	1.46	**3.74**
CO_II / O_2, 500 W, 5 min	**3.41**	1.57	1.13	1.42	**5.65**	**2.93**	**2.21**	**3.87**
CO_III / O_2, 900 W, 2 min	**2.89**	**2.36**	1.83	0.53	**4.61**	**4.69**	**3.57**	1.31
CO_IV / O_2, 900 W, 5 min	**2.07**	0.99	1.41	0.82	**3.10**	1.81	**3.22**	**2.27**

Footnote: parameters gas flow rate of 50% and pressure 0.25 - 0.46 mbar are the same for all treatments.

a) CO_UN b) CO_O_2/ ink jet printed c) CO_Ar/ ink jet printed

Fig.1 SEM Images of untreated and plasma pretreated+inkjet printed cotton samples: **a)** untreated samples, magnification 1000x; **b)** oxygen plasma, magnification 2000x and **c)** argon plasma, magnification 4000x

When analyzing the surface morphology, a significant effect of the plasma on the distribution and coverage of the pigment paste and polyacrylate binder on the fiber surface with distinct pigments of the printing paste on the surface is visible. Such an effect is particularly visible after pretreatment of the surface with argon plasma; the print is permanent and embedded in the deeper parts of the fiber surface created by the physical action of argon in the plasma.

ACKNOWLEDGMENTS

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ID 186
ASSESSMENT OF THE PHYSICAL-MECHANICAL PERFORMANCE OF MAGNESIUM-BASED FIBER CEMENT SUBMITTED TO ACCELERATED CARBONATION

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ABSTRACT
This work evaluated the influence of the accelerated carbonation process in the production of fiber cement boards with magnesium oxy-sulfate cement matrix (MOS). The boards showed improvements in the mean values of MOR, MOE, LOP, and SE after curing in a CO_2-rich atmosphere. The carbonation process was confirmed by X-ray diffraction and thermogravimetric analysis, which showed the formation of new phases after the carbonation process.

INTRODUCTION
Magnesium oxy-sulfate cement (MOS) is a type of magnesia cement produced by the reaction between magnesium oxide (MgO) and a magnesium sulfate solution ($MgSO_4$) [1]. This type of cement has the advantages of being lightweight, low energy consumption, good cohesion, and thermal insulation. Similar to the production of Ordinary Portland Cement (OPC) from limestone, MgO can be produced by calcining magnesite ($MgCO_3$) at lower temperatures (1450°C for OPC and 750 °C for MgO production), which reduces the amount of energy used in the calcination process and minimizes the release of greenhouse gases into the atmosphere [2,3]. The final properties of MOS cement are dependent on the hydration products formed after the hardening of the pastes. The MOS phase with the highest chemical stability at temperatures near 23ºC is phase 5.1.7 ($5Mg(OH)_2$-$MgSO_4$-$7H_2O$) and presents high physical-mechanical performance and water resistance. The worldwide need to replace asbestos fibers with alternative reinforcements is a concern for fiber cement industries. The use of lignocellulosic fibers represents an alternative due to its properties. The use of MOS matrices in fiber cement production is used to ensure greater chemical stability of vegetable fibers due to the lower alkalinity of the system after curing the materials [3]. The objective of this work is to investigate the production of MOS fiber cement and evaluate the influence of accelerated carbonation in the improvement of the physical and mechanical properties of the composites.

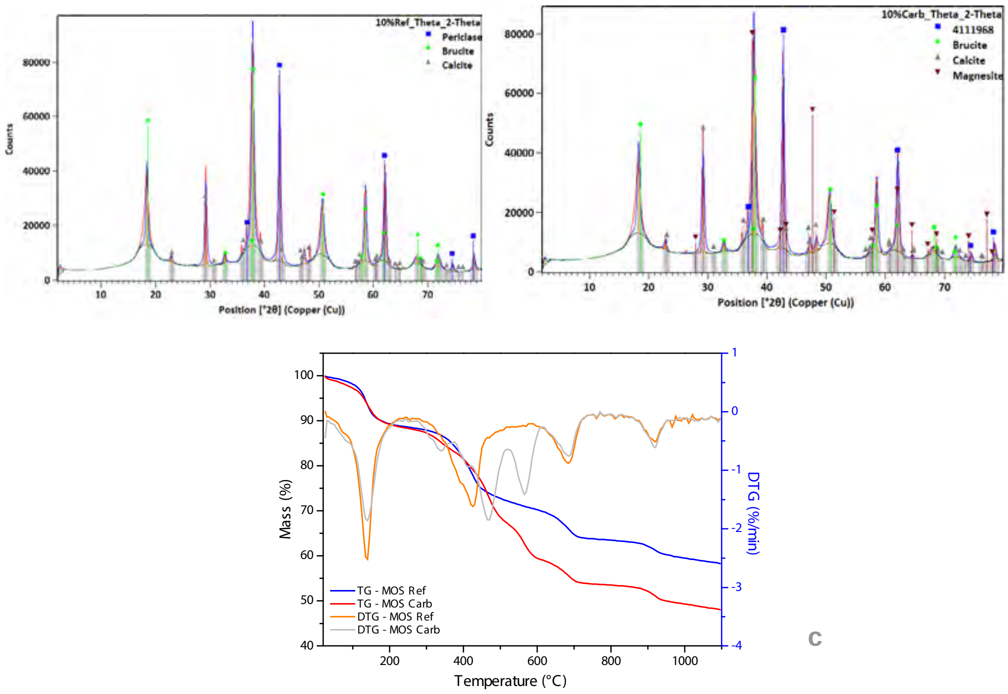
RESULTS AND CONCLUSIONS
The mechanical and density results of the materials are presented in Table 1. It is possible to observe that the carbonation process promoted an increase in the mechanical strength of the investigated slabs. The increase in MOE and SE values are related to the formation of carbonation products after curing in an at-

mosphere rich in CO_2. The X-ray diffraction results (Fig 1a-b) showed the formation of magnesite ($MgCO_3$), which is the main carbonation product of magnesian cement. The formation of this new compound occurs preferentially in the pores and voids between the fiber and the matrix, which reinforces the energy transfer during mechanical loading and increases the specific energy of the composite, as observed in this work. The formation of $MgCO_3$ was confirmed by thermogravimetry analysis (Fig 1c), which showed a higher mass loss stopping the carbonate materials. Such an increase in mass loss is related to the decomposition of magnesite at temperatures near 550 - 600°C.

Table 1. Mechanical and physical proprieties of the MOS-based fiber cement (carbonated and non-carbonated).

Sample - MOS	MOR (MPa)	StD	LOP (MPa)	StD	MOE (GPa)	StD	SE (kJ/ m^2)	StD	Density (g/ cm^3)	StD
Ref	17.6	2.2	11.2	0.8	10.2	0.3	3.7	0.2	1.59	0.01
Carb	21.2	0.6	12.7	1.3	11.6	0.4	3.4	0.5	1.64	0.02

a



b

c

Fig 1. X-ray diffraction (non-carbonated (a) and carbonated (b)) and TGA(c) of the MOS-based fiber cement.

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ID 187
AUTOMATED, NEAR-NET-SHAPE MANUFACTURING OF BIO-BASED LIGHTWEIGHT SANDWICH STRUCTURES

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ABSTRACT
As part of the ecoWing junior research group, a fully automated, reproducible manufacturing process for bio-based sandwich structures with high geometric flexibility was developed. In the sandwich structures, flax fibers with bio-based epoxy resin are used as cover layers and veneer wood as sandwich core. Natural fiber rovings are processed using fiber placement technology. A novel bio-based sizing improves the textile processability of the natural fibers and increases the fiber-matrix adhesion. The automated process chain and the materials used were mapped and evaluated in a life-cycle engineering model during the project. The production of a rotor blade for small wind turbines demonstrates the transferability of the project results to practical applications on a laboratory scale.

INTRODUCTION
Energy and resource efficiency are key objectives in research in view of the growing world population and the associated increase in demand for energy and raw materials. Product sustainability, lightweight construction and digitalization are three approaches to meeting this objective. These can be achieved by using new innovative materials and manufacturing technologies. Sustainable and bio-based lightweight sandwich structures from automated, near-component shape production have a high potential that must be exploited. Compared to GFRP These materials are characterized by a good CO_2 balance, allow a waste-reduced production process thanks to stress-appropriate material design and near-component-contour production. Since they are based on the use of renewable raw materials and are also characterized by a low material density, resource efficiency is high.
It should be emphasized that they have a significantly lower ecological impact than conventional sandwich composites made from GFRP and polyemeric Form cores and thus make a significant contribution to the so-called circular economy?? Why ??. By using the project results, new sustainable and bio-based lightweight products can be developed. A first example of this is a rotor blade for a vertical axis wind turbine, which was created as part of the interdisciplinary junior research group ecoWing.

RESULTS AND CONCLUSIONS

Within the framework of the ecoWing project, various goals along the entire process chain were achieved, which are divided into the areas of material characterization of the sandwich structure component, development of bio-based sizing and adhesion promoters, analysis and optimization of the adhesion properties of the sizing, micro- and macroscopic investigation of the interface systems, automation of natural fiber preforming, investigation of the fiber tape stabilization and fiber layer fixation, characterization of the ecoWing fiber structure, automation of the production of bio-based sandwich structures, development of demonstrator plant of the ecoWing process chain, characterization of the ecoWing sandwich structure, economic and ecological evaluation, development of a multi-objective life cycle engineering concept, development and production of the demonstrator of a wing for a vertical axis wind turbine, and life cycle oriented economic and ecological evaluation of the ecoWing process.

Finally, it could be shown that a sustainable bio-based manufacturing process of blades for vertical wind turbines is possible and that significant CO_2 savings can be realized. Using LCE, it was possible to demonstrate that, depending on the energy mix available in the country of production, very large savings in CO_2 emissions are achieved compared to conventional wind turbine blades made of glass fiber-reinforced plastic with petrochemical resin. However, it must be critically acknowledged that with the current CO_2 equivalents per kWh in Germany (402 g/kWh), there is still a clear need for optimization. This is mainly due to the higher processing temperatures and the associated higher energy consumption during the processing of the biobased fiber-reinforced plastic composites. However, if the current energy mix of countries such as Portugal, France or Sweden is assumed (220 g/kWh, 67 g/kWh, 9 g/kW), there are already significant CO_2 savings compared to conventional production, since the main CO_2 driver in the process chain of bio-based fiber-reinforced plastic composites is the required process energy.

In the course of further investigations, the CO_2 savings potential is to be optimized further and transferred to blades for conventional horizontal-axis wind turbines.

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ID 188
THE FATIGUE AND MOISTURE PERFORMANCE OF BIAXIAL FLAX COMPOSITES: IN SEARCH OF MICRO WIND TURBINE BLADE MATERIALS

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ABSTRACT
Manufacturers of wind turbines are seeking alternatives to conventional composites for blades to reduce embodied emissions and end-of-life landfill waste. This work targets the replacement of short fibre glass/polyamide (30% fibre content) with flax/bio-epoxy (*ca* 30% fibre content) for a 1kW micro wind turbine (*ca* 1m blade length). A focus is on characterising and understanding the fatigue and moisture behaviour of flax biocomposites, produced with a new non-crimp biaxial reinforcement developed as part of the FLOWER project by industry partners.

INTRODUCTION
Rotor blades are constructed with non-recyclable composites – 2.5 Mtonnes annually – that are landfilled at scale at their 15-25 year end-of-life. Europe alone will decommission 25ktonnes of blades a year by 2025, pushing the European wind energy agency, WindEurope, to call for a ban on landfilling of decommissioned wind blades by 2025 (WindEurope, 2020). Wind energy firms are also aiming for carbon neutrality by 2030, and zero-waste wind turbines by 2040. Plant fibre composites may have some role to play in sustainability of the wind-energy sector – at the very least, they offer incineration for energy recovery, and sequester 1.5kg of CO2eq in every kg of plant fibre used. This study aims to replace short glass/polyamide (30% fibre content) in 1m blades with a flax biocomposite. Flax fibres have low density, impressive specific properties, and can be produced into a plethora of composite reinforcement forms like no other natural fibre (Bourmaud et al., 2018; Pil et al., 2016).
A few previous studies have explored the potential of multi-axial flax/epoxy composites for micro and small wind turbine blades: from 0.6m blades for car roof-tops (Bottoli and Pignatti, 2011) to 3.5m blades for a farm (Shah et al., 2013). While a few studies, including (Wuzella, 2018) have demonstrated that biocomposites can meet stiffness and strength requirements for blades, data on fatigue performance and resistance against weathering is limited. Knowledge at the material-scale is nevertheless increasing (Bensadoun et al., 2016; Pantaloni et al., 2022). This study focuses on characterising the fatigue and moisture behaviour of flax biocomposites, produced with a new non-crimp biaxial reinforcement developed as part of the FLOWER project (https://flower-project.eu/) by industry partners Depestle Group.

RESULTS AND CONCLUSIONS

Samples of biaxial flax/biepoxy composites (31.5% fibre volume fraction) have been produced and basic mechanical evaluation completed (±45◦: E_x = 8.0±0.1 GPa, strength σ_x = 93±1 MPa). These properties are comparable to that of similar multiaxial plant fibre composites, and exceeds that of short fibre E-glass composites. The moisture ingress process in these composites is being observed through mass evolution analysis (Fig. 1) and microstructure analysis. Micro-CT scans suggest an initial porosity content of 1.2% through micro-CT scanning. Tension-tension fatigue testing is also in progress and stress-life curves will be produced and compared to literature. An aspiration is to use this stress-life data of the unweathered samples to predict the degradation in fatigue behaviour when the samples are/have been wet. This is a un-studied subject in literature. Simultaneously, a set of three flax bioepoxy turbine blades is being produced (Fig 1).

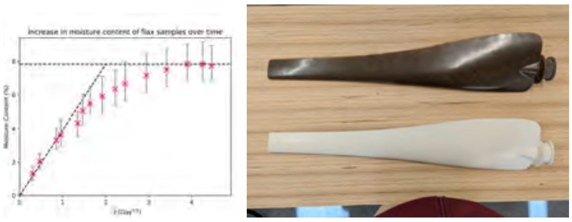

Fig.1. (left) Moisture behaviour of biaxial flax composites to measure diffusion coefficient. (right) Injection moulded E-glass/polyamide and demonstrator flax/bioepoxy blade.

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ID 189
IDENTIFYING NEW SOLUTIONS FOR SUSTAINABLE ADVANCED COMPOSITES

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ABSTRACT
The paper presents a solution to use textile-based waste to produce a textile reinforced plastic composite for replacing wood panels in civil engineering applications. It discusses the manufacturing process, specific characteristics of the resulting composite panels and a comparison with the properties of an 8 mm OSB board (wood panel).

INTRODUCTION
Composite materials have shown a huge potential for replacing traditional materials, many times with improved performance due to their specific architecture and characteristics. Textile reinforced composites (TRC) are well used in a large variety of applications, from mechanics to sports, automotive and civil engineering sector (Echeverria, 2019). The main advantages of TRCs refer to controlled anisotropy, excellent weight to strength ratio, resistance to corrosion, climate factors and environmental humidity (Nermin, 2017). Still, the last two decades and the environmental changes that happened in this period put a lot of pressure on the textile sector including the production of textile reinforcement for plastic composites. There is a lot of research regarding the replacement of high-performance materials with more sustainable variants without affecting the composite`s level of performance.
The paper describes a new composite material designed to replace wood panels for civil engineering applications, developed using wastes generated by the textile sector as reinforcement and by the packing industry as matrix and evaluates the options for increasing the sustainability of such products.

RESULTS AND CONCLUSIONS
The idea of using textile waste as reinforcement is well suited for thermoforming manufacturing of composites, as this process is low cost and with high productivity. Currently one of the main issues of the textile industry is the huge amount of industrial and post-consumer waste that need to be recycled. The new waste based composite material proposed are produced using textile waste, cut to dimension, as reinforcement. Current developments used polyester woven fabrics, but any type of waste can be considered: natural fibres, like cotton, hemp, linen, wool, as well as synthetic fibres, like acrylic, PP and PE (Ailenei, 2021). The main design criteria involved in the textile waste reinforcement are the dimension of the scraps, the inherent material strength, the direction of the scraps and the fibre volume fraction. The steps followed for the production of waste-based composite panels is illustrated in Figure 1. It involves mixing the waste for reinforcement with the one for the matrix, create the panels according to shape and thermoforming.

Fig.1 Technological process for producing waste-based composite material

The characteristics of the experimental variants produced (matrix type, temperature, thermoforming duration, pressing force and matrix proportion/FVF) are presented in the Table 1 as variation intervals.

Table 1 Variation interval for the characteristics of textile waste-based composites

Characteristic	Density [kg/m3]	Bending [N/mm^2]	Young Modulus [N/mm^2]	Tensile strength [N/mm^2]
Variation interval	936 - 1283	4.7 – 35.2	239.0 – 1437.4	3.95 – 23.19

The variation intervals recorded for the panels produced show a large range of possibilities, according to the requirements of the specific application. This shows that by controlling the manufacturing process and the type of waste materials used, panels with accurate properties can be designed and produces. Table 2 present in comparison the properties for an 8 mm OSB board.

Table 2 Characteristics of an 8 mm thickens OSB boards

Characteristic	Density [kg/m^3]	Bending [N/mm^2]	Young Modulus [N/mm^2]	Tensile strength [N/mm^2]
Variation interval	643	9.9	1499	2.26

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ID 190
INFLUENCE OF ACCELERATED WEATHERING ON MECHANICAL PROPERTIES OF NATURAL FIBER REINFORCED BIOCOMPOSITE LAMINATES

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ABSTRACT
The increased sensitivity to environmental issues is responsible for the extraordinary acceleration towards the development of new sustainable materials potentially capable of replacing synthetic ones even in semi- or structural applications. In this frame, special interest is directed to composites based on “green” matrices also used in blends and reinforced with natural fibers. This work explores the mechanical properties of composite laminates obtained by combining poly(lactic acid)/polybutylene adipate-co-terephthalate blends with commercial woven fabrics consisting of jute and flax fibers.. Furthermore, attention is paid to the possible influence of wet-dry cycles on the overall performances and, therefore, on the durability of the same composite systems intended for applications in the transport and construction sectors.

INTRODUCTION
The extensive use and consumption of plastics all over the world and the recognized environmental impact of products at the end of their useful life, often also related to incorrect waste disposal, are the leitmotif that has increasingly oriented interest in new sustainable materials, alternative to synthetic ones, intrinsically biodegradable or in any case derived from renewable sources. Among these plastics, polylactic acid (PLA) resins, derived from the fermentation of corn starch and other polysaccharide sources, are widely used for numerous industrial applications ranging from biomedical/pharmaceutical to food packaging fields. However, the intrinsic brittleness of PLA resins often limits further expansion of their potential range of uses and this drawback can be brilliantly overcome by different strategies (Zhao, 2022). Previous research efforts showed that blending PLA with poly(butylene adipate-co-terephthalate) (PBAT) is an adequate solution to face this challenge (Yan, 2020) (Chen, 2021).
In the light of current knowledge on this topic, this work explored hot compacted laminated composite structures prepared starting from two PLA/PBAT blends with composition 20/80 and 80/20 by weight and reinforced with commercial fabrics based on jute fibers and flax fibers.. Specimens of suitable dimensions, cut from each sample plate both as produced and after exposure to specific environmental aging conditions, were characterized by flexural and low-velocity impact tests. Furthermore, the results obtained were integrated and supported by the morphological analysis of the damaged areas.

RESULTS AND CONCLUSIONS

Both fibers were effective in enhancing the mechanical response of neat PLA/PBAT blends. Considering the low velocity impact (LVI) response of laminates, the improvement in mechanical strength was highlighted by the increase in maximum peak force recorded during LVI tests: for PLA/PBAT/JUTE laminates, the increase at 25 °C was about 105% at an energy level of 2 J and 104% at an energy level of about 6 J. Under the same impact conditions, flax proved to be more effective than jute and the peak force increased by a further 12% at 2 J and 14% at 6J with respect to PLA/PBAT/JUTE. Survey on permanent indentation revealed the penetration of PLA/PBAT from 4 J as energy level, while at an energy level of 6 J, permanent indentation of about 772 μm was measured for PLA/PBAT/JUTE laminates. The flax-based composites featured also a significant decrease in permanent indentation extent. The better performance of flax-based laminates was confirmed also after the exposition to wet-dry cycles.

Overall, the results achieved so far allow to appreciate the performance of the materials studied in semi-structural applications and their reliability even in outdoor conditions. Further tests are underway to investigate the degradative mechanisms induced by the specific environmental exposure conditions.

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ID 191
ENERGY ABSORPTION OF FLAX FIBRE REINFORCED COMPOSITES AND THE EFFECT OF HYBRIDISATION

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ABSTRACT
This study provides an assessment about the improvement on the strength of natural fibre reinforced composite through hybridisation with synthetic fibres. Three different composite laminate configurations were considered reinforced with flax and carbon, and samples tested with a pendulum impact system. Hybridisation improves the energy absorbed and this depends on the percentage and location of the synthetic fibres within the laminate.

INTRODUCTION
Composite materials have received appreciation for their lightweight and high strength to weight ratio of the mechanical properties. Also, the advantage that the properties can be tailored to specific requirement has made them attractive. Composites reinforced with natural fibres are sometimes given preference over synthetic fibres as they are eco-friendly and are made from renewable and naturally abundant resources; hybridisation helps to improve on the properties. Flax is one the kind of natural fibre that has today attracted application in sports equipment, automotive industries etc.
Lopez-Alba etal [1] presented results about the energy absorption of non-hybrid natural fibre reinforced composites. The samples tested were made of flax woven fabrics (twill and hopsack) and non-woven mat mixture of hemp and kenaf, with matrix materials of polyethylene and polylactic acid. In another study, Meredith etal [2] presented the report comparing the specific energy absorption of separate samples of non-hybrid composites reinforced with carbon fibre, Woven flax and woven jute and observed that the results varied with the fibre strength and the volume fraction. While in the review conducted by Jusoh etal [3], they discussed about the characterisation of natural fibres and noted that corrugated cores of metallic materials in sandwich structures could be replace with natural ones such as kenaf, wood dust etc. Prasad etal [4] presented the work on the tensile, flexural and interlaminar shear properties of unmodified and nano TiO_2 coated flax fibre composites. Water absorption test was used to examine the effect of the coating on the fibres.
This investigation covers the energy absorption of composites reinforced with flax and carbon as reinforcement and the establishment of a balance on the laminate strength and the energy dissipation depending on desired applications of choice.

RESULTS AND CONCLUSIONS
The pendulum impact tests were conducted on non-hybrid and hybrid composite samples measuring 40 mm length and 10 mm width using TECQUIPMENT' TE15 energy absorbed at fracture system and representative images samples after testing shown in Figs 1. The non-hybrid composite has flax as the

reinforcement and the hybrid ones constitute of flax and carbon fibres. Four samples of each laminate configuration were tested and the [90/±45]s laminate absorbed the highest energy (0.37 J) for the non-hybrid composite as shown Fig. 2. This is likely because of the position of the ±45 plies resisting the impact energy. The difference compared to the energy absorbed by the [0/±45]s composite is small.

Fig.1 Photographs of tested samples.

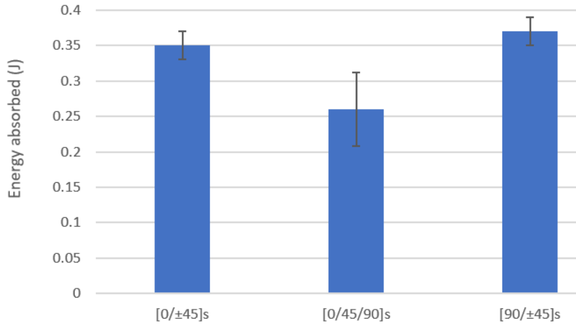

Fig.2 Energy absorbed by non-hybrid composites.

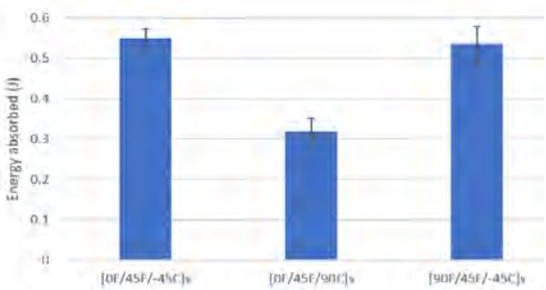

Fig.3 Energy absorbed by hybrid composites (66% flax).

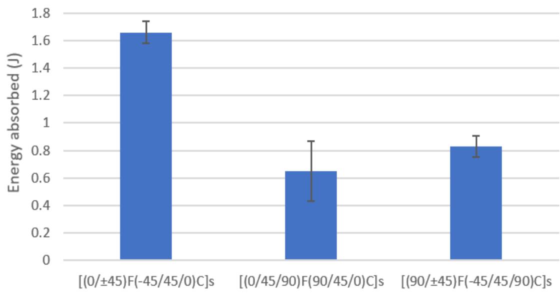

Fig.4 Energy absorbed by hybrid composites (50% flax).

Hybridization improves the damage resistant as shown by the energy absorbed values in Figs 3 and 4. The $[0_F/45_F/90_C]_S$ composite absorbed the least energy as shown in Fig. 2, this is because of the position of the 90 degree carbon fibres ply having little or no resistance to the impact load. Although the strength of natural fibres gain improvement because of the process treatments with chemicals, they are still low compared to synthetic fibres like carbon; this study is in progress and intend to provide detailed information about the improvement of flax reinforced composites by controlled hybridization with carbon fibres in the percentage between 10 – 70% of the volume.

ACKNOWLEDGMENTS

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ID 192
MULTIFUNCTIONAL FIBROUS STRUCTURES: THE ROLE OF NATURAL FIBERS AND METAL OXIDE NANOPARTICLES

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ABSTRACT
This work aims to develop multifunctional natural fibres, using metal oxide nanoparticles (NPs), for several applications, namely the degradation of chemical and biological harmful agents. Calcium oxide (CaO), magnesium oxide (MgO), titanium dioxide (TiO_2) and silica (SiO_2) NPs were synthetized via sustainable methods. Jute and flax fabrics were functionalized with these NPs by the *in-situ* method and electrospray. All the developed samples were characterized by Field Emission Scanning Electron Microscopy (FESEM), Attenuated Total Reflectance-Fourier-Transform Infrared Spectroscopy (ATR-FTIR) and Ground-State Diffuse Reflectance (GSDR). Several properties were also analysed, including harmful chemicals degradation, antibacterial activity and ultraviolet (UV) protection.

INTRODUCTION
Multifunctional fibrous systems (MFS) are a target of interest for researchers all over the world since they can be used in a wide array of areas, such as healthcare, sports, and in the protection against chemical and biological warfare (Araújo et al., 2020). The functionalization of fibrous structures with NPs, especially metal oxides, is a great strategy for the development of functional textiles, due to not only to their nanoscale, but also good structural characteristics. Metal oxide NPs can provide several properties, such as UV protection, easy/self-cleaning and especially the adsorption/decomposition of harmful agents, without adding extra weight to the structures (Araújo et al., 2020). These NPs, such as CaO, MgO and TiO_2 have been gaining special attention for degradation of harmful compounds, due to their high surface area, large number of reactive sites, high absorption capacity and decomposition ability (Araújo et al., 2021). One good strategy for the improvement of the intrinsic properties and the NPs' anchorage onto the substrate is the addition of SiO_2, due to its high surface area (Araújo et al., 2020). Natural fibers are an excellent example of a sustainable and adaptable substrate, since they are highly abundant, biodegradable and biocompatible (Costa et al., 2018). Therefore, the functionalization of natural fibers with metal oxide NPs emerges as a great alternative for the development of MFS. In this work, flax and jute fabrics were functionalized with CaO, MgO, TiO_2 and SiO_2 NPs by a simple methods (*in-situ* synthesis and eletrospray). All the samples were characterized by FESEM, ATR-FTIR and GSDR and properties like UV protection and degradation of harmful chemicals and bacteria were evaluated.

RESULTS AND CONCLUSIONS
Jute fabrics were functionalized with CaO and SiO_2 NPs using a very simple *in-situ* method, using water as solvent and minimal concentrations of sodium hydroxide (NaOH). The same method, with the addition of citric acid to the reactional mixture, allowed the functionalization of flax fabrics with MgO-SiO_2 core-shell NPs. The synthesis of TiO_2 under very simple conditions (low reaction times and calcinations temperatures)

was also performed, being that in this case the NPs were electrosprayed onto the surface of flax fabrics. The FESEM images of the jute fabrics functionalized with a) CaO and SiO_2 and flax fabrics functionalized with b) MgO-SiO_2 and TiO_2 NPs are shown in Fig.1. It's possible to observe not only the presence of the NPs, but also their homogenous distribution. Other techniques (ATR-FTIR and GSDR) also corroborated this successful synthesis (data not shown in this abstract).

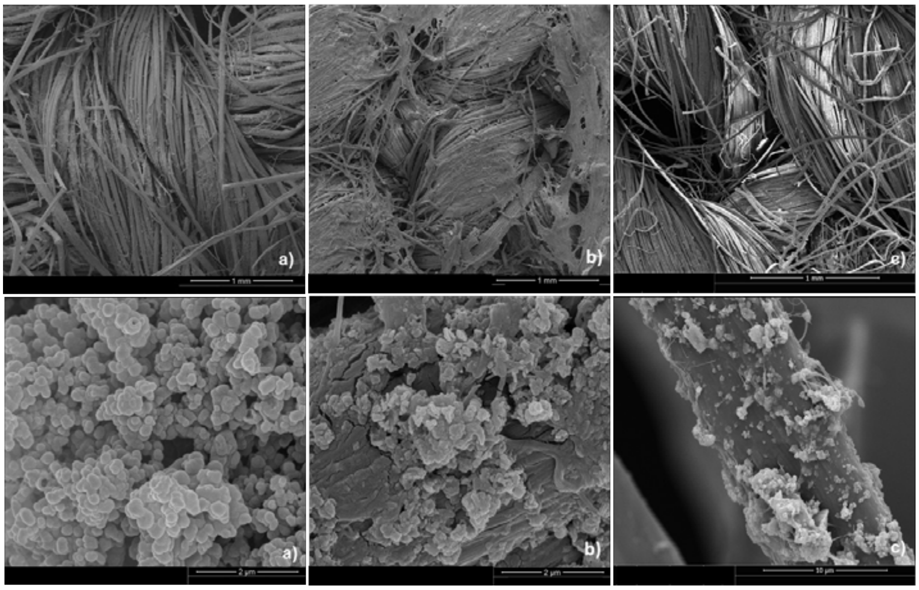

Fig.1 FESEM images of jute fabrics functionalized with a) CaO and SiO_2 NPs and flax fabrics functionalized with b) MgO-SiO2 and TiO_2 NPs.

All the samples presented strong antibacterial activity against *Escherichia coli* and *Staphylococcus aureus.* The degradation of methylene blue (a poisonous dye) and UPF values of 50+ were also achieved for the functionalized samples.

ACKNOWLEDGMENTS

The authors are thankful to project UID/CTM/00264/2023 of 2C2T – Centro de Ciência e Tecnologia Têxtil, funded by National Founds through FCT/MCTES" and FCT PhD Scholarship (SFRH/BD/147812/2019). Diana Ferreira is thankful to CEECIND/02803/2017.

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ID 193
LIGNIN-POLYMER COMPOSITE FIBERS AS PRECURSORS FOR CARBON FIBERS

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ABSTRACT
Lignin is the most abundant aromatic organic polymer of natural origin on the face of the earth. It is a residue produced in large quantities by the pulp and paper industries [1]. The chemical composition of the lignin residue varies with the extraction processes applied industrially, such as the Kraft process, the organosolv method, steam explosion, enzymatic digestion, etc [2]. For this reason, the composition of lignin obtained as industrial waste is variable, depending on the plant of origin but also on the extraction process applied [1,2]. The present work reports the characterization and stabilization of a purified Kraft lignin [3] aiming at the preparation of carbon fiber precursors.

INTRODUCTION
The aromatic nature of lignin and the high fraction of residual carbon resulting from its carbonization have attracted the interest of researchers for its application as a precursor of carbon reinforcements [2,4]. The present work reports the study of lignin recovered as waste from the paper industry, in powder form, in terms of its chemistry and thermal characteristics. The lignin was melt mixed with selected polymers to prepare mixtures with a high lignin composition (equal to or greater than 50%). Fibers were produced at the higher draw ratio possible and their viability as carbon fiber precursors (green fiber) was studied. These fibers were heat treated under oxidative atmosphere to evaluate their thermal stability and potential to withstand the carbonization process.

RESULTS AND CONCLUSIONS
Kraft lignin from softwood was supplied by West Fraser Co. Ltd., after purification by the LignoForce process. The lignin is here designated as “LA”. This lignin has a percentage of solids of 50% and ash between 0.5 and 1.5%. Its molecular weight is in the range 6 - 10 kDa with a polydispersity index of 3 - 4.6. Thermal characterization of the lignin by differential scanning calorimetry depicts an amorphous structure with a glass transition temperature near 155 ºC, thermally stable under oxidative atmosphere up to approximately 250 ºC.
Composites of LA/polymer were produced using a micro-extruder Xplore MC15 equipped with a die of 250 mm. The composite filament was drawn using an Xplore Double Winder Line and collected on a Xplore Lignin Fibre Winder. Composite filaments were produced with polycarbonate/acrylonitrile-butadiene-styrene (PC/ABS) blend and glycol-modified polyethylene terephthalate (PETG), at lignin contents of 50 and 60% in weight. The filaments were thermo-stabilized in an oven under tension, heating at 0.05 ºC/min from room temperature to 250 ºC, forming the “green fibres”.
The lignin was analysed by Raman spectroscopy before and after thermal stabilization, as well as its poly-

mer composites. The Raman spectra of thermally stabilized LA and composites is presented in Figure 1a. Figure 1b shows the results for the fibre's specific weight before and after thermal stabilization under stress to produce green fibre, showing densification of the fibres during this process.

a) b)

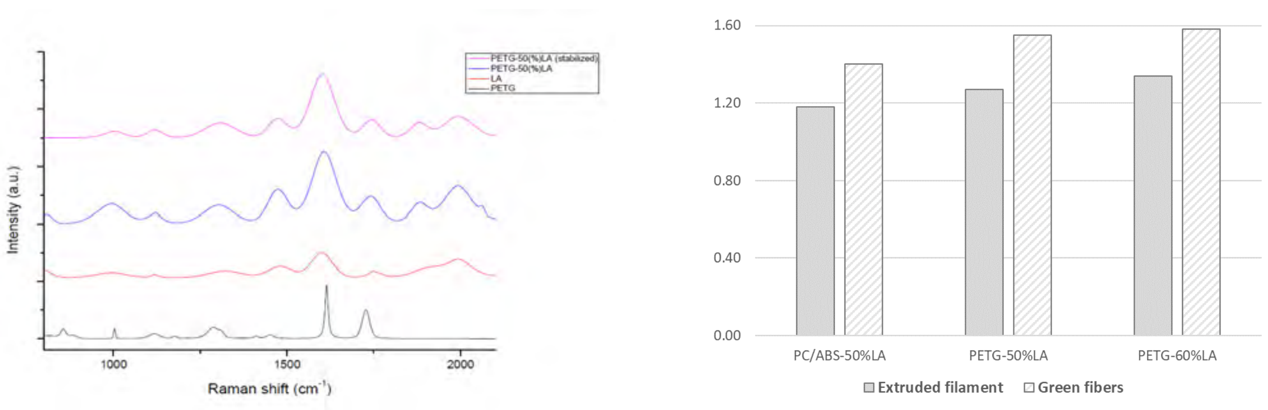

Fig.1 a) Raman spectra of thermally stabilized LA and PETG-50%LA composites; **b)** Green fibres specific weight before and after thermal stabilization

This work demonstrated the possibility of forming continuous composite fibres with high lignin content that can withstand thermal stabilization under tension and produce green fibre. The green fibre formed has good potential for further heat treatment under inert atmosphere for carbon fibre production.

ACKNOWLEDGMENTS

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ID 194
BIODEGRADABLE LOCALIZED DRUG DELIVERY SYSTEMS BASED ON ELECTROSPUN NANOFIBERS FOR CANCER PHOTODYNAMIC THERAPY (PDT)

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ABSTRACT
Core-shell nanofibers were produced by co-axial electrospinning using biodegradable polymers, poly(vinyl alcohol) (PVA) as core and gelatin (Gel) as shell, in order to develop localized drug delivery systems (DDS) for cancer photodynamic therapy (PDT). Subsequently, a porphyrin was incorporated into the core of the nanofibers. All the developed membranes were characterized by Field Emission Scanning Electron Microscopy (FESEM), Attenuated Total Reflectance-Fourier-Transform Infrared Spectroscopy (ATR-FTIR), Thermogravimetric analysis (TGA), Ground State Diffuse Reflectance (GSDR) and Confocal Laser Scanning Microscopy (CLSM). The porphyrin release profile was also assessed by UV-Vis spectroscopy.

INTRODUCTION
In recent years, PDT is attracting tremendous attention to treat a variety of diseases, including cancer and infections. PDT is a localized and minimally invasive therapy based on the generation of reactive oxygen species (ROS), which lead to cell death and tissue destruction. The production of these cytotoxic species occurs by combining the use of photoactive molecules, called photosensitizers (PSs), light, and molecular oxygen present in tissues. Porphyrins and their derivatives are the most used PSs for PDT, because they present high chemical purity, the energy of the triplet state is relatively high to promote the reaction with molecular oxygen, high quantum yield of 1O2* generation ($\Phi\Delta$), no cytotoxicity in the dark and in some cases rapid clearance from patients' body. Nevertheless, they present some drawbacks that limits its photodynamic effect, including high dose requirements, which can result in prolonged and inappropriate photosensitivity; low selectivity and solubility of the drug; the compound lost before reaching the target tissue and difficulty in reaching deep tissues. Therefore, the use of DDS is a suitable approach to overcome these obstacles. Among them, nanofibers produced by electrospinning have emerged as a promising alternative due to their great characteristics, such as high surface area associated with high drug loading capacity, possibility to use a wide range of matrix materials and to incorporate several bioactive molecules, possibility to control the drug release profile, flexibility, biocompatibility and biodegradability. In fact, the production of electrospun nanofibers using biodegradable polymers (either natural or synthetic) allows an effective drug release as well as avoids surgical removal of the implanted DDS. The development of core-shell nanofibers has been attracting great attention, not only because allows an infinite combination of polymers, but also because the PSs can be incorporated into the core of the fibers, which will be covered by the shell, thereby offering the possibility to prolong the drug release and action time as well as to protect the drug from damage (Costa, 2022 & Ferreira, 2016). Hence, the aim of this work was the development

of electrospun core-shell nanofibers using biodegradable polymers. Afterwards, a synthesized porphyrin was incorporated into the core of the fibers at different concentrations. The developed membranes were characterized by FESEM, ATR-FTIR, TGA, GSDR, CLSM. The drug release profile was also monitored.

RESULTS AND CONCLUSIONS

Defect-free PVA-Gel nanofibers (average diameters of 170 nm) were successfully developed using the following electrospinning parameters: 29 kV, 250 mm, 0.8 mL/h (core) and 0.2 mL/h (shell) (Figure 1a). ATR-FTIR and TGA also confirmed the presence of the two polymers. The incorporation of porphyrin also allowed the production of uniform nanofibers with increased average diameters (204 nm). The presence of the porphyrin onto the membranes were confirmed by GSDR, by the appearance of the compound's characteristic absorption bands. The distribution of the porphyrin within the fibers was observed by CLSM, where it was possible to verify red fluorescence when excited at 405 nm (Figure 1b), which would be expected since these molecules emit fluorescence in the red spectral region upon excitation in the Soret-band (Lourenço, 2014).

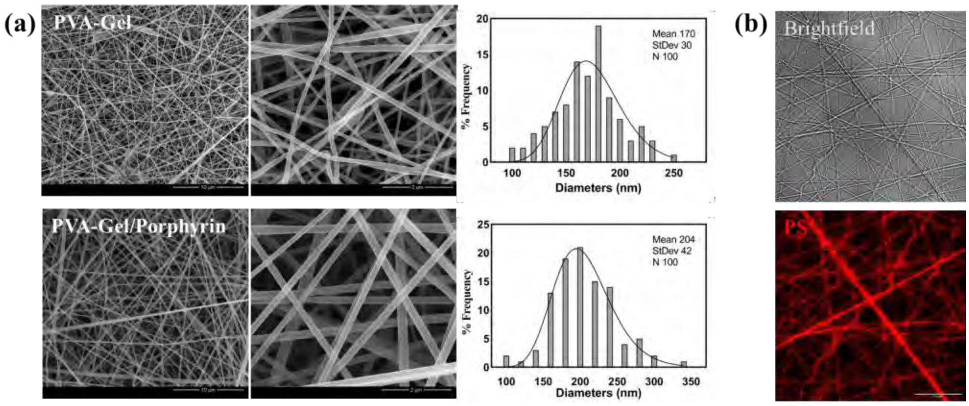

Fig.1 (a) FESEM images and the respective diameter distribution histogram of PVA-Gel and PVA-Gel/Porphyrin core-shell nanofibers; **(b)** CLSM images of PVA-Gel/Porphyrin nanofibers excited at 405 nm and detected at 500-699 nm.

Porphyrinwas continuously released from membranes over time. The cytotoxicity of these membranes in the dark and under irradiation will be evaluated using HeLa cells.

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The authors are thankful to project UID/CTM/00264/2022 of 2C2T - Centro de Ciência e Tecnologia Têxtil, funded by National Founds through FCT/MCTES. S. M. C. is thankful to FCT PhD Scholarship (SFRH/BD/147517/2019). D. P. F. is thankful to CEECIND/02803/2017.

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ID 195
SUSTAINABLE CELL SOLUTION® BIOACTIVE FIBERS WITH INHERENT ANTIBACTERIAL AND ANTIVIRAL FEATURES

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ABSTRACT
Cellulosic fibers are modified towards the incorporation of bioactive features. Lyocell fibers are modified with an ion exchange resin and subsequently loaded with copper ions and fixed. Fixation by carbonate increases considerably the washing resistance. Disperse, reactive and HT dyeing maintain the copper content up to 50%. In nonwoven blends with a share of only 6% copper fiber a strong antimicrobial (KBE > log 5) and full antiviral effectiveness (> log 3) was received even after 50 washing cycles. Further, the antiviral behavior is active already after 30 sec.

INTRODUCTION
Cellulose exhibits the most abundant biopolymer with unique properties applied not only for textile fibers but also for versatile materials. In the sense of demanded sustainability and green chemistry it receives more and more attraction with respect to overcome fuel-based polymers. Using the circular Lyocell process combined with raw materials of natural origin (wood, cotton, hemp) and/or usage of end-of-life textiles, the expectations of more and more ecologically convinced consumers are to be met. Already in ancient times copper was used to sterilize chest wounds and drinking water. In opposite to silver it is an essential trace element and, therefore, harmless for human beings. Further, silver does not work as an antimicrobial surface when dry, meaning moisture needs to be present. Copper surfaces even in communal areas help reduce the transmission of respiratory viruses like covid-19 (Warnes, 2015).
Lyocell fibers were produced with different ion exchange resins with varying concentrations in the range of 5 – 15%. Mechanical properties like tenacity were measured and the copper ion uptake capacity was determined. Fixation procedure was optimized, especially under valence change of copper to the formation of copper (I) oxide (Wendler, 2022). Fibers were dyed using disperse, reactive and HT procedures with subsequently washing (50 cycles) and determination of copper content. Antimicrobial and antiviral effectiveness were measured. Antiviral behavior was performed in dependence of time.

RESULTS AND CONCLUSIONS
Fleeces of Tencel and bioactive fibers equipped with copper were produced and analyzed on their bioactive behavior. Fig. 1 displays the antibacterial effectivity against bacteria strain Staphylococcus A. The fiber shows a strong antibacterial effect even after 50 washing cycles. Although the share of fiber in the fleeces amounts to only 6%, a strong antibacterial effect was proven even 50 washing cycles.
Further, antiviral investigations were performed on these fleeces as shown in Fig. 2. Antiviral activity was also measured in dependence on time. Even after 30 sec a complete antiviral effectivity could be proved. Reference bioactive materials were used for comparison.

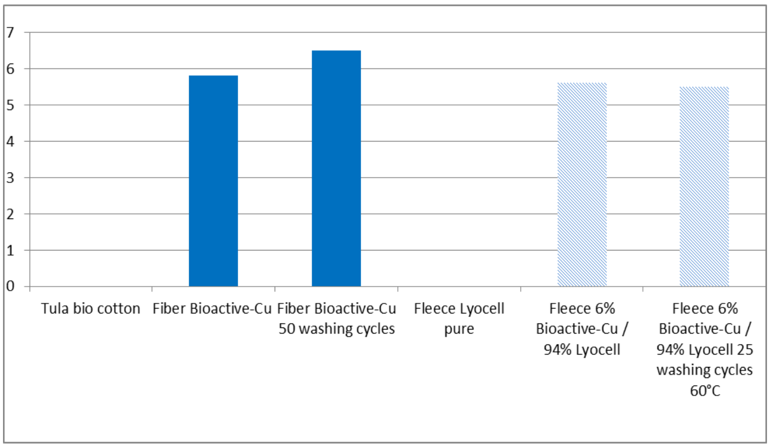

Fig. 1 Antibacterial effectivity (log KBE) against Staphylococcus A.

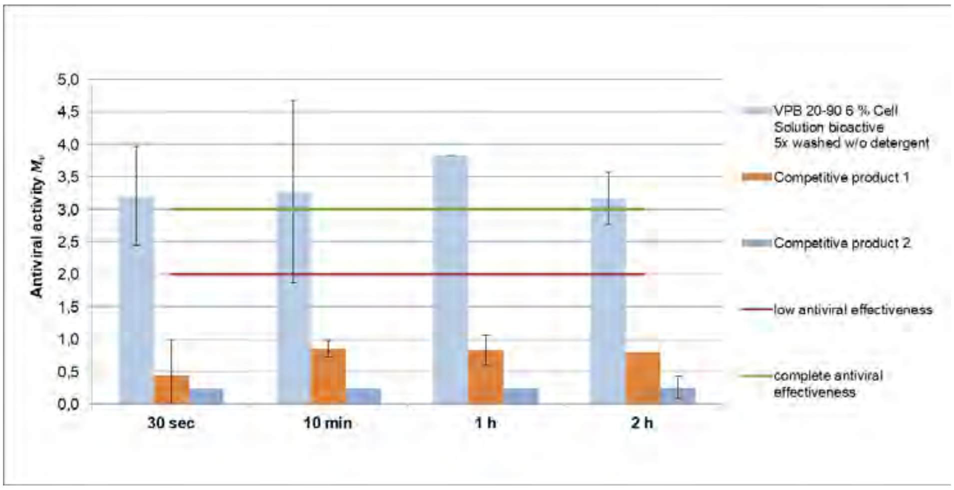

Fig. 2 Antiviral activity acc. to ISO 18184.

The study shows that Lyocell fibers can be modified with low amount of additives like ion exchange resins. Further equipped with copper, these bioactive fibers are ready for applications in hospital textiles sector, home textiles or face masks.

ACKNOWLEDGMENTS

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ID 198
INVESTIGATION OF DAMPING PROPERTIES AND LOW VELOCITY IMPACT RESPONSE OF FLAX FIBRE HYBRID COMPOSITES

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ABSTRACT
The vibration damping behaviour and low velocity impact response of flax/carbon and flax/glass fibre reinforced epoxy hybrid composites were investigated in this study. To determine the damping loss factor, cantilever beam test specimens were subjected to a signal sweep over the first three resonance frequencies. An impact energy of 45 J was used to compare the impact response of the three different composites. Three-point bending tests were also performed using CFCFCFC and GFGFGFG composite layups, where C, G and F represent ±45 Carbon, glass and flax plies, respectively. Fully flax composite specimens were used as the reference material to investigate the effect of hybridisation. The results of this study suggest that hybrid composites are more suitable for applications requiring higher damping, impact resistance and bending strength compared to composites with single reinforcement material, without hybridisation.

INTRODUCTION
The challenges associated with recycling synthetic fibers and the escalating global waste predicament have stimulated the exploration of natural fibers as viable, sustainable, and environmentally friendly alternatives. These natural fibers are derived from renewable raw materials that offer cost-effective solutions (Dhakal, 2013). Flax fibers (FF) represent a prominent category of natural fibers utilized in composite applications. To exploit the advantages offered by different types of reinforcements, hybrid composites, which incorporate multiple reinforcing materials, have been employed. This approach enables a comptonisation between various factors such as cost, strength, stiffness, and other pertinent properties (Jefferson et al., 2019).

Understanding impact toughness and vibration damping behaviors of natural fibre reinforced composites is important for these materials to be used in semi-structural and structural applications (Assarar et al. 2015, Barouni and Dhakal, 2019). This study presents the effect of hybridisation on the low-velocity impact, vibration damping and flexural properties of three different composites namely Flax (F), Flax/Glass (FG) and Flax/Carbon (FC). The lay-ups for these materials are shown in Table 1.

Table 1 Lay-up for each material

Material	Symbol	Lay-up
Flax	F	[FFFFFFF]
Flax/Glass	FG	[GFGFGFG]
Flax/Carbon	FC	[CFCFCFC]

RESULTS AND CONCLUSIONS

The energy-time results from the low velocity impact tests are shown in Fig. 1. The low velocity impact test results were assessed using energy absorption analysis, which showed that the fully flax sample had the greatest damage and absorbed 97 % of the impact energy, while the hybrid flax/glass and flax/carbon absorbed 79% and 73%, respectively, with less severe damage.

Results from vibration damping tests revealed that the fully flax composite showed the highest loss factor of 1.7 %, while the hybrid flax/glass and hybrid flax/carbon had values of 1.64 and 1.31 %, respectively.

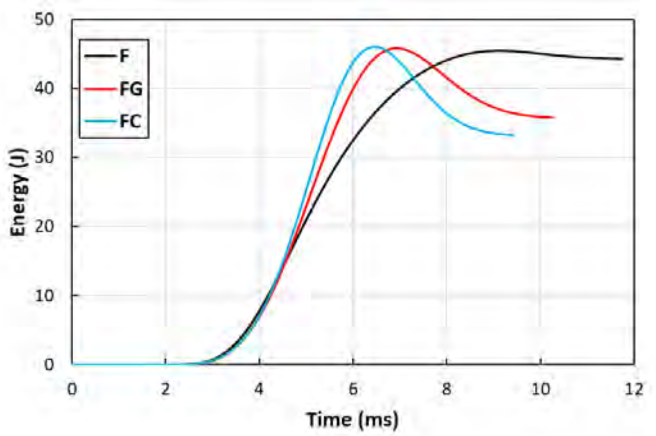

Fig.1 Low velocity impact test results

Lastly, the bending strength of the flax/carbon hybrid composite was found to be 10% higher than that of the fully flax composite, while the flax/glass hybrid composite had a 45% higher bending strength.

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ID 199
DEGRADATION OF DYED COTTON FABRIC IN LOAM SOIL

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ABSTRACT
The motivation behind this work is the colossal increase in textile waste ending in landfills. The study aims to investigate the fate of various classes of dyes on post-consumer cotton fabric waste that ends in landfills. For the initial understanding, bleached and reactive blue 19 (RB19) dyed cotton fabrics were degraded in loam soil. Carbon dioxide emissions, degraded cotton fabric, and soil after degradation were characterised. These tests help understand the degradation process of dyed cotton textiles landfilled.

INTRODUCTION
The development of fast fashion, which stands for affordable and often low-quality textiles, has directly led to the production of large volumes of post-consumer solid textile waste (PCTW). According to data from the Environment Protection Authority (DeVoy, Congiusta et al. 2021), more than 66% of PCTW were sent to landfill sites, only 15% of them were recycled, and the rest were combusted for energy recovery in the U.S in 2018. In addition, chemicals often applied to textiles during or after the production to impart either aesthetic or functional properties, such as dyes. These chemicals raise environmental concerns when substrate cotton fabric decomposes in the landfilling site. As landfilling is the most employed disposal means of textile waste, it is crucial to understand better how dyes influence the degradation process of textiles and what degradation products might leach out into the surrounding environment.
Raw plain cotton fabric was first scoured and bleached to remove impurities, then dyed with RB19 in the laboratory. Afterwards, the dyed cotton fabrics were cut into 5 cm x 5 cm. Five experimental replicates were performed. The degradation experiment was undertaken by the ASTM D5988-18 standard test method for determining aerobic biodegradation of plastic materials in soil (Sui, Feng et al. 2020). Photo images and SEM characterised fabric morphological analysis. The chemical change of the surface of the retrieved fabric was analysed using FTIR. Soil samples were analysed via LCMS using the QuEChERS method (Feng, Sui et al. 2021). The amount of CO_2 produced during the decomposing process was monitored.

RESULTS AND CONCLUSIONS
The fabric before and after degradation experiments are shown in Fig. 1. RB19 dyed cotton fabric has been visually damaged, and most of the colour faded out. Several areas of the degraded cotton fabric appear much thinner than that of the pristine RB19 dyed cotton fabric under the action of microorganisms in the loam soil.
Cotton is composed mainly of cellulose, which is nutrient source for the microorganisms existing in the soil. As expected, the faded areas on the degraded cotton fabric showed much more severe damage than that of colour retained areas, as shown in Fig. 2.

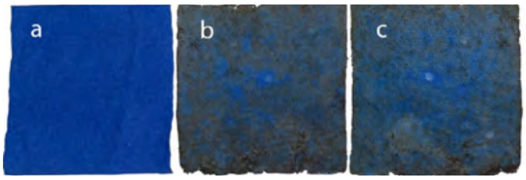

Fig. 1 RB19 dyed cotton fabric: **(a)** pristine RB 19 dyed cotton fabric; **(b)** the fabric after degradation-face up; **(c)** the fabric after degradation-face down

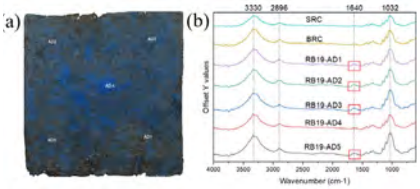

Fig. 3 **(a)** The FTIR data sampling positions on degraded RB19 dyed cotton fabric. **(b)** FTIR results

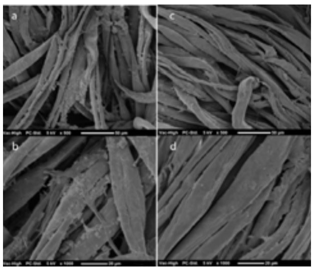

Fig. 2 SEM of #1 RB19 dyed cotton fabric after eight weeks degradation. **(a)** severe degraded aera where the colour visually faded at x500 magnification; **(b)** severe degraded aera where the colour visually faded at x1000 magnification; **(c)** relatively intact aera at x500 magnification;**(d)** relatively intact area at x1000 magnification

The mass of the fabric before and after degradation was used to calculate the degradation rate. After eight weeks of soil degradation, the RB19 dyed cotton fabric mass decreased by more than 35%.

The FTIR results presented in Fig. 3. It was noted that after degradation (AD) in eight weeks, a new band at 1640 cm^{-1} was observed, and similar results were obtained by Milosevic et al. (Milošević, Krkobabić et al. 2017), which is attributed to amide I of proteins originated from grown microorganisms.

The amount of CO_2 emitted gradually increased in the first three weeks, then relatively steadily emitted until retrieved for fabric analysis. In addition, soil sample analysis detected dye degradation products such as the removal of vinyl sulphone group and hydrolysed dye. The initial study shows that the method is feasible to examine dyed cotton textiles' degradation products systematically.

ACKNOWLEDGMENTS

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ID 202
DEVELOPMENT OF AN INNOVATIVE WATER RETAINER MADE OF BANANA PSEUDOSTEM PULP

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ABSTRACT
This work presents the development of an innovative water retainer prototype for reforestation, mainly made of banana pseudostem pulp obtained as a subproduct of banana fibre extraction. Apart from this main component, other natural materials such as starch and pine resin were tested as binder and waterproofing agents, respectively. Two different production methodologies were tested: wet moulding (pulp, starch, and water) with pressure, and dry flat moulding (pulp and pine resin) with pressure and high temperature. The results suggest that wet moulding facilitates the procedure, but cracks appear after drying. However, with dry flat moulding the process is more complex but the retainer is more compact. Regarding the waterproof agents, the solution of pine resin with turpentine works better than with alcohol because the latter absorbs moisture over time.

INTRODUCTION
In areas with low rainfall or unavailability of irrigation systems, the use of water retainers is commonly used for agriculture or reforestation. These retainers consist of tanks, usually ring-shaped, which are buried with the seedling in the middle and which provide the water necessary for the first months of life of the plant. There are three main commercial water retainers: the “Groasis Waterboxx®”, “Groasis Growboxx®” (both from Groasis B.V.) and “Cocoon” models (Land Life Company). The main differences are that the “Groasis Waterboxx®” are made of polypropylene, while the other two are made of recycled organic paper (biodegradable). In terms of design, both Groasis retainers are designed to collect rainwater and have a capacity of 15 and 16 L, respectively. The difference is that the first transfers the water to the plant by capillarity with a nylon wick, while the second transfers it through the capillary bottom [1]. Finally, the “Cocoon” also transfers water through the bottom of the retainer, has a simpler design, more capacity (25L), but it is not prepared to collect rainwater, it is only filled at the beginning of the use. In more arid areas, such as the Canary Islands, this is an advantage as a larger capacity is more efficient than rainwater collection and makes more feasible the manual refilling of the retainer. In addition, there are cellulose wastes that could present a similar behaviour to the recycled paper material used in these commercial biodegradable retainers. One example is the pulp waste resulting from the banana fibre extraction process. Banana is the most important crop of the Canary Islands and during cultivation, each pseudostem bears fruit only once, so when it is harvested, the pseudostems are cut and left in the plantation to decompose, generating a large amount of waste. However, there is an option for their revaluation which is the mechanical extraction of the fibre [2]. This process generates two different raw material, a high quality natural fibre and a residual pulp whose main component is cellulose [3]. Therefore, the use of this pulp for the manufacture of the retainer would provide value to a material that is currently considered a waste and this retainer would help in the agriculture and reforestation of an arid area such as the Canary Islands.

For the manufacture of a prototype retainer, two different processes were studied. The first process was based on wet moulding of the retainer with banana pulp of approximately 10 mm thickness and a cold solution of starch and water. This mixture produced a paste that was easy to mould and to adapt to the geometry of the mould. The second process was based on dry flat moulding with pressure and high temperature. In this case the mixture was made with approximately 10 mm thickness of mixture of pulp and 30% w/w pine resin powder uniformly stirred into the pulp, and after compression moulding, plates were obtained which were then joined together to form the retainer. The process and result of both methods are shown in Figure 1.

Then, two methods were used for waterproofing, a 2:1 solution of pine resin powder and alcohol, and a 3:2 solution of pine resin powder in turpentine. In both cases, two layers of resin were painted all over the model, giving better results the mixture made with turpentine because the alcohol solution picks up moisture over time.

Figure 1: Result of the retainers. A) Wet moulding B) Flat moulding

RESULTS AND CONCLUSIONS

The results obtained from both processes were different. In the wet forming process, the moulding process is simpler and faster. However, the drying time may be long, and cracks appear due to the contraction of the pulp with water. On the other hand, in the hot forming process, the manufacturing is more complex as only flat surfaces can be moulded, which implies a subsequent bonding between plates to form the retainer. Additionally, the plates are very stiff, which makes the handling more difficult.

Finally, in both cases, in the process of waterproofing it was observed that any small surface that is not perfectly sealed with the solution can give rise to a leakage area, but these areas could be used as a method of irrigation.

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ID 203
EXTRACTION AND USE OF RACHIS FIBRE AS REINFORCEMENT OF POLYLACTIC ACID COMPOSITES

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ABSTRACT
This work assesses a first approach to the extraction and processing of banana rachis fibre for its recovery as reinforcement in polylactic compounds. Several extraction techniques (mainly based on the retting process) and post-processes (pressing, washing, scraping, and combing) were used to improve the quality of the fibre. Finally, alkaline treatments were carried out to improve the matrix-fibre compatibility. Different rachis fibre-PLA composites were mechanically tested (tensile, flexural and impact tests) and thermally characterised (thermogravimetry analysis and differential scanning calorimetry). The first results showed that the rachis fibre could improve some mechanical parameters of the polymeric matrix, but further studies are needed.

INTRODUCTION
Considering the serious problem of the large production of single-use plastic packaging and the management of this waste, the composite industry have been forced to look for alternative materials, such as the use of biodegradable materials, biobased polymers, or natural fibres as reinforcement, in order to reduce the plastic weight. On the one hand, of the bioplastics available today, in this work it is used polylactic acid (PLA), which has received considerable attention due to its potential applications [1,2]. On the other hand, banana is the most important crop of the Canary Islands [3] and, at the end of the cultivation cycle, it produces three main by-products: leaves, pseudostems and rachis. Leaves and pseudostem wastes are usually left to decompose in the banana plantations, while rachises are shredded and discharged by banana handling centers. The pseudostem forms the main part of the plant and its fiber content and length have made it the most investigated part. However, the rachis also has fibres, being a novel element to characterize and valorise, but there are still some challenges that need to be addressed, such as the development of an efficient fibre extraction methodology.
For fibre extraction, different techniques have been analysed. On the one hand, retting has been carried out in water under different conditions with unpeeled and peeled rachis, changing water, and with double retting. On the other hand, different post-processing methods have also been studied, such as pressing, washing, scraping and combing. In addition, to improve the compatibility of the fibre with the polymeric matrix, the fibre was subjected to a chemical treatment with a 1N sodium hydroxide solution for 1 h at room temperature. Once the fibre was obtained, it was used as reinforcement of the PLA (Inzea F29 H10 from Nurel S.A.), both in short and long fibre format. For the short fibre composites, the fibre was cut to a length of about 2-3 mm, followed by sieving, twin-screw extrusion and finally compression moulding. These composites were made with fiber content at 10 and 20% w/w, with alkali-treated (T) and untreated fibre (UT). In the case of long fibre, the composites were manufactured by compression moulding with bidirectional alkali-treated long fibre (BID-T) and untreated fibre (BID-UT) at 16% w/w fiber content. Once the plates

were obtained, the specimens for characterisation were cut by laser cutting. Finally, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), optical microscopy and mechanical tests (tensile, flexural and impact) were carried out.

RESULTS AND CONCLUSIONS

According to the experimental observations, the best fibre extraction method was the retting of the peeled rachis in unchanged water, followed by washing combined with light scraping.

The DSC analysis revealed that the fibre decreases the melting temperature for all the samples. The TGA analysis showed that the degradation temperature increases with the combing of the fibre because it removes the pulp residues. Additionally, the treatment further increases the thermal stability.

The results of the mechanical tests are shown in Table 1. Firstly, the elastic modulus and maximum tensile strength decrease with the fibre content in the short fibre composites. In the bidirectional composites, the elastic modulus increase compared to the unreinforced PLA, and the tensile strength increase only with treated fibre. Regarding the flexural elastic modulus, all the composites showed lower values than neat PLA, except the bidirectional composites with treated fibre, which increased the modulus. A similar behaviour was observed in terms of impact strength, although in this case, none of the composites was able to maintain the impact strength of neat PLA. Finally, the chemical treatment improves the mechanical properties in all the samples, which leads to the conclusion that it improved the fibre-matrix adhesion.

Table 1 Mechanical test results

Sample	Fibre (%w/w)	Tensile elastic modulus (MPa)	Max tensile strength (MPa)	Flexural elastic modulus (MPa)	Impact strength (kJ/m^2)
PLA	0.00	1121.01 ± 44.02	26.19 ± 1.36	4032.73 ± 55.09	9.52 ± 3.71
PLA+10%RAQ-UT	10.00	1027.05 ± 74.74	19.27 ± 1.08	3485.04 ± 157.86	3.67 ± 0.52
PLA+10%RAQ-T	10.00	1049.70 ± 21.18	21.82 ± 1.82	3582.68 ± 164.30	3.70 ± 0.27
PLA+20%RAQ-UT	20.00	946.29 ± 71.40	16.44 ± 2.24	3726.48 ± 184.29	2.79 ± 0.61
PLA+20%RAQ-T	20.00	990.13 ± 65.96	17.48 ± 2.17	3775.90 ± 176.00	3.24 ± 0.36
PLA+BID-UT	16.31	1317.72 ± 84.89	21.93 ± 2.12	3907.78 ± 268.617	4.00 ± 0.40
PLA+BID-T	15.95	1365.81 ± 82.45	34.20 ± 2.23	4202.17 ± 419.71	7.94 ± 1.27

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ID 204
TOWARDS THE PREDICTION OF MECHANICAL PROPERTIES OF FLAX BUNDLES BY INFRARED AND COLORIMETRIC ANALYSIS

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ABSTRACT
In this study four batches were hand-sorted among scutched flax fibers from the same harvest based on visual color and touch. This empirical separation, characterized by colorimetric and morphological analyses, highlights several types of heterogeneity possibly due to the different impacts of retting. The tensile mechanical properties of the bundles were measured and their surface compositions were determined by infrared spectroscopy. We showed that a combination of infrared and colorimetric analysis allows a classification of the fibers according to their different mechanical properties, in relation to their intra-bundle cohesion.

INTRODUCTION
Flax fibers are natural resources used for many applications, such as textiles and materials, notably thanks to their mechanical properties. The process of their extraction includes a biological pretreatment of the plant stem in the field, the retting, followed by a mechanical treatment, the scutching, to remove the woody parts from fibers. Combined with the initial genetic, agronomical, and environmental factors, this complex process contributes to the variability of the fibers, and thus to their different physico-chemical quality (Baley, 2020).
This variability is therefore the subject of many studies looking for quality indicators, some of which attempt to predict the mechanical behaviors of natural fibers using spectral analyses that are quick to perform (Rafidison, 2020). Here, we investigated the possible relationship between the surface composition of flax fiber bundles (spectral signature) and their mechanical properties. The four batches selected manually based on sight and touch were first characterized both by colorimetry (CIELab) and morphology. The cross-section of fiber bundles was determined using automated laser scanning (Garat, 2018). Then, after spectroscopic characterization by ATR-FTIR in Mid-Infrared, tensile tests were performed on 30 mm long bundles in a controlled environment (20 °C and 55% humidity) at a displacement rate of 3 mm.min-1. Two regions are used for the determination of the traction modulus: at the bottom of the curve (0.15-0.35% elongation) and at the final linear zone before the break.

RESULTS AND CONCLUSIONS
Colorimetric and morphological measurements confirmed the heterogeneity of the empirically separated fiber bundles, based on color (Light (L) or Dark (D)) and fineness (Fine, Medium, or Thick). A correlation

between these two parameters is highlighted, except for the D/Thick batch whose color comes from remnants of retting. D/Thick, with fineness equivalent to that of the L/Thick batch, has mechanical properties close to the L/Medium one (Table 1).

The slope ratios of the two regions (afinal zone over a0.15-0.35%) are close to 1 for L/Thick and L/Fine samples and close to 1.5 for L/Medium and D/Thick ones (Fig. 1). Higher value for L/Medium and D/Thick is due to a lower tensile modulus at low elongation (E0.15-0.35%). For L/Fine the slope ratio of 1 is caused by a lower tensile modulus at high elongation too (Efinal zone), with an additional much lower value of strength (σmax) and strain (εmax) at break (Table 1). Both observations could be attributable to the weakening of the intra-bundle cohesion on top of the fineness variation. These differences in mechanical behavior could be predicted by an infrared indicator based on the intensity ratio of the bands at 1427 and 896 cm-1.

Table 1 Colorimetric, morphological, mechanical and spectroscopic data of the four batches of flax fiber bundles (Values with different letters within a column are significantly different, p ≤ 0.05)

	L*	b*	Diameter(μm)	$E_{0.15\text{-}0.35\%}$(G-Pa)	$E_{finalzone}$(G-Pa)	σ_{max}(MPa)	ε_{max}(%)	$A_{1427/896}$
L/Fine	62,70^{a}	15,45^{a}	78,8^{a}	16,5^{a}	18,8^{a}	278,6^{a}	1,60^{a}	0,88^{a}
L/Medium	61,55^{b}	21,44^{b}	101,1^{b}	16,0^{a}	25,1^{b}	530,6^{b}	2,44^{b}	1,08^{b}
L/Thick	55,01^{c}	24,62^{c}	115,9^{c}	22,5^{b}	25,3^{b}	601,6^{b}	2,45^{b}	1,38^{c}
D/Thick	51,97^{d}	18,03^{d}	118,5^{c}	15,9^{a}	25,8^{b}	545,9^{b}	2,30^{b}	1,11^{b}

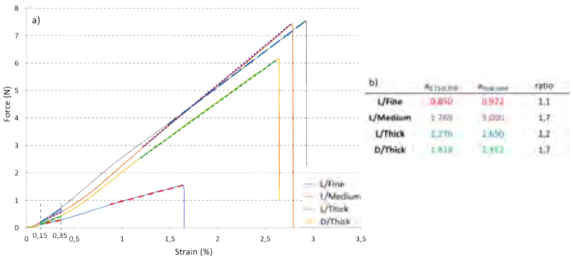

Fig.1 a) Force-strain curves of the four batches of flax fiber bundles **b)** Values of the curve slopes between the respective strain ranges and of ratios

Our findings underline the potential of infrared spectroscopy to discriminate between samples of interest and predict to some extent a favorable mechanical behavior. Furthermore, a simple color analysis would allow to separate some samples from others, enabling an upstream classification of their fineness. Finally, the combination of the two methods could be used as a tool to predict the use of fibers in the adequate value chain, regarding their tensile properties.

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ID 205
FLAX365 - DEMONSTRATION PROJECT FOR AN INTEGRATED REGIONAL PROCESS CHAIN FOR HIGH-QUALITY NATURAL FIBER PRODUCTS IN BADEN-WÜRTTEMBERG

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ABSTRACT
This project aims to create an industrial network concept for the production of high-quality products from the domestic fiber plants flax and hemp, by means of a vertical production line from cultivation to the industrial end product.
In addition, a regional competitive flax cultivation is to be revived. This also by substituting the traditionally necessary flax retting, as well as by novel textile products, preferably based on oil flax, to allow a cascade use.
The project includes the development of a production chain for textile fibers and flax products, respectively, with special consideration of the interests of the industry in Baden-Württemberg. The industrial processing and production of textile pre- and end-products from flax should stimulate the market introduction and increase the acceptance in the industry of Baden-Württemberg. With regard to the special conditions and current technological possibilities, applications in higher-quality textile products are to be demonstrated in the short term as alternatives to the use of synthetic or imported fibers in technical areas.

INTRODUCTION
To stimulate the market introduction of flax fibers, especially short fibers, in higher-value areas, it is necessary that suitable fiber quantities are made available to users (textile industry, nonwovens and composites industry) in sufficient quantities. From the findings of innovative processes such as chemical-free steam explosion processing for cellulosic agricultural products, a modified process procedure for fiber plants can be derived. For this purpose, it is necessary to narrow down the raw material selection accordingly and to adapt the pre- and post-treatment procedures according to industrial requirements and infrastructures. In principle, the modified process is intended to replace the time-consuming field retting process, which is sensitive to climatic influences. Compared with purely mechanically extraction fiber materials, however, modified extraction already brings a considerable technological improvement in terms of purity, fineness and processing properties as well as application-related fiber design, thus increasing acceptance among "processing newcomers".
In this project, the entire process chain from agriculture through fiber preparation/refining to the marketable product is to be investigated and demonstrated in the form of a practice-based, semi-industrial feasibility study. Through close cooperation of raw material producers, fiber processors, industrial users and research, the industrial suitability of domestic fiber raw materials and their marketability as end products of

high added value will be demonstrated.

By establishing a new process concept as well as the production of innovative and high-quality materials from domestic renewable fiber raw materials, new perspectives for the domestic industry can also be created. Both as a production alternative for agriculture and as new fiber raw materials and substitutes for imported and synthetic fibers for the processing industry, domestic bast fibers can finally live up to their overall economic importance. The clearly increasing trend towards a sustainable bioeconomy and the related demand for high-quality and industry-ready natural fibers creates an immediate market for the targeted production volumes.

In the long term, the resulting new possibilities in the material and textile sectors would create completely new markets and areas of application. The domestic industry would then benefit from the created raw material potential, which would lead to new diversification possibilities of textile and technical product ranges and thus trigger decisive impulses for sustainable competitiveness.

RESULTS AND CONCLUSIONS

By using the Steam Explosion process, we get fine and flawless spinnable fibers with a staple length between the length of cotton fibers and wool fibers.

As blended yarns, e.g. with viscose or lyocell, these bast fibers can be used for textile applications. Another field of application is the production of dry nonwovens for various applications.

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ID 206
ENHANCING MOSQUITO REPELLENCY IN TEXTILES THROUGH OPTIMIZED NATURAL ESSENTIAL OIL FORMULATION

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ABSTRACT
An optimized formulation combining Mentha piperita and citronella essential oils showed excellent mosquito repellency in textiles. The concentration and method of application were crucial factors. Essential oils offer a safe and sustainable alternative to synthetic repellents in textile products. This formulation has the potential to decrease the spread of mosquito-borne diseases. The study highlights the possibility of creating environmentally friendly and effective mosquito repellent textiles. Further research may refine the use of essential oils in textile products.

INTRODUCTION
Mosquitoes and the diseases they transmit have a significant impact on global health and well-being. Repellent textiles have the potential to make a significant contribution to the global effort to control and prevent mosquito-borne diseases, and their continued development and use is an important part of a comprehensive approach to addressing this public health challenge. (Sajib et al., 2020)
Essential oils have been found to have potential as natural mosquito repellents. These oils contain compounds that have been shown to have repellent properties against mosquitoes, and their effectiveness varies depending on the concentration of the active ingredients, the duration of exposure, and the species of mosquito. (Sutthanont et al., 2022)
Several strategies have been proposed to improve the repellent effect of essential oils on textiles. Among others, the blending of essential oils with other natural or synthetic ingredients can help to improve their efficacy and longevity when applied to textiles. However, the major challenges associated with the application of essential oils to textiles as a means of insect repellency are longevity, because essential oils tend to evaporate quickly and lose their efficacy as a result and stability as they can be chemically unstable and can break down over time, reducing their effectiveness. It is important to note that the development of these strategies is ongoing, and more research is needed to determine the most effective and practical methods for improving the repellent effect of essential oils on textiles.

RESULTS AND CONCLUSIONS
Different essential oil based formulations are developed and applied to 100% cotton fabrics. Mixtures of eucalyptus (E), mint (M) peppermint (P), tangerine (T) and free (C) and encapsulated (EC) indeterminate oils and citronella in biodegradable nanoparticles were performed. Five different blenders were optimized and used to functionalize the fabrics. A self-crosslinking polyether polyurethane was used to improve fastness in all samples. The efficacy mosquito repellence was evaluate using cage box test (Table 1). Briefly,

the test involves placing mosquitoes into a small cage that contains the test material and observing their behaviour as illustrated in Fig. 1. The number of mosquitoes that land on or bite the test subject is recorded and used to calculate a repellence score as described by Khanna e Chakraborty (2018).

Table 1 Mosquitoes repellency results

Samples (Number)	Mix of Essential oils in Finishing formulation (%)	Bitting (Number)	Repellency (%)	Bitting (%)	Mortality (%)
1	EC (8)	120	40	60	1,5
2	EC, P, C (6:1:1)	14	93	7	80
3	EC, E, T (6:1:1)	51	74.5	25.5	1
4	EC, P (5:3)	19	90.5	9.5	1
5	EC,T, E, C (5:1:1:1)	195	2.5	97.5	2.5

Fig.1 – Repellency cage test of sample 4 (after 9 min exposure)

The most significant results were obtained with a blend composed by Mentha piperita and citronella (free and encapsulated).

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ID 207
PINEAPPLE LEAF FIBRES FROM AZORES PLANTS: EFFECT OF THE DIFFERENT EXTRACTION PROCESSES AND TREATMENTS ON THEIR MECHANICAL PROPERTIES

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ABSTRACT
This work compares the physical and mechanical properties of pineapple leaf fibre (PALF), extracted from plants of different ages (11 and 18 months), between different regions of the same fibre and also from different extraction methods. Extraction methods were hand scratching, natural retting at different temperatures (25 and 35 ºC) as well as alternative methods (soil covered leaves and leaves boiled in water). PALF were manually extracted from the leaves of *Ananas comosus* of Cayenne variety, harvested in the Azores Islands. The fresh leaves were weighted and measured in length and width. PALF was extracted by hand scratching method and characterized by Attenuated Total Reflectance-Fourier-Transform Infrared Spectroscopy (ATR-FTIR), Optical Microscopy (OM), X-ray diffraction (DRX), Field Emission Scanning Electron Microscopy (FESEM) and Thermogravimetric Analysis (TGA). The mechanical properties were also evaluated as well as linear density.

INTRODUCTION
Mainly composed of cellulose, natural fibres (NF) can replace partially or totally the conventional synthetic fibres (Araujo et al., 2021). Besides the traditional cultures (i.e., cotton), NF can also be extracted from wastes like hemp stems, banana tree, or pineapple leaves in order to improvement the circular economy and avoid competition with food production. Pineapple plants, one of the most cultivated tropical fruits around the world can provide high-quality and competitive fibres (pineapple leaf fibers – PALF) for the textile industry. PALF presents low cost, low density, and light weight and are renewable and biodegradable. Their properties vary with plant age, plant variety, or extraction method (Sarah et al., 2018; Jose et al., 2016). Therefore, these characteristics are very important to not only evaluate the most suitable extraction methods, but also to better understand their potential of applications. In Portugal, pineapples are mainly cultivated in the islands (i.e., Azores Islands) and studies about extracted PALF from the waste of these plants are rare or non-existent in literature. In this work, the physical and mechanical properties of PALF, from plants cultivated in the Azores Islands, were analyzed and compared between different ages (i.e., 11 and 18 months) and different regions of the same fibre. The conventional scratching method was compared with the natural retting at 25 and 35 ºC and also with alternative methods (i.e., soil covered leaves and leaves boiled in water).

RESULTS AND CONCLUSIONS

Pineapple leaves were harvested from plants in São Miguel Island, Azores, Portugal. The leaves were weighted, measured and washed with water before PALF be extracted. The fresh fibers were washed several times in pure water for removing of remaining fresh residues and dried in the air for 5 days (Fig. 1-a). The 11-months and 18-months fresh leaves weighted in the range of 22.0 – 45.5 g and 29.9 – 53.1 g, respectively. They ranged from 5.7 – 7.4 cm width and 55.5 – 77.0 cm in length (11-monts), 5.9 – 6.5 cm in width and 65.7 – 96.5 cm in length (18-months). The productivity using hand-scratching method was around 7 leaves/ h. The PALFs yield and physical properties (i.e., max length and fineness) are shown in Table 1. Dry fibers yield 0.8 % wt for both ages of 11-months and 18-months PALF. Measurements were taken from 10 single fibers of each age and average diameters was estimated from optical microscopy images (Fig. 1-b) at different regions of the fibers: the initial region (region that arises from the plant) and the tip.

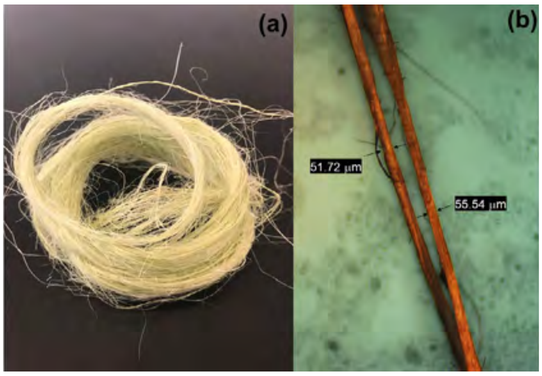

Fig.1 Extracted PALF (a) and 11-months fibers image from 5x magnification (b)

Table 1 Fibre yield and average diameters of PALF obtained from plants of different ages and different fibre regions

Pineapple Leaves	Fibre yield (wt %)	Max length (cm)	Fibre Region	Diameter (mm)
11 months	0.8	49,9	Beginning	79.85 ± 18.65
			Tip	53.27 ± 9.81
18 months	0.8	52,6	Begining	83.98 ± 19.25
			Tip	51.92 ± 8.24

Longer fibers were found for 18-months PALF since these plants had more time to grow its leaves. No differences of the fibers diameter were noticed between the different ages, but there was a significant reduction in diameter between the different regions of the same fibers. Further tests still being performed to analyze the effect of diameter reduction for the tip on mechanical properties, such as tensile properties.

ACKNOWLEDGMENTS

The authors are thankful to the project BE@T – Investimento TC-C12-i01 – Bioeconomia Sustentável, FCT PhD Scholarship (SFRH/BD/147812/2019), CEECIND/02803/2017 and to Boa Fruta for supplying the leaves.

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ID 208
THE EFFECT OF WASHING CYCLES ON THE SEWING PROPERTIES OF SHIRTING COTTON FABRICS

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ABSTRACT
In this study, 100% cotton poplin shirting fabric was procured and the sewing quality in different washing cycles was evaluated. For the seam strength and tensile strength tests, 5, 10 and 15 washing cycles were performed on the samples prepared both in the weft and warp directions, in accordance with the standard, using a laboratory type washing machine. At the same time, the seam efficiency values from the obtained data were calculated using the formula. The results showed that the highest tensile strength and seam strength values in both fabric directions were in the unwashed samples. It was observed that these values tended to decrease with the increase of washing cycles.

INTRODUCTION
Sewing quality is one of the important parameters that directly affect the overall quality of a garment. Washing cycle, which is a natural part of the usage cycle of a textile product, is one of the important parameters that affects the mechanical and performance properties of the fabric. There are many studies in the literature examining the effect of washing cycles on the garment. The effects of different washing types on fabric tensile and seam strength (Zerent et al., 2018), the effect of fabric softener usage on seam quality (Kozenıauskıene et al., 2013), and the effects of washing cycles on the properties of elastane-containing denim fabrics (Çeven et al., 2017) were investigated. Shawky (2022) examined the seam puckering, seam strength and seam elasticity after 10 washing cycles of two different 100% cotton fabrics and revealed that the fabric type and stitch density had a negative effect on the pre- and post-wash seam strength.

METHODS
In the study, 100% cotton poplin fabric, which is frequently preferred in shirting fabrics, was examined. The fabric was procured from the market. The fabric construction was 1/1 plain weave, the mass per unit area was 130 g/m^2, fabric thickness was 0,21 mm, and the fabric density was 90 threads/cm in weft and 110 threads/cm in warp. For the preparation of sewn samples, a Juki DDL-9000B lockstitch machine, a 90 Nm sewing needle and a polyester corespun sewing thread were used, while other parameters were kept constant.

The washing process was carried out using a James Hill Wascator FOM71MP washing machine with 5, 10 and 15 repetitions of washing. For washing operations were performed according to the "TS EN ISO 6330:2022" standard and the 4N program was preferred, which simulates daily use in this standard. Tensile strength tests were carried out on the Zwick Z010 (Roell) testing equipment in accordance with the "TS EN ISO 13934-1:2013" standard. Seam strength tests were also carried out in this equipment in accordance with the "EN ISO 13935-1:2014" standard both in the weft and warp directions with 5 repetitions. Moreover, for calculating the seam efficiency, equation 1 was used.

$$Seam\ Efficiency\ (\%) = \left(\frac{Seam\ Strength\ (N)}{Tensile\ Strength\ (N)}\right) x\ 100 \quad Seam\ Efficiency\ (\%) = \left(\frac{Seam\ Strength\ (N)}{Tensile\ Strength\ (N)}\right) x\ 100 \qquad (1)$$

RESULTS AND CONCLUSIONS

The results of the seam strength, tensile strength and seam efficiency examined in the study are given in Table 1.

Table 1 Results of seam strength, breaking strength and sewing efficiency

	Test direction	Unwashed	5 repeats	10 repeats	15 repeats
Seam strength (N)	Weft	272.71	260.27	239.1	235.61
	Warp	257.48	249.98	245.23	244.33
Tensile strength (N)	Weft	464.96	460.78	411.53	408.71
	Warp	677.41	671.53	662.06	638.15
Seam efficiency (%)	Weft	59.17	57.2	59.91	58.45
	Warp	38.23	37.25	37.05	38.32

According to the results of the one-way ANOVA test performed to examine the effect of washing repetitions on tensile strength, it was determined that there were statistically significant differences in both weft and warp directions ($F_{weft} = 8.90 - p_{weft} = 0.01$, $F_{warp} = 6.45 - p_{warp} = 0.02$). It was determined that the unwashed samples had the highest tensile strength in both directions. The tensile strength values tend to decrease with the increase of washing repetitions. When the effects of washing repetitions on the stitch strength were examined, it was determined that repeated washing in both weft and warp directions had an effect according to the results of one-way ANOVA ($F_{weft} = 17.56 - p_{weft} = 0.00$ and $F_{warp} = 12.61 - p_{warp} = 0.00$). According to the results of the stitch strength in the weft and warp directions, the highest strength was observed in the unwashed samples. With increasing washing repetitions, the dimensional differences in the fabric structure are balanced, and the decrease in seam strength remained stable between 10 and 15 repetitions. When the effect of repeated washing on the sewing efficiency was examined, no statistically significant result was obtained according to the results of the one-way ANOVA test ($F_{weft} = 0.58 - p_{weft} = 0.98$ and in the warp direction $F_{warp} = 0.13 - p_{warp} = 0.94$). Sewing efficiency is a criterion that changes depending on seam strength and fabric strength, and since the effect of washing is effective on both parameters, it has been determined that the seam efficiency remains stable in multiple washing processes.

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ID 210
EFFECT OF MAPP AND TALC ON RECYCLED POLYPROPYLENE AND RICE HUSK BIOCOMPOSITES FOR 3D PRINTING

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ABSTRACT
This work explores the effect of the implementation of polypropylene-graft-maleic anhydride and industrial talc as additives of composites based on rice husk and recycled polypropylene for 3D printing applications. Different formulations, where additives and rice husk concentrations were varied, were evaluated in terms of the materials' physical, thermal, and rheological properties. Obtained results demonstrate the rice husk and recycled polypropylene composites' potential to be used in 3D printing applications and the importance of using additives to make them more suitable for this field.

INTRODUCTION
Plastics represent the third most important worldwide municipal solid waste stream that is challenging to manage [1]. Also, about 5.5 billion tons per year of agroindustrial waste are produced globally [2]. Most of it is untreated and deposed into the soil, generating greenhouse gases, or it is incinerated, contributing to CO_2 global emissions [3]. Growing environmental awareness and increasing interest in sustainability have led industries to adopt responsible production and consumption methods aligned with Circular Economy principles. Accordingly, there has been a great interest in using recycled and biobased materials for manufacturing cost-competitive and biodegradable goods. Further, one industrial process well-suited to reusing post-consumer plastic is 3D printing, considered environmentally friendly due to the reduced use of raw materials. Polypropylene is one of the most used polymers, and although it is already available for 3D printing applications, its processing is still complex. Pure PP has some challenges in the 3D printing process due to the warpage induced by its semicrystalline nature and isotactic macromolecular structure. In the present study, the implementation of polypropylene-graft-maleic anhydride (MAPP) and industrial talc (T) as additives of rice husk (RH) and recycled polypropylene (rPP) composites was evaluated. Physical, thermal, and rheological properties were determined to explore the possibility of manufacturing a suitable 3D printing filament.

RESULTS AND CONCLUSIONS
Fig. 1. reports density values for the neat rPP and the composites. The density increased with increasing MAPP. However, it decreased with increasing fiber and talc content. This variation was due to the different components' densities. Composites where just the MAPP was used do not present a significant variation compared to neat rPP because of their similar densities, while variations in composites with talc results from their different densities. Melt flow index results are also presented in Fig. 1. Melt flow index is a key

property to control the 3D printing final product quality in terms of precision and interlayer adhesion. The MFI values can suggest how fast a polymer flows in the molten state. This property affects the material's processability and is linked to bonding between adjacent layers in 3D printing pieces. According to the results, MAPP increases MFI by 34.48% compared to the neat rPP. On the other hand, using talc causes a decrease in the MFI value.

Table 1 presents the principal thermal properties of the materials. Melting temperature presents minor changes. Amorphous materials are preferred in 3D printing processes as they solidify fast with less shrinkage, while crystalline polymers cause a high shrinkage and part distortion. Both MAPP and Talc cause an increase in the crystallinity of the composites. A Principal Effects Analysis was carried out: the interaction of RH and talc affects the crystallinity of the components, while the interaction between RH and MAPP does not.

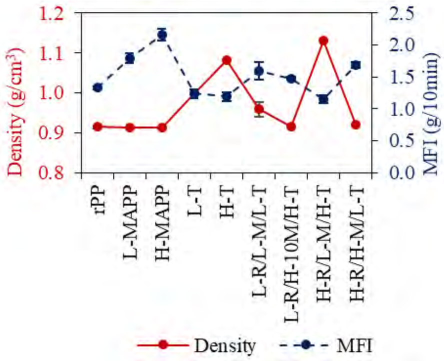

Fig.1 Density, and MFI values of the evaluated composites. (L: low level; H: high level)

Table 1. Principal thermal properties of rPP and rPP composites

Configuration	Crystallization		Melting		Crystallinity
	T_c (°C)	ΔH_c (J/g)	T_m (°C)	ΔH_m (J/g)	X_c (%)
rPP	129.96	85.05	165.98	81.98	39.60
L-MAPP	128.55	89.4	165.45	83.09	40.14
H-MAPP	127.76	87.52	163.93	85.45	41.28
L-T	130.21	75.27	165.73	73.56	41.81
H-T	130.26	63.26	165.81	61.92	42.73
L-RH/L-MAPP/L-T	129.12	69.78	165.25	70.26	44.66
L-RH/H-MAPP/H-T	125.29	57.32	163.51	53.21	46.74
H-RH/L-MAPP/H-T	128.62	52.38	164.83	52.34	45.15
H-RH/H-MAPP/L-T	127.62	67.56	165.18	63.84	47.45

This work explores the implementation and influence of MAPP and talc as additives for RH/rPP composites in physical, thermal, and rheological properties to study their possible applications in 3D printing technology. Results show the potential of using MAPP to improve the interaction between the RH and the rPP polymeric matrix. In the evaluated concentrations, talc induces high crystallinity and low MFI values.

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ID 211
A NUMERICAL STUDY ON THE OPTIMIZATION OF THE BONDING PERFORMANCE OF SNFT BIO COMPOSITES

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ABSTRACT
This work focusses on the implementation of short natural fibre reinforced thermoplastic (SFNT) bio composites within larger structures. Considering adhesive bonding as an assembly technique, a multi-scale three-phase modelling approach of the SFNT is used, based on the rheological behaviour of the composite flow in the mould. To improve the bonding performance of a single lap joint (SLJ), this work presents an optimization algorithm where the performance of the bonded structure is linked back to the injection moulding process parameters. By optimization of the fibre architecture within the composite structure, the bonding performance was improved.

INTRODUCTION
The use of short natural fiber reinforced composites has shown a promising effect on the mechanical properties of the composites. This allows the use of this type of material in high demanding applications. In this study, flax stems are considered as natural fiber reinforcement. These stems consist out of fiber and wooden parts, known as shives. The use of these flax shives as reinforcements has proven to significantly affect the mechanical properties of the composite (L. Nuez, L. Nuez, K. Soete). This work therefore focusses on using the full flax stem, cut at a length of approximately 10 mm, as reinforcement within a bio-based polylactic-acid matrix and use this composite material as substrate material for an adhesively bonded structure. The mechanical performance was numerically determined using a three phase, multi scale modeling approach, in which local fiber orientations originating from the injection molding process, are considered. The mechanical behavior was studied for conditions at room temperature with a RH of 50%. A single lap joint (SLJ) configuration was considered in this study, as this is one of the most common joint designs. This work focusses on the effect of the fiber architecture of the SNFT substrate on the bonding performance.
Due to the occurring eccentricity when a SLJ is loaded in tension, stress concentrations in shear and peel occur at the edge of the overlap. These stress concentrations are responsible for failure of the bonded joint (S. Her). Among other variables, which are kept constant in this work, these stress concentrations mainly depend on the mechanical properties of the substrate material. Considering short fiber composite substrates, it was found by (S. Verstraete) that variations in fiber orientations affect these stress concentrations. In this work, a first step exists of studying the effect of local fiber orientations trough the thickness of the substrate on the stress distribution in a single lap joint were studied. These mainly depend on the production process, where a skin-shell-core orientation occurs. Process parameters such flow rate, mold wall temperature and packing pressure were used to optimize the injection molding process by maximizing

the shear layer.

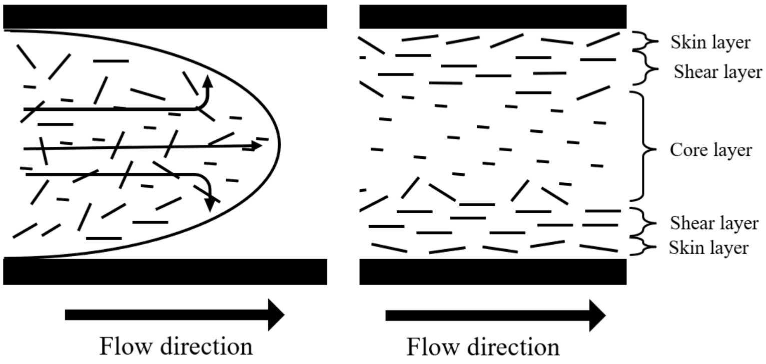

Figure 1: Schematic view of the fiber orientation trough the thickness off SNFT substrates.

RESULTS AND CONCLUSIONS

Numerical results on a dedicated study on the effect of variations in fibre orientation of the short natural fibres through the thickness of the SNFT substrate indicate to directly influence the stress concentrations in a single lap joint. Due to the occurring eccentricity, the effect of the bending stiffness plays a large role in the deformation behaviour of the joint, which is directly affected by this fibre architecture. By optimizing the process parameter of the production process, a maximization of the shear layer could be obtained, resulting in an increase of the bonding performance.

ACKNOWLEDGMENTS

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ID 212
EFFECT OF ECO-FRIENDLY CHEMICAL TREATMENTS ON THE MECHANICAL PROPERTIES OF THE FLAX FIBRE REINFORCED BIOCOMPOSITES

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ABSTRACT
It is well known that natural fibres have a strong polar characteristic, which may drastically affect the fibre-matrix interaction in the composite structure. There are already well-established chemical treatment techniques for fibre treatment, but in this work, we used eco-friendly chemicals to treat the natural fibres. The coffee ground extract, citric acid, and fatty acids were used as basic chemicals to pretreat the fibres, and the conditions for each technique were optimized in this work. The change in surface energy of the treated fibres was characterized using contact angle measurement. The treated fabrics were impregnated with bio-based epoxy resin using resin infusion technique. The fibre volume fractions were between 30-35%, and the mechanical properties of the composites were characterized. An increase in flexural strength was observed after the pretreatment. Long-term water absorption was carried out to assess the water resistance as well as the quality of the impregnated composites. Additionally, atmospheric plasma polymerization (physical treatment) was also carried out using bio-based precursors at a nanoscale on the fibres, which is an alternative to conventional chemical treatment techniques. The research results are summarized in this paper

INTRODUCTION
Currently, more than 95% of the market share is held by glass fibre, especially in the composite industries. Natural fibres have limited external use due to low mechanical strength, inferior water resistance, and weak interfacial adhesion between the matrix and the resin [1]. However, the use of natural fibres as renewable reinforcement for composites provides several advantages such as eco-friendliness, lightweight natural fibre composites that improve fuel efficiency, reduce CO_2 emissions, and provide carbon credits from recovered energy through incineration of natural fibres [2]. Interfacial adhesion can be improved by surface treatment, which is generally classified into two major categories: physical and chemical treatment techniques. Our aim is to replace classical chemical treatment techniques, such as sodium hydroxide, peroxides, etc., with eco-friendly chemicals in order to facilitate sustainable manufacturing processes. In this work, we investigated the feasibility of using these chemical treatment methods to enhance the mechanical strength of the composites indirectly by means of improving the fibre-matrix interaction.

RESULTS AND CONCLUSIONS
For the surface treatment, the coffee ground extract was prepared by boiling the coffee grounds in water for 2 hours, and then the concentrate was obtained by filtering the mixture. The final filtered concentrate was

infused into the dry flax scrims using the resin infusion technique, and after surface treatment, the scrims were washed. The treated fibers were then combined with the bio-based epoxy resin from Sicomin (Greenpoxy 56) to make composites. In addition to citric acid and fatty acids treatment, acetic anhydride was used as reference. Contact angles were measured, and the results showed that the untreated flax had a contact angle of 118°, whereas an increase in contact angle was observed for the acetic anhydride-treated flax fibers (124°). The citric acid and coffee ground extract treatment decreased the contact angle values to below 100° (Figure 1). The results confirmed that surface treatment techniques have changed the surface structure both chemically and physically. The mechanical properties of the composites made using treated fibers showed improvement in flexural strength, reaching up to 185 MPa, and modulus values between 6 to 10 GPa (Figure 2). A reduction in stiffness was observed for the citric acid-treated composites. Physical treatment using atmospheric plasma polymerization (bio-based methacrylate precursor) showed a change in contact angle values, but a marginal change in mechanical strength was observed. This phenomenon might be due to the low quantity of deposited polymer precursors on the surface.

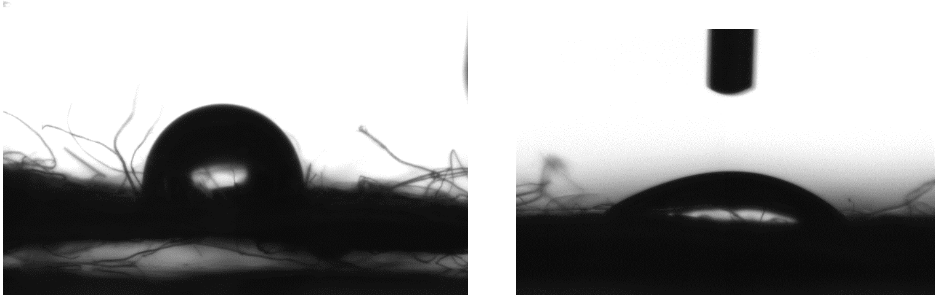

Fig.1 Water drops on the untreated flax scrim and on the citric acid treated flax

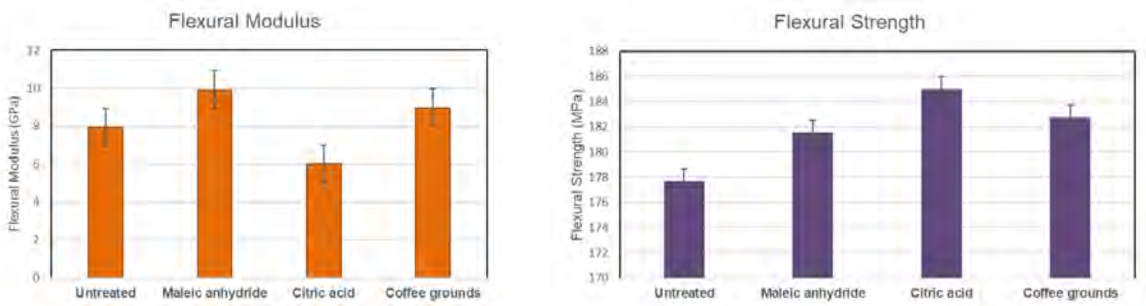

Fig.2 Flexural strength and modulus of the composites made using surface treated fabrics

The results show that eco-friendly chemicals can also provide comparable results with classical chemical treatment techniques. In the future, it will be interesting to study the pretreatment using chemicals extracted from plant waste. The physical treatment using plasma polymerization for fiber treatment is quite new and needs to be investigated with different precursors and coating thicknesses on the fiber surface.

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ID 213
FATIGUE LIFE PREDICTION OF HYBRID CARBON/FLAX-EPOXY COMPOSITES UNDER STRAIN- CONTROLLED LOADING USING A 3D PROGRESSIVE FATIGUE DAMAGE MODEL.

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ABSTRACT
A 3D progressive fatigue damage model 3D PFDM was developed and incorporated into ABAQUS finite element software through a user-defined subroutine. The 3D PFDM Abaqus was utilized to predict the fatigue life of hybrid carbon/flax-epoxy composites and the failure modes of these hybrids. The model input parameters of the hybrid composites laminates were obtained from experimental testing and literature. The proposed 3D PFDM was firstly validated on pure UD and angle ply flax/epoxy laminates, showing a good agreement between the experimental and the predicted results at different strain loading levels. This proposed 3D PFDM highlighted the effect of the void content on the fatigue life and the failure modes of the hybrid composite laminates.

INTRODUCTION
Natural fibers as a reinforcement in polymer composites have gained popularity in many engineering fields as concerns about environmental and sustainability issues are on the rise. While natural fibers have many advantages, they also have some disadvantages, which has led researchers to investigate hybridization of natural fibers with synthetic fibers. Fatigue and fatigue related phenomena are responsible for most failures in engineering applications therefore knowing the fatigue behaviour of these hybrid composites is integral. Numerous tests are needed to determine the fatigue properties of a composite and to plot the S-N curve. These tests are costly and time consuming so many researchers have developed several fatigue models to predict the fatigue properties and behaviour of composites, however very few models have been developed for hybrid composites (Linde & de Boer, 2006). In this study, hybrid composites made of 12-layers of unidirectional flax sandwiched between 4-layers of 4-harness satin woven carbon. The 3D PFDM model incorporates several failure criteria. The maximum stress was employed to identify fiber breakage in tension, while 3D Hashin failure criterion was used to detect matrix cracking in both tension and compression, fiber failure in compression, and fiber/matrix shear failure. The Ye-delamination criterion was used to identify delamination in tension and compression in flax laminates (Kolasangiani et al. 2021 & 2022). The interface between woven carbon layers as well as between flax and carbon layers were modeled using a Cohesive Zone Model (CZM) where the cohesive elements are defined by a traction-separation law that relates the interface stress to local separation at the crack tip. The Benzeggagh-Kenane (B-K) (Benzeggagh and Kenane, 1996) fracture criterion was used to determine the failure in the cohesive layers.

RESULTS AND CONCLUSIONS

Fig. 1 shows that the predicted strain-life curves for both layups are in good agreement with the experimental results. For high strain levels, the effect of the void content on the fatigue life of laminates is more pronounced for unidirectional plies compared to angle plies. Fig. 2 plots the variation of failure indices for a weak element in the $[\pm45]_{4S}$ laminate under a 1.1% maximum strain loading. The matrix in angle plies failed at around 70% of fatigue life and the load is carried out by fiber in the remained of fatigue life. The delamination failure index is negligible until 50% of fatigue life, after which it increases exponentially until the final failure. This corroborates with SEM image taken at 75% of fatigue life and under 1.1% maximum strain loading (Fig 2b, showing matrix cracking before 75% of fatigue life.

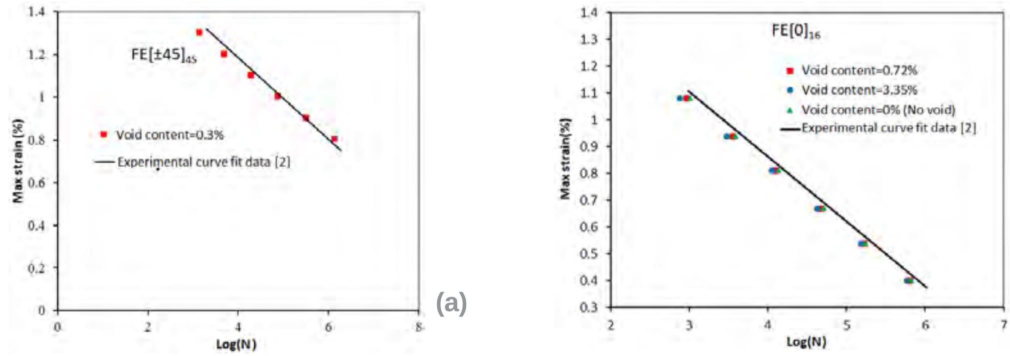

Fig.1 Strain life curves for flax epoxy laminates **(a)** UD and **(b)** Angle ply.

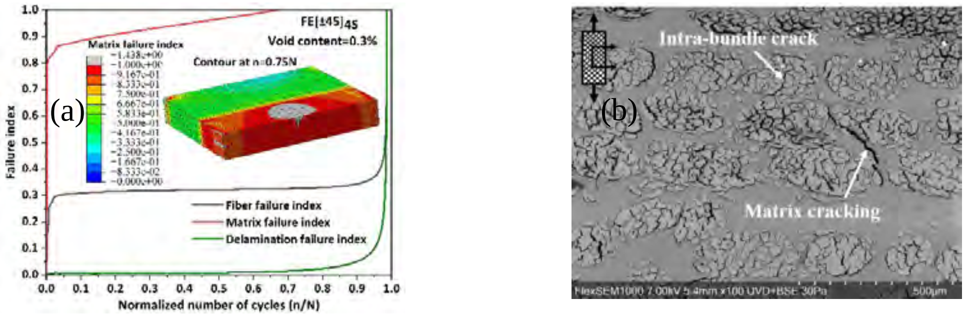

Fig.2 Results of fatigue analysis for [±45] laminate under a 1.1% maximum strain loading **(a)** Failure indices evolution versus number of cycles (n), **(b)** SEM micrograph after 75% of n.

ACKNOWLEDGMENTS

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ID 214
EVALUATION OF ANTIOXIDANT AGENTS FROM THE JATOBA (HYMENAEA COURBARIL L.) FIBER FOR POTENCIAL APPLICATION AS BIODEGRADLE 3D SCAFFOLDS

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ABSTRACT
Hymenaea Courbaril L. is a tree native from the cerrado and its parts such as bark, leaves and fruit are used for many purposes. Traditionally, in some Brazilian regions, the *H. Courbaril* pulp is used for medicinal purposes, but its bark, which is rich in fibers and antioxidant agents, is commonly thrown away. Considering the possibility of using the reinforcement and antioxidant properties of this fibers in biomedical devices, this work describes the qualitative characterization of the brown powder obtained from drying and crushing the *H Courbaril* barks.

INTRODUCTION
Plants are a good source of fibers and active compounds for reinforcement and controlled release purposes in biomedical devices, such as scaffolds for wound healing and bone regeneration. Since medicinal and aromatic plant derivatives are non-narcotic and have no side effects, even if used for prolonged time in permissible doses, the demand for these plants is increasing in both emerging and developed countries (Males et al., 2022). In this context, the Brazilian flora plays an important role in the search for different fibers with therapeutic properties (Dutra et al., 2016). As well as several important medicinal species, *H. Courbaril L*. is a plant genus with great chemical and pharmacological potential (Jacob et al., 2022). This species is popularly called "Jatobá" and known as a medicinal plant for cough, bronchitis, flu, weakness, anemia, prostate problems and gastritis aches and pains, fever, respiratory diseases and lung disorders, and diarrhea (Ribeiro et al., 2017). It is also used for wound healing and as an anti-inflammatory (Pacheco and Almeida, 2022). All parts of the fruit possess medicinal properties, especially the bark, leaves, sap, resin and pulp, but the bark is the least explored part. Thinking about the great demand of the population for green alternatives of medicinal treatment, an in-depth study on the constituents present in this fruit is of scientific interest. It was already reported in literature, the presence of diterpenoid alkaloids in *H. Courbaril* fruits (Rodrigues et al., 2021). This compounds are a family of extremely important natural products that have long been a research hotspot due to their myriad of intricate structures and diverse biological prop-

erties. Besides that, it is also expected the presence of succinic acid (Rodrigues et al., 2021), a powerful natural antioxidant, capable to stimulate the nervous system and to help the body's immunity. Widely used in the pharmaceutical industry, succinic acid is known for its immunostimulating, analgesic, healing and anti-inflammatory properties.

This work reports the qualitative characterization of diterpenoids and succinic acid in the fibers from *H. Courbaril*'s bark. This is a preliminary result of a study that will involve the preparation of scaffolds containing these agents and evaluating their potential application in wound healing devices.

RESULTS AND CONCLUSIONS

The FTIR spectrum of treated fibers from *H. Courbaril*'s bark (Fig. 1) revealed bands at 2930 and 2870 cm^{-1} related to C-H single bond, at 1697 and 1630 cm^{-1} which is attributed to C=O and C-C bonds stretching, respectively and at 1446, 1366 1233, 1195 and 889 cm^{-1} representing skeletal C-O and C-C vibrations. The 1233 and 1195 cm^{-1} bands indicate the presence of esterified succinic acid, succinate (Rodrigues et al., 2021). The bands observed at *H. Courbaril*'s bark spectrum are consistent with its major components, diterpenoids based on labdane structure, such as eperuic and copalic acids (Doménech-Carbó et al., 2009).

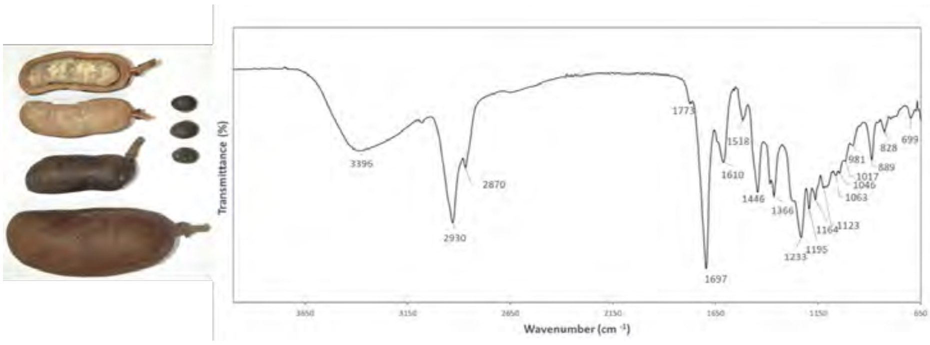

Fig.1 *H. Courbaril* image and FTIR spectrum of treated fibers from *H. Courbaril*'s bark.

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ID 218
A STUDY OF HEMP STRAWS RETTING: COMPARISON OF DEW, WATER AND CHEMICAL RETTING ON HEMP FIBRES QUALITY

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ABSTRACT
Satisfying both industrial and societal demands for innovative, less energy-consuming (production, use, end-of-life) and local materials, the hemp fibre market is experiencing significant growth resulting in a sharp increase in cultivated areas. However, the supply of fibre to these new markets (automotive, construction, textiles) imposes new quality standards, resulting in an increased demand for retted fibre. However, through its usual protocol in Europe, dew retting, which is highly dependent on climatic and soil conditions, a high degree of fibre quality heterogeneity is potentially expected. In this context, various studies have been carried out with the aim of comparing different retting processes and initiating the development of methods/tools that will eventually enable industrials to monitor the quality of retted straw with a view to contractualization. This work studies the effect of different methods of hemp straws retting: dew, water and chemical retting were performed on the same batch of unretted hemp straws. Chemical analyses were carried out to compare the obtained samples.

INTRODUCTION
Hemp production is gradually increasing in France, with 22 000 ha harvested in 2022 in response to the development of new markets. Originally destined for the paper industry, hemp fibres are now increasingly used in technical markets such as plastics for the automotive industry, insulation for the building industry and now for textiles. However, these new markets require a review of the fibre supply chain and in particular the quality of the fibres. Previous work conducted by FRD and the UMR FARE has shown that retting plays a key role in fibre quality (MAPROFI & SINFONI Projects, Chabbert et al, 2020). Numerous studies have been carried out on the retting of fibre plants (flax, hemp, kenaf, etc.) proposing a variety of methods: dew retting, water retting, chemical retting (Mazian et al, 2020; Konczewicz et al, 2017)…. These aim at modifying the chemical characteristics of the straw and/or fibres through the action of microorganisms, enzymes or chemical agents. The quality of obtained fibres depends on the protocol, the duration, the quality of the straw at the beginning of retting, and the weather conditions for dew retting (Tahir et al, 2011).
The growing industrial demand for retted hemp fibre requires i. a better understanding of the process, ii. the consequences of retting on the final properties of the products and iii. how to assess the quality of retted

fibres. The RIGHTLAB project (Requilé et al, 2021) identified retting indicators that led to current projects SUNRISE, NF-Aureo, and the associated partner laboratory 4FM with FRD and UMR FARE. These projects and the 4FM laboratory respectfully aim to test these indicators operationally and then provide industry with decision support tools to manage biomass quality for material use.

RESULTS AND CONCLUSIONS

The effect of different retting strategies on hemp variety Futura 75 was investigated in order to link the chemical properties of the fibres, using the Van Soest method, to their final quality. Dew retting was carried out during 8 weeks, with straws collected every 2 weeks. Water and chemical retting were performed on a same batch, typical of unretted hemp straws entering in a factory, on the laboratory scale: water retting for 1, 5 and 11 days, and chemical retting with a sodium hydroxide solution for 1 and 5 hours.

The solubles rate decreases with every retting method, being reduced in half at the end. Fig. 1 shows that the tendencies are similar, except for the timeline. A 6-week dew retting gives a similar chemical composition than an 11-day water retting and a 1-hour chemical retting.

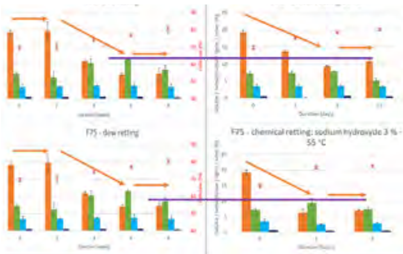

Fig.1 Chemical composition results according to the method of retting

This study shows the differences and similarities between different retting methods on hemp fibres chemical composition. Further analyses should be performed to analyse other properties, such as IR spectroscopy, odours, and mechanical properties of fibres and composites made from these fibres.

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ID 219
LOAD – SETTLEMNET ANALYSIS REINFORCES SOIL WITH COCONUT LEAF MAT – A SUSTAINABLE NATURAL MATERIAL FOR GROUND IMPROVEMENT.

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ABSTRACT
In this paper, the authors aim is to evaluate the advantageous application of coconut leaf mat as a reinforcement material in sand, as demonstrated through large scale plate load tests. Several tests were conducted to vary the depth of the first layer of reinforcement, the width and distance between two layers of reinforcement, as well as the number of layers. The laboratory test results showed that the maximum improvement factor for the first layer of reinforcement and the distance between the reinforcement was obtained at a depth equal to 0.4 times the width of the footing. Additionally, the critical width of reinforcement was found to be three times the width of the footing. Finally, no significant improvement was observed beyond the use of four layers of reinforcement.

INTRODUCTION
Over the past two decades, the use of geosynthetic materials as geotextiles for soil reinforcement has become increasingly popular. However, with concerns over adverse environmental issues such as soil pollution and sustainability, there has been a growing interest in using geotextiles made from natural materials. Many past researchers have made numerous attempts to prove the effectiveness of natural fibers as reinforcement materials for soil. Examples of natural geotextile materials include coir, jute, kenaf, bamboo, and rattan. The load-settlement behavior may vary depending on the form and type of material used for reinforcement. Furthermore, the use of natural materials has been shown to be a cost-effective and sustainable solution for reinforcing large areas.
Coir fiber is a popular natural material for soil reinforcement due to its cost-effectiveness and easy availability. Ajitha and Jayadeep (1997) conducted a comprehensive study to investigate the effect of soil-coir frictional interfacial behavior. The results showed that the shear improvement factor in terms of frictional resistance was 1.5 times higher than that of unreinforced soil. Vinod et al. (2009) evaluated the load settlement behavior of square footing on sand reinforced with braided coir ropes and found that it could improve the bearing capacity of the soil. Praveen and Bajinder (2010) demonstrated the suitability of incorporating randomly included jute fibers into soil for road embankment strengthening. CBR tests showed that the improvement factor increased by up to 2.5 times with the inclusion of bitumen-coated jute fibers. Akhil et al. (2019) conducted a series of large-scale laboratory tests with square footing on planar reinforced bamboo mats, and also elucidated the effect of surface roughness and aperture size. The results showed that the improvement factor increased up to three times that of bare soil.

Previous literature indicates that there has been little exploration of coconut leaf mat geotextiles. Fig. 1 illustrates the coconut leaf mat, and Fig. 2 depicts the scaled model mat used for large-scale plate load testing. The authors of this study aimed to examine the loadsettlement behavior of the reinforcement material. The study's objective was to determine the optimal values for:

The depth of the first layer of reinforcement
The distance between two layers of reinforcement
The width of reinforcement
The number of layers of reinforcement

Fig.1 Woven coconut leaf mat **Fig.2** 30cm x 30cm coconut leaf for testing

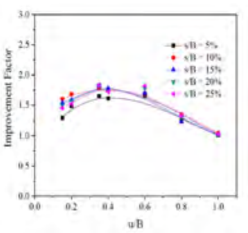
RESULTS AND CONCLUSIONS

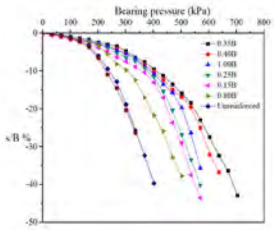
In this paper, in order to study the bearing capacity characteristics of reinforced soil, a non-dimensional parameter were adopted, which is Improvement factor (I. F) as represented in Eqn. 1 This value defines the ratio of bearing capacity of reinforced soil to the bearing capacity of unreinforced soil at a specified settlement. The ultimate bearing capacity was found out corresponding to different s/B% (5%, 10%, 15%, 20% and 25%) with 30% and 60% relative density. All the identical test series were tested two times in addition to the main test to assure the uniformity of results.

$$I.F = {B.P_R}/{B.P_u} \quad \text{Eqn. 1}$$

Influence of first layer of reinforcement.

Present set of experiments were conducted to evaluate the optimum depth from the footing base to the first layer of reinforcement. The analysis of uR/B ratio is very significant in ground improvement because it may vary with frictional pull-out resistance of reinforcement, rapture strength of reinforcement, types of soils etc.





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ID 220
CULTURE MEDIUM EFFECT ON PROPERTIES OF BACTERIAL CELLULOSE MEMBRANE PRODUCED BY KOMAGATEABACTER MELOMENUSUS AV436^T BACTERIAL STRAIN, USING TWO DIFFERENT CULTURE MEDIA

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ABSTRACT
Valorisation of the industrial and agricultural wastes and by-products in biotechnological processes can be a cost-efficient alternative to defined media used in bacterial cellulose (BC) production. Herein, we investigated the influence of culture medium on selected properties of BC membrane, produced by *Komagateabacter melomenusus* AV436^T bacterial strain, using two different culture mediums: reinforced acetic acid-ethanol (RAE) medium and grape pomace (GP) extract. Results indicate on presence on significantly finer microstructure, higher crystallinity, tensile strength, and elongation at break for BC membrane prepared in RAE culture medium, comparing to GP extract product.

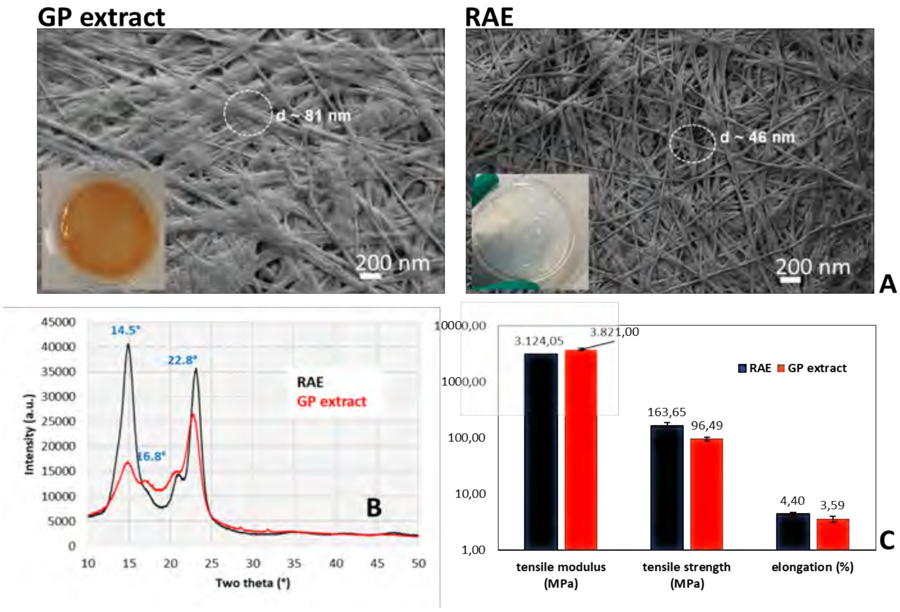
INTRODUCTION AND EXPERIMENTAL WORK
BC is a unique exopolysaccharide produced extracellularly by different bacterial strains (Gorgieva and Trček 2019), where the static, undisturbed cultivation results in the formation of a 3D gelatinous pellicle with highly entangled nanofibrils network. BC synthesis's biotechnological process often uses defined media (Hestrin–Schramm, Zhou,Yamanaka). The cost-effectiveness of the biotechnological process for nanocellulose synthesis is tightly related to the selection of the culture medium, where the carbon source accounts for 30% of the net cost. The sugar-rich wastes and by-products, such as corn steep liquor, date syrup, stillage from rice mine distillation, wheat straw, and confectionery waste, can serve as a valuable alternative to the defined mediums in terms of cost and availability. On the other hand, diverse applications are foreseen for BC (Gorgieva and Hribernik 2019; Gorgieva 2020; Żywicka et al. 2023), require defined properties and quality attributes, which can be affected mainly by biotechnological process, considering the culture medium as an essential variable.

Herein, we process the BC described in our previous work (Gabrys et al. 2023), using the *K. melomenusus* AV436^T bacterial strain from the microbial culture collection at FNM, UM. GP extract was prepared from Slovenian grape (2021 autumn season), according to the procedure which is under patenting. The surface morphology of BC membranes was assessed by Scanning Electron Microscopy (SEM) on a microscope Supra 35 VP (Carl Zeiss) using 1.00 kV acceleration voltage and low vacuum. X-ray diffraction (XRD) data was recorded on D2 X-ray diffractometer (Bruker Siemens) in reflection mode, using the monochromatic

CuKα radiation with λ = 0.15418 nm, generated at a voltage of 30 kV and current of 10 mA. Reflections at 2θ were scanned in the 10 - 50° range, with an increment of 0.03°. The tensile test was performed on wet samples on Shimadzu, AG-X plus 10 kN electromechanical universal testing machine. Application of tensile force (10 kN load cell) proceeded at 1 mm min^{-1}.

RESULTS AND CONCLUSIONS

The morphological features of BC membranes visualised by SEM (Fig. 1A), revealed the ribbon-like microfibrils, randomly entangled into a dense network-like structure. Twice wider nanofibrils were observed in GP extract, related to the ˝gluing˝ effect of some GP components.



XRD data (Fig.1B) shows peaks at 2θ =14.4°, 16.8°, and 22.6°, which are assigned to the (101), (10$\bar{1}$), and (002) planes of cellulose I, in both cases. The amorphous segment (~ 18°) is visibly higher in samples produced in GP extract, and the CI (%), calculated by using the deconvolution procedure, is significantly lower (< 50%) compared to 72% in the case of RAE. We anticipate that a different type of reducing sugars in the culture medium can affect the cellulose chain arrangement. The organisation of BC fibrils within the network allows stress transfer and cohesion, whereas the non-cellulosic components can destroy the compactness, resulting in the loosening of the BC network. Compared to RAE, the lower tensile strength and elongation were measured in GP extract BC. Our work demonstrates significant differences among properties of BC membranes produced by the same bacterial strain when different culture mediums are used, especially in terms of morphology and crystallinity. This points to the importance of selecting the medium culture source when the final material's properties (purity, crystallinity….) are required.

ACKNOWLEDGMENTS

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ID 222
CIRCULAR ECONOMY: DEVELOPMENT OF FUNCTIONAL TERRY TOWELS FROM COTTON RESIDUES

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ABSTRACT
The main objective of this study is to understand the viability of replacing virgin raw materials with recycled ones in the development of terry fabrics to give a second life to the waste generated. To this end, mechanical recycling was carried out with subsequent application of the residues in the production of yarn and later a terry fabric. This work studies the functional properties (mechanical and absorption) of the terry towels developed. Although the recycling process tends to change fibers resistance, the performance of the terry fabrics wasn´t affected, this result is more evident in the absorption properties. These terry towels have the potential to be applied in the home textile market.

INTRODUCTION
In 2022 world population has reached 8 billion and, consequently, the society's needs as well as the consumption of resources [1]. The linear strategies adopted by the companies in the past years increased the consumption of virgin material, energy, water, and chemicals, as well as the production of waste. Circular strategies have emerged as an alternative to solve these problems [2], [3].
This study aimed to validate the reintroduction of textile waste in production cycles. The residues were mechanically recycled, introduced in the production of yarn, and finally, used for the production of terry. To evaluate the effectiveness of these processes, two terry fabrics -R1 (40% recycled cotton + 40% weaving preparation residues + 20% virgin cotton) and R2 (50% recycled cotton + 50% weaving preparation residues) were characterized in terms of mechanical properties and moisture management. These samples were compared with terry made from virgin cotton (S). Their construction is the same, 3-pick terry and 18 ends/cm.

RESULTS AND CONCLUSIONS
The residues from different phases of terry production were analyzed and, it was concluded that the ones from weaving preparation have potential to be reintegrated in the production process. In this sense, these residues were mechanically recycled and a yarn with these materials was produced, as well as a terry fabric. As shown in Table 1, it was observed that the introduction of a higher percentage of residues in the yarn composition (R2) impaired static absorption (-8%), however, it increased the performance of the structure in all other properties. It was also observed that sample R1 was able to improve its performance over sam-

ple S in most properties, except for wet sensation (5%) and tensile strength in the weft direction (-95%). Comparing the two developed samples, it was observed that R1 sample presented the best performance of absorption properties, probably due to the presence of higher amounts of virgin cotton. Regarding the mechanical properties it was observed that R2 sample stood out, and the higher percentage of residues did not appear to negatively impact the performance of the terry fabric.It is frequently observed that the introduction of residues can negatively affect the properties of the final product, however, this study proves the viability of the reintroduction of waste in production systems. Radhakrishnan et al. observed the same conclusions in their studies [4]

Table 1: Functional properties results of Standard, sample R1 (40% recycled cotton + 40% weaving preparation residues + 20% virgin cotton) and sample R2 (50% recycled cotton + 50% weaving preparation residues).

			Standard	R1	R2
	Absorption Properties				
Absorption properties	**Aerial Mass [g/m^2]**		517	450	523
	Moisture release [%]		47	50 21%	48 0%
	Static Absorption [%]		519	486 8%	480 -8%
	Wet sensation [%]		73	67 5%	73 -1%
Mechanical properties	**Tensile strength [N]**	Warp	425	280 35%	364 51%
		Weft	454	220 -95%	354 8%
	Abrasion resistance [%]		-1.7	-2.2 0%	-1.7 -36%

Based on these results, it is possible to conclude that the introduction of residues in the development of terry fabric did not negatively influence the mechanical properties and absorption problems, contrary to expected. So, it was possible to achieve the behavior that suits the applicability of this product, that is, excellent absorption properties and mechanical performance.

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ID 226
CHARACTERIZATION CELLLULOSE NANOCRYSTAL RAINFORCED CELLULOSE DERIVATIVES COMOSITE FOR SUSTAINABLE APLICATIONS

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ABSTRACT

In the present research, Jute cellulose nanocrystal (CNC) is reinforced with jute-based biodegradable biopolymer material (Sonali Bag™) and cellulose acetate film for preparing composite materials for sustainable applications. CNC has been extracted from jute by a 64% sulfuric acid hydrolysis process. The characteristics of the synthesized CNC and composite films were studied using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray diffraction (XRD) analysis. thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), mechanical properties, contact angle, water vapor transmission rate, and biodegradability in soil.

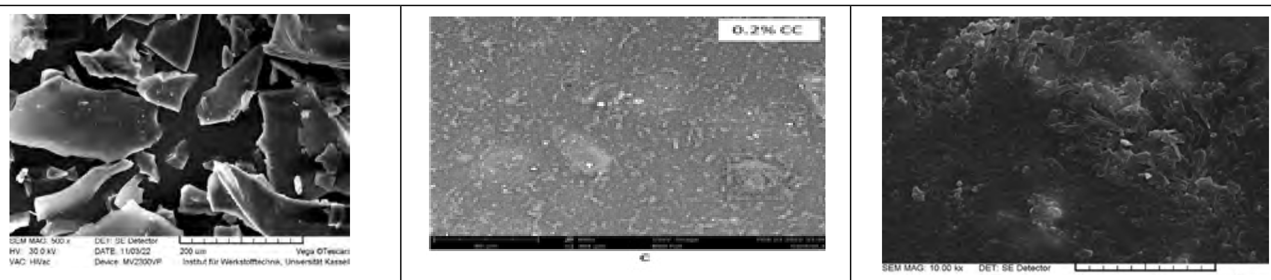
INTRODUCTION

Cellulose is a sustainable source of chemicals and energy due to its biodegradability, chemical resistance, and thermal durability. A cellulose derivative is one of the most important biopolymer and is widely used in various industries (Qiu, X., 2013). Nano cellulose and cellulose nanocrystals (CNC) are derived from cellulose and are used to produce high-tech materials for different applications such as drug delivery, hemodialysis, water treatment, and polymeric composites (Eichhorn, 2010). CNC has a low density, a high specific surface area, high mechanical properties, and surfaces accessible to chemical functionalization (Siqueira, 2009). The combination of cellulose derivatives and CNC can solve the problem of low strength and provide sustainable raw materials (Reising, 2013). Composites are produced by dispersing dried cellulose nanoparticles into a cellulose or cellulose derivative solution followed by casting and drying. Melt-extrusion is another processing technique that can be used in the thermoplastic industry. The aim of this research is to increase the physical and biodegradation properties of by reinforcing jute cellulose nanocrystal with jute cellulose derivatives based bio–composite as the resources is same of both.

RESULTS AND CONCLUSIONS

In the modern era, the development of biodegradable materials is one of the most popular topics in the field of materials science because of the adverse effects of plastics. Recently, the use of elongated cellulose nanocrystals (CNC) for bio-nanocomposites development has attracted more and more attention in the field of nanotechnology. The Sonali Bag and cellulose acetate film were dissolved in water and N, N

Dimethylformamde respectively then reinforced with cellulose nanocrystals at 0, 0.1, 0.2, 0.3, and 0.4 wt.% loadings. The results found that 7 nm crystal size spherical shape cellulose nanocrystal was found where highly crystallinity, and morphological test represent the presence of nanoparticles with tears on the surface (Figure 1). The SEM micrographs showed that CNC loading up to 0.2% assisted reduce the pore formation, however increasing the CNC loading any further caused agglomeration and pores (Figure 1). The mechanical properties were increased (about 30% than control) after reinforcing 0.2% CNCs with Sonali Bag and cellulose acetate film composite. Mechanical prperties was also slightly increased for reinforcing CNC with both bio-composites. Another result of water absorption capacities such as contact angle, water vapor transmission rate, and water uptake showed that 0.2% CNCs reinforced film represents about 30% better result than others. The composites were tested for biodegradability and all the films degraded in the soil after 100 days and the addition of cellulose crystal decreased the degradation rate.



a b c

Figure 1: SEM image of **(a)** highly nano-cellulose crystal **(b)** 0.2% CNC reinforced Cellulose Sonali Bag film,**(c)** 0.2% CNCs reinforced Cellulose Acetate film composite.

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ID 228
THE POTENTIAL USE OF ACACIA SPP. IN THE DEVELOPMENT OF POLYMERIC COMPOSITE MATERIALS

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ABSTRACT
This work shows the mechanical and thermal properties of the composites produced by injection molding using polyethylene and recycled polyethylene matrices with reinforcement of natural fibers extracted from Acacia spp. plants, at 20% and 40% w/w. The results showed that the NaOH-treated plant material improved the mechanical properties of the composites. In addition, it was proved that the use of recycled polyethylene does not reduce the final properties of the composite; in fact, flexural properties increase is higher for the materials with the recycled matrix.

INTRODUCTION
High Density Polyethylene (HDPE) is one of most widely used polymers worldwide and has multiple advantages when it is used with fibrous reinforcements. However, these good properties, together with the overuse of such materials, are causing environmental problems related to accumulation and lack of appropriate waste management. Despite this, recycled plastic has become popular in recent studies because it could be a promising raw material source for natural fiber-plastic composites (Gulitah,2019). The research in the field of fiber-shaped reinforcements shows special attention to vegetable fibers because of the comparable results to generally used synthetic reinforcements, such as glass or carbon fiber. There are several sources for extraction of fiber from plants, among them, the campaigns for the eradication and control of invasive plants species, such as Acacia spp., which is registered by the Spanish Catalog of Invasive Alien Species.
In this work, HDPE (HD6081 from Total) and a recycled grade of polyethylene (HDPE.Re) were used in combination with ground lignocellulosic material from the stems of the invasive plant Acacia spp. (AC) in percentages of 20% and 40% w/w. The obtained composites were mechanically characterized, and their thermal properties were analyzed by differential scanning calorimetry (DSC) and thermogravimetry (TGA), including the variation of the fiber properties due to the chemical treatment with 1M NaOH. The reprocessing of the composites after going through a previous grinding and a second injection cycle was also assessed.

RESULTS AND CONCLUSIONS
The natural fibers and the plastic matrices were mixed using a co-rotating twin-screw extruder. Standard test samples were obtained by injection molding. The results of the mechanical characterization of the different formulations, with 20% and 40% w/w filler, and without and with chemical treatment of the fibers (AC and ACt), are shown in Table 1.

Table1 Mechanical properties of HDPE and HDPE.Re matrices reinforced with Acacia spp. fibers (first injection cycle).

Formulation	Tensiletest		Flexuraltest	
	σmax (MPa)	E(MPa)	σmax (MPa)	E(MPa)
PE	20.63±0.47	699.17±82.73	23.78±0.15	990.46±25.96
HDPE.AC.20	19.47±0.17	860.41±36.87	30.62±0.91	1463.97±111.86
HDPE.AC.40	19.38±0.18	955.71±68.57	35.58±0.75	2137.63±290.63
HDPE.ACt.20	20.43±0.10	921.03±38.55	31.37±0.42	1616.51±80.92
HDPE.ACt.40	22.55±0.15	1044.63±73.44	37.21±0.38	2745.99±32.66
HDPE.Re	20.66±0.20	697.04±39.65	25.88±0.29	886.86±33.84
HDPE.Re.AC.20	21.13±0.63	806.65±52.68	32.79±0.43	1488.89±35.86
HDPE.Re.AC.40	19.55±0.59	964.53±87.23	38.52±0.37	2524.97±73.66
HDPE.Re.ACt.20	23.91±0.15	904.36±70.68	36.42±0.42	1835.24±33.33
HDPE.Re.ACt.40	21.33±0.39	971.47±114.49	39.42±1.10	2789.79±81.32

Based on the results obtained, the addition of Acacia spp. material into HDPE and HDPE.Re matrices resulted in an increase in Young's modulus; the higher the lignocellulosic content, the higher the modulus. The chemical treatment with 1M NaOH of the fibers led to the removal of the hemicellulose, with a subsequent improvement in the polymer-reinforcement ratio (Dawit, 2020). This improvement can be observed in the 10.50% increase in the flexural modulus, being the values of 2524.97 MPa and 2789.79 MPa for the HDPE.Re.AC.40 and HDPE.Re.ACt.40 formulations, respectively. For the PE virgin matrix, an increase of 28.46% was observed for the same parameter, going from 2137.63 MPa to 2745.99 MPa for the HDPE.AC.40 and HDPE.ACt.40, respectively. Composites with recycled matrix and treated fibers show the highest stress values for the flexural tests. Regarding the bending tests, the maximum strength also increases with the percentage of fibers, taking the maximum values for the treated fibers and HDPE.Re matrix.
This study demonstrates how the recycling of PE and its mixing with fibrous reinforcements of vegetable origin is a striking option for the manufacture of composites due to the small variation of results compared to the non-recycled polymeric matrix reinforced with the same type of fibers.

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ID 229
RECYCLABILITY STUDY OF HDPE NATURAL FIBER COMPOSITE

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ABSTRACT
This study analyzes the effect of recyclability, and more specifically reprocessability, on the mechanical properties, stress and bending, and on the index flow rate of a composite material. The material used has been a composite material made up of a matrix of HPDE and fiber from Ricinus communis (20 and 40% w/w), an invasive species in Macaronesia, obtained from its eradication and control campaigns. Five recycling cycles have been made. The analyzed properties, maximum tension and elastic modulus, in tensile and bending tests, show the degradability of both the fiber and the matrix. This effect is also reflected in the fluidity index. Variations of the same properties between matrix, matrix with untreated and treated (alkaline treatment with 0.1 N NaOH) fiber are also shown.

INTRODUCTION
Currently, production processes are changing from a linear economy model, which causes a series of problems related to pollution and resource depletion, to a circular economy model, which is based on extracting the maximum of resources and where materials can be regenerated or reconverted into new products. [1]. Among the resources that can be exploited as sources of renewable raw materials are those of biological origin, thereby reducing the use of non-renewable raw materials. In this aspect, the use of natural fiber in the manufacture of composites provides a series of benefits, such as greater biodegradability, reduction of greenhouse gases and cost reduction [2].
The beneficial effect of the use of natural fiber for the composites manufacturing is increased by the potential use of plant fiber of invasive species form the different eradication campaigns carried out by different governmental institutions, such as Ricinus communis, present in the Macaronesia`s islands and the subject of this study.
On the other hand, the main advantages of the use of thermoplastics are their recyclability once they have reached the end of their useful life, however, the use of these as matrices in the formulation of composites with natural fibers offers a series of difficulties in their recycling [3]. In this study, HDPE-based composites were formulated with 20 and 40 % w/w Ricinus communis fiber, pretreated with 1M alkaline solution for one hour at room temperature and untreated, using a twin-screw (ThermoFisher Process 11). The composite was used in the manufacture of samples of standardized dimensions to determinate its mechanical properties by means of tensile and flexural tests according to ISO 527-1:2019 and ISO 178:2019. Likewise, the flow index of the material with and without fiber, treated and untreated, was determined using Noselab ATS A-MELT equipment, carrying out the tests according to ISO 1133-1. Once the specimens had been tested, they were crushed using a grinder (3Devo GP20 Hybrid), reshaped and new specimens were obtained, and the new properties and flow index were determined. This process was repeated until 5 cycles were obtained with cycle 0 being the initial cycle.

RESULTS AND CONCLUSIONS

In the flow index tests, HDPE undergoes slight variations, becoming more fluid the final cycles, probably due to chain breakage in the matrix. For the material with Ricinus communis, mainly 20% the flow index decreased in the first cycles and increases again in the last cycles. The fiber substantially modifies the behavior of the separate matrix. The results showed in table 1.

Table1: HDPE and 20% w/w Ricinus communis HDPE composite melt fluid index for cycle 0, 1 and 5.

	Ciclo0	Ciclo1	Ciclo5
HDPE(g/10min)	7.26±0.03	6.93±0.01	7.55±0.13
RC-20(g/10min)	5.07±0.07	4.00±0.05	5.65±0.01

The results of the tensile tests show an increase in the elastic modulus in the first cycles, the material become stiffer, returning to the initial values in the last cycle, denoting degradability of the fiber. However, with respect to the maximum stress, there is a slight increase as the number of cycles increases.

Table2: Tensile strength and elastic modulus for HDPE and 20% w/w composite for cycle 0, 1 and 5.

Tensiletest	Cycle0		Cycle1		Cycle5	
	Strength(MPa)	Elasticmodulus(MPa)	Strength(MPa)	Elasticmodulus(MPa)	Tensilestrength(MPa)	Elasticmodulus(MPa)
HDPE	20.63±0.47	699.16±82.73	19.70±0.19	777.79±43.84	23.02±0.38	788.96±47.32
RC20%w/w	18.63±0.21	629.30±87.04	18.20±0.19	964.02±41.73	20.84±0.20	858.575±34.31
RC40%w/w	16.16±0.14	765.60±102.28	16.23±0.25	1179.83±56.54	19.09±0.85	1006.40±84.63
RCt20%w/w	19.39±0.11	916.49±43.45	19.16±0.10	1002.97±50.83	20.98±0.20	872.80±64.01
RCt40%w/w	20.58±0.24	777.10±41.89	19.36±0.36	1223.10±56.94	21.68±0.41	1074±37.80

In the flexural test, the elastic modulus showed a slight increase when working with 20% fiber and remains constant at 40%. The maximum stress increases slightly with the number of cycles performed.

Table 3: Flexural strength and elastic modulus for HDPE and 20% w/w composite for cycle 0, 1 and 5.

Flexuraltest	Cycle0		Cycle1		Cycle5	
	Strength(MPa)	Elasticmodulus(MPa)	Strength(MPa)	Elasticmodulus(MPa)	Tensilestrength(MPa)	Elasticmodulus(MPa)
HDPE	23.78±0.15	990.46±25.96	25.13±0.31	1019.70±29.68	30.03±0.91	1205.84±17.17
RC20%w/w	30.02±0.25	1618.86±47.17	30.42±0.70	1601.60±63.53	34.69±0.44	1740.62±96.75
RC40%w/w	32.75±1.03	2517.15±163.60	30.99±0.72	2440.172±81.36	35.66±0.68	2309.03±107.91
RCt20%w/w	32.51±0.99	1609.48±79.84	30.63±0.34	1482.75±34.29	34.54±0.74	1696.65±108.21
RCt40%w/w	35.88±0.40	2857.42±53.57	35.97±0.38	2682.04±125.27	39.87±0.84	2625.99±134.62

This study demonstrates the potential recycling of an HDPE composite filled with Ricinus communis particles int at least five steps without significant loss of mechanical properties.

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ID 231
MECHANICAL AND FLAME RETARDANT PROPERTIES OF HYBRID NATURAL FIBER REINFORCED COMPOSITES

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ABSTRACT
The fabrication of long fiber pellets, especially long flax fiber pellets (natural fiber), is complex due to a combination of expertise, specialized equipment, and careful handling of the fibers. This study uses unique extruder – hot pressing – injection molding processing to fabricate long flax fiber (LFF)/short basalt fiber (BF)/ rice husk powder (RHP)/polypropylene (PP) hybrid composite by optimum LFF2/30%BF/6%RHP/PP achieved significantly improved flexural strength, and flexural modulus by 60.02%, and 129.86%, respectively, compared to the LFF reinforced PP composite. Moreover, the hybrid composites demonstrated a high char residual of 19.18%, significantly higher than that of LFF/PP (497.51%). The cone calorimeter test showed that the hybrid thermoplastic composites had improved pHRR and THR with a 57.25% and 13.28% decrease, respectively. These results suggest that hybrid thermoplastic composites offer a promising approach for utilizing LFF in the automotive industry. Hybrid natural fiber/bio-waste powder reinforced thermoplastic composites are tough and lightweight materials that can be used in the automotive industry. They are made from a combination of natural fibers and bio-waste, which makes them sustainable and cost-effective. These composites can improve fuel efficiency and reduce emissions, making them a smart choice for eco-conscious drivers.

INTRODUCTION
The current study development on hybrid natural fiber/bio-waste powder reinforced thermoplastic composites with their ability to provide a sustainable and cost-effective alternative to traditional materials while offering strength and durability. The creation of new inherently flame-retardant green hybrid composites involves a specific emphasis on the distinct processing required for synthesizing LFF/BF/PP-RHP composites. These composites are comprised of a combination of lignin in LFF, high thermal and flame retardant components such as primary silica and metal oxides in BF, and RHP with a substantial weight percentage of silica. The mechanical, thermal, and flame retardancy properties of these composites were studied to achieve multi-target goals for building flame-retardant materials. The unique extruder-hot pressing-injection molding, was employed. Three rounds of mixing were performed to effectively disperse the BF and RHP bio fillers in the basic polymer (Thanh, 2021). To investigate the influence of the reinforcements on the mechanical, and flame retardancy properties of the composites, a universal testing instrument, and a cone calorimeter were utilized. Based on the study's findings, several research opportunities in the fields of environmental issues and thermal and flame retardancy were identified (Rhoda, 2022). Hybrid composites can be used in automobile sub-structural parts due to their lightweight and high strength properties. This makes them a smart choice for applications such as suspension components, door panels, and underbody

parts. Their sustainability also offers an eco-friendly alternative for the automotive industry.

RESULTS AND CONCLUSIONS

Mechanical behavior

Table 1 shows the mechanical (tensile, flexural) results for pure PP, LFF2/PP, LFF2/30BF/PP, and LFF2/30BF/6RHP/PP hybrid composites.

Table 1. Mechanical properties of hybrid composites

Specimens	Mechanical properties			
	Tensile strength (MPa)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)
PP	20.12	0.78	24.08	0.75
LFF2/PP	29.28	2.71	51.92	2.11
LFF2/30BF/PP	42.75	4.15	81.09	3.95
LFF2/30BF/6RHP/PP	46.21	4.54	83.08	4.85

Flame retardant behavior

The results of pHRR, THR is illustrated at Fig. 1. Table 2 shows the cone calorimeter test results for pure PP, LFF2/PP, LFF2/30BF/PP, and LFF2/30BF/6RHP/PP hybrid composites. The graph shows improved flame retardant properties of LFF2/30BF/6RHP/PP hybrid composites by pHRR and THR with 57.25% and 13.28% decreases compared with LFF2/PP composites. Due to the synergistic effect of combining different fibers and fillers, creating a more effective flame retardancy with basalt fibers have good thermal stability and can help delay the ignition and reduce the heat release rate (HRR) of the composite. Rice husk powder, on the other hand, has a high content of silica, which is an effective flame retardant that can form a protective layer on the surface of the composite to prevent the propagation of fire. Additionally, the combination results in improved char formation (RHP), reduced thermal conductivity (BF), and enhanced mechanical properties (can help prevent the formation of cracks and holes in the char layer), leading to a significant reduction in both pHRR and THR compared to LFF2/PP composites.

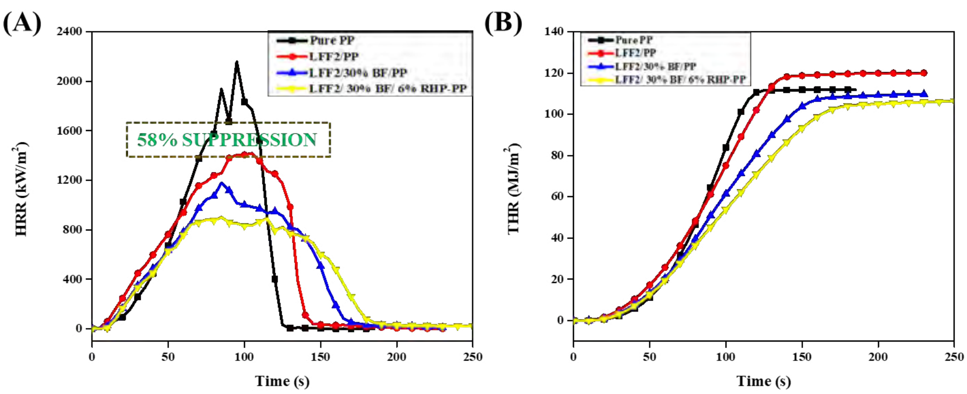

Fig. 1. Cone calorimeter results of PP, LFF2/PP, LFF2/30%BF/PP and LFF2/30%BF/6%RHP-PP hybrid composites.

Table 2. Cone calorimeter data of hybrid composites

Specimens	Cone calorimeter test					
	pHRR (kW/m^2)	THR (MJ/m^2)	TTI (s)	T_{PHRR} (s)	FPI[a] ($s/(kW/m^2)$)	FGI[b] ($(kW/m^2)/s$)
PP	2157.08	111.6	18	95	0.008	22.71
LFF2/PP	1418.44	119.4	13	105	0.009	13.51
LFF2/30BF/PP	1180.08	108.9	16	85	0.014	13.88
LFF2/30BF/6RHP/PP	902.00	105.4	17	85	0.019	10.61

a. FPI = TTI/PHRR.
b. FGI = PHRR/T_{PHRR}

The present study has determined that LFF/BF/PP-RHP hybrid composites can be successfully manufactured through a complex process involving extruder-hot pressing-injection molding. Each stage of the processing improved the mechanical and thermal properties of the composite constituents, including LFF, BF and RHP. Specifically, adding BF reinforcement to LFF/PP-RHP composites (creating LFF/BF/PP-RHP hybrid composites) increased mechanical (tensile and flexural) strength by 57.82% and 60.02%, respectively and thermal stability by producing 18.71% char residue at 700 °C. The cone calorimeter results confirmed that the hybrid composites exhibit flame retardancy, with a pHRR with 139.14% lower than that of pure composites and 5.88% THR of approximately 902 kW/m^2 and 105.4 MJ/m^2, respectively, compared to LFF/PP matrix (1418.44 kW/m^2 and 119.4 MJ/m^2). This study demonstrates the potential of utilizing biowaste materials and specialized processing techniques to manufacture LFF/PP composites with improved mechanical strength and thermal and flame retardancy properties that meet manufacturing standards for sub-structural components in automotive applications.

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ID 232
BIODEGRADABILITY ASSESSMENT OF NATURAL FIBER-PLA COMPOSITES

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ABSTRACT
This study assessed the modifications in the properties of compressed polylactic acid (PLA) composites obtained with 20% of two different natural fibers, mainly focusing on the alterations due to a bio-disintegration process. Thermal properties (melting temperature and crystallinity degree), morphology (via optical microscopy), and chemical changes (by Fourier Transformed Infrared spectroscopy) were studied. The results confirmed that PLA based biocomposites with all tested natural fibers are biodegradable. The behavior of the materials under marine conditions was also evaluated, although long-duration tests need to be carried out.

INTRODUCTION
Biodegradable polymers have gained much attention around the world in recent years as a more environmentally friendly alternative to conventional plastics. Among biodegradable polymers, PLA is a versatile polymer made from renewable agricultural raw materials which offers a number of benefits over the other biopolymers. On the other hand, biodegradable polymers can be reinforced with fibers, and particularly natural fibers, in order to create environmentally friendly and biodegradable composites (Ilyas, 2022). The residual biomass generated from campaigns to eradicate and control invasive plant species can be considered as an interesting source of natural fibers. Such species represent a threat to the preservation of ecosystems, with their presence especially dangerous in areas with high biodiversity.
In this study, natural fiber obtained from 2 different invasive plant species present in Macaronesia (Ricinus communis (RC) and Acacia spp (AC)) were used at a 20 % w/w for obtaining PLA (PLA L105 5% nucleant) based biocomposites. The composites were characterized to determine their mechanical and thermal properties. Besides, the aerobic biodegradation of these biocomposites under simulated composting conditions has been evaluated, as well as their biodegradability in marine environment. Various analytical techniques like differential scanning calorimetry (DSC), FTIR, TGA, volatiles determination and microscope observations were used for the evaluation of the biodegradation. The mechanical properties of samples after the biodegradability under marine conditions assays were also determined.

RESULTS AND CONCLUSIONS
The results from the mechanical characterization of original compression moulded plates with PLA L105 and 5% of nucleant as matrix and fiber (20 % w/w) are shown in Table 1.

Table1 Mechanical properties of natural fiber reinforced PLA based biocomposites

Formultion	Tensiletest		Flexuraltest	
	σmax(MPa)	E (MPa)	σmax(MPa)	E (MPa)
PLA.5N	9.23±1.63	454.54±53.20	20.56±1.27	3827.59±136.58
PLA.5N.AC.20	6.24±0.78	575.53±58.53	15.89±1.89	3362.26±233.71
PLA.5N.RC.20	4.92±1.14	638.04±76.43	14.07±1.79	3734.35±252.64

As can be seen, the introduction of any fiber in PLA resulted in a reduction of maximum tensile and flexural stress; a rigidization of PLA properties was also observed (tensile modulus was increased).
The degree of disintegration of plastic materials when exposed to a laboratory-scale composting environment was evaluated according to UNE-EN ISO 20200:2015. The disintegration of the material was evident, as the samples were broken from week one and the recuperation of the material from composting material at the end of the assay was difficult. After 45 days of assay the degree of disintegration of the samples was higher than 25% for all the species. A mineralization effect was observed in volatile content values, which decreased after disintegration test. The degradation of the samples was further confirmed by the reduction of the melting point and crystallization temperature, while FTIR analysis also showed the degradation of the matrix, with the increase in the intensity of bands related to carbonyl and ester groups.
On the other hand, the determination of aerobic biodegradation of materials in a seawater/sediment interface (sublittoral zone) was evaluated according to UNE-EN ISO 19679. Moreover, the degradation of plastic samples in eulittoral zones was evaluated according to (Tosin, 2012). In both tests, the mechanical properties of the different composites were reduced significantly after the three months of the test. Optical microscope observation revealed some small cracks on the surface of the specimens. In all cases weight loss was lower than 1%. FTIR and DSC analysis did not show significant alterations in the materials. In the case of the test in a seawater/sediment interface, the analysis of evolved carbon dioxide was evaluated by titulation of a NaOH solution introduced in the assay recipient, which allowed to determine the extent of the biodegradation. In all the samples, the biodegradation due to microorganisms was negligible (less than 0.6 % in all species), so the changes were due to just a chemical/physical degradation.
The composting of biodegradable materials is becoming an alternative option to conventional disposal methods such as landfills or incineration. The results obtained in this study confirmed that PLA based biocomposites with natural fiber are biodegradable. In the case of marine environment, the duration of the tests was not enough, and further tests should be performed to analyze their behaviour under these conditions.

ACKNOWLEDGMENTS

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ID 234

EFFECT OF IGNIMBRITE POWDER ON THE MECHANICAL AND THERMAL PROPERTIES OF ROTOMOLDED POLYPROPYLENE COMPOSITES

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ABSTRACT

This work assesses the possibility of producing composite parts by rotational molding with the blend of polypropylene and silica-based dust, produced as a waste in the extraction of the volcanic stone in the quarries of Gran Canaria island. After obtaining the composite with 5, 10, 20 and 30% w/w loading, by simple dry blending, the parts were characterized to determine the mechanical (tensile, flexural and impact strength) and thermal (by thermogravimetry and differential scanning calorimetry) properties. The particulate material and the polymer-reinforcement interaction were also studied by microscopy.

INTRODUCTION

Worldwide, polypropylene (PP) is one of the most widely used plastic polymers, mainly in the packaging, automotive, textile and daily-use items. PP is characterized by its hardness, flexibility, and high durability. In contrast, the overuse of such materials is causing environmental pollution due to the accumulation and lack of an appropriate waste management. As a result of this problem, there is a need to investigate reinforcements of the polymeric material that allow reducing the amount of virgin plastic used without losing its properties and forming an environmentally friendly composite.

The use of natural fibers as an alternative to polymeric reinforcement in the manufacture of composites, specifically fibers of mineral origin such as basalt, marble, or plaster, have not been as widely studied as other fibers, such as lignocellulosic ones. However, these materials have excellent mechanical properties and high thermal resistance (1). Most of these studies focus on the use of fibers, obtained after processing of mineral materials, and not as much on the direct use of powdered mineral material, found naturally in the environment or generated as residues in obtaining stone in mines and quarries (2). In the Canary Islands, the high-quality stone products, with volcanic origin, is of great economic, ethnographic, and historic importance. During the different operations performed on the stone, a high volume of residues, currently without any use, is produced. This waste (residual ignimbrite dust) is the raw material for the production of PP-based composites, using 5, 10, 20 and 30% w/w loadings. The composite materials were obtained by rotational molding and thermally characterized by differential scanning calorimetry (DSC) and thermogravimetry (TGA). The mechanical properties were determined by tensile, bending and impact tests. Finally, microscopic observations were made for the evaluation of the particulate material and the study of the polymer-reinforcement interaction.

RESULTS AND CONCLUSIONS

In the production of composites by rotational molding, the problem of agglomeration of fillers and reinforcing materials must be considered (3). For this reason, prior to obtaining the composite material, ignimbrite powder was sieved in sizes of 250μm, 125μm, 75μm and <75μm and combined with PP, in powder form, varying the amount of reinforcement, 5, 10, 20 and 30% w/w. Dry-blending was used in this first stage of the research. Several preliminary cubical pieces of PP-stone dust composite were successfully obtained, generally showing good processability by rotational molding. Figure 1 shows the appearance of some of the parts obtained.

a) b)

Figure 1. Some composites obtained by rotational molding of PP with: **a)** 0, 5, 10, 20 and 30% w/w ignimbrite dust reinforcement and **b)** 5% w/w ignimbrite dust reinforcement.

Figure 2. Distribution of ignimbrite powder in the composite parts.

This study is a first approach to the recovery of residual dust obtained from the extraction of stones in the quarries of the island of Gran Canaria as there are no studies focused on the use of welded ignimbrite in obtaining composite materials through the rotational molding process. This invites to continue with the valorisation of this industrial residue to obtain more sustainable new materials, while also getting improved properties and functionalities.

ACKNOWLEDGMENTS

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ID 235
EXTRACTION OF FIBERS FROM BANANA PLANT WASTE: A COMPARATIVE STUDY

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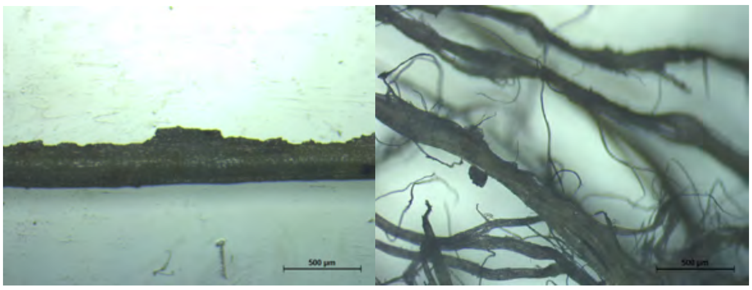
ABSTRACT
This work aims to extract fibers from banana plant waste using different extraction processes and treatments. The fibers were manually extracted and then treated with sodium hydroxide (NaOH). Another method used for fiber extraction was water maceration for 21 days followed by manual extraction. All developed samples were characterized by Optical Microscopy (OM) and Fourier Transform Infrared Spectroscopy with an Attenuated Total Reflectance (ATR–FTIR) accessory. FESEM analysis was also performed to evaluate the fibers morphology.

INTRODUCTION
Currently, the use of agricultural wastes as sources of natural cellulosic fibers has become urgent, given the demand for natural and synthetic fibers (Fonseca-Pinheiro et al., 2022). Natural fibers have gained increasing relevance in the textile area due to their excellent characteristics, including biodegradability, low density, low cost, less dangerous manufacturing processes, good electrical resistance, sound insulation, among others (Ferreira et al., 2019). Banana fiber is of great interest, given that the banana is one of the most consumed fruits worldwide and therefore generates large amounts of waste (Balda et al., 2021). Thus, one of the most promising approaches may consist of extracting fibers from these wastes (pseudostems and leaves) for the development of new sustainable materials. In this work, banana fibers were obtained using two different methods, a chemical method (mercerization) and a biological method (maceration), both combined with manual extraction. All samples were evaluated by Optical Microscopy, ATR–FTIR and FESEM.

RESULTS AND CONCLUSIONS
Four different banana plant leaves (A, B, C and D) were manually extracted. For the mercerization, the previously decorticated fibers were subjected to 15% NaOH treatment for 1 hour at 45 ºC. The OM images of the obtained fibers as well as their diameters are shown in Fig.1 and Table 1, respectively. Before the mercerization, the diameters of the fibers are higher, when compared to the ones after this treatment, which can be explained by the higher percentage of binding material.

a)



b)

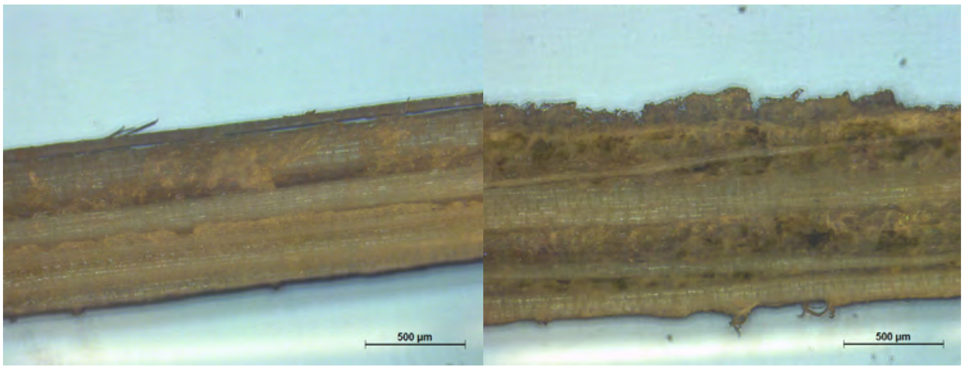
Fig. 1 OM images of the samples a) decorticated and b) decorticated and treated with NaOH

Table 1 Range of diameters of the samples A, B, C and D before and after mercerization

Sample	Before mercerization (µm)	After mercerization (µm)
A	198,39 – 255,28	11,61 – 167,28
B	59,14 – 87,29	45,06 – 83,35
C	135,07 – 158,77	15,43 – 21,61
D	–	13,75 – 171,07

The maceration of the decorticated fibers was made by immersing them in water during 7 and 21 days. This method is way more sustainable than the mercerization. However, and from the OM images and the measurements of the fibers' diameters (Fig. 2 and Table 2), the maceration led to the obtention of fibers with higher diameters, which is probably related to percentage of non-cellulosic components. In this way, the use of a higher and controlled temperature in this method should be tested.

a)



b)

Fig. 2 OM images of samples with a) 7-day maceration and b) 21-day maceration

Table 2 Range of diameters of the samples with 7 and 21 days of maceration

Sample	7 days of maceration (µm)	21 days of maceration (µm)
A	233,34 – 254,77	239,79 – 270,46
B	303,11 – 332,39	208,15 – 222,01
C	400,63 – 453,35	678,59 – 679,95

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ID 236

ANALYSIS OF THE EFFECT OF EMBROIDERY DENSITY ON THE STRENGTH OF THE COMPOSITE CONTAINING TECHNICAL EMBROIDERY MADE OF FLAX FIBERS AS REINFORCEMENT

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ABSTRACT

This work presents the results of research on the effect of embroidery density on the strength of the composite, which contains technical embroidery made of flax fibers as a reinforcement. The embroidery was made on a ZSK type JCZA 0109-550 computer embroidery machine. Then scans were made using computed tomography to assess the degree of damage to the packets by the embroidery needle. These results were compared with the results of strength tests of composites, which contained technical embroidery as a reinforcement.

INTRODUCTION

Currently, companies producing composites are facing the problem of waste management (Chegdani, 2019). The answer to this may be composites containing technical embroidery made of flax fibers as reinforcement. Thanks to the use of TFD (Tailored Fibre Placement) technology, the amount of waste generated during production is reduced to a minimum (Wright, 2019). This technology is therefore in line with the idea of sustainable development .

The main purpose of the research is to verify whether the increased density of the embroidery reduces its strength.

The embroidery was made with the use of a ZSK type JCZA 0109-550 computer embroidery machine. It was made of Safilin's flax roving with the linear mass 400 tex. It was fastened with Gunold's polyamide monofilament with linear mass of 11 tex. 3 types of embroidery were made, differing in zig-zag stitch length: 2 mm, 4 mm, and 8 mm. The width of stitch was 1.2 mm.

Based on previous studies, it was observed that the tensile strength of the composite containing the embroidery varies depending on the density of the embroidery itself (Poniecka, 2022). For this reason, it was decided to perform a CT scan to analyze the impact of needle punctures on the structure of the embroidery. Micro-CT scanning was performed using SkyScan 1272. 3D reconstruction were obtained using NRecon 1.7.4.2 and CTvox 3.3.0 r1403 software made by Bruker. Geometrical parameters were calculated using CTAn 1.17.7.2+ and Data Viewer 1.5.6.2 software made by Bruker.

RESULTS AND CONCLUSIONS

Figure 1 presents the results of tensile strength tests of composites containing different types of technical embroidery made of flax fibers as reinforcement.

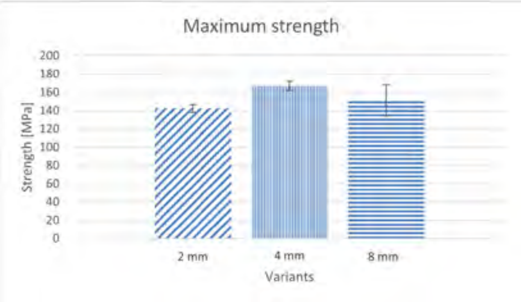

Fig.1 Strength of produced composites

It can be clearly seen that the strength of the composite containing embroidery with a stitch length of 4 mm is the highest and the one with 2 mm is the lowest.

Figures 2 and 3 show tests performed with the use of a computed tomography. In the marked areas, damage caused when the embroidery needle passes through the embroidery structure can be seen.

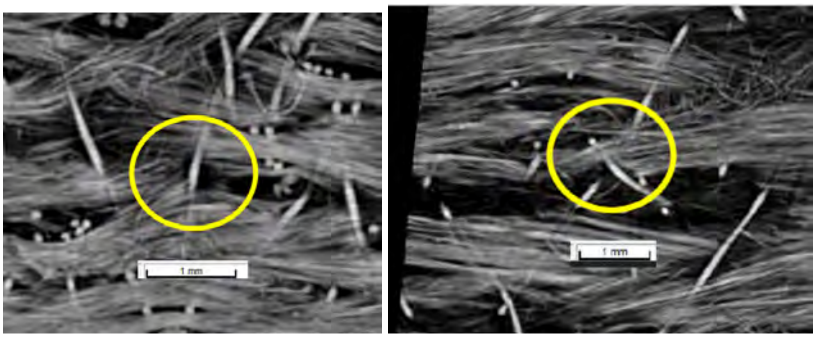

Fig.2-3 View from computed tomography, 2 mm embroidery

Based on the tests carried out, it can be concluded that in some places the continuity of the flax roving is interrupted, which makes the produced composite characterized by weaker mechanical properties. Due to the highest density of the embroidery, this feature reduces the strength of the embroidery with the stitch length of 2 mm the most.

ACKNOWLEDGMENTS

Composites were produced and strength tests were carried out at The Department of Mechanical Engineering of the University of Zaragoza. Computed tomography was made at Institute of Material Science of Textiles and Polymer Composites of the Lodz University of Technology.

The research was carried out as part of the project "Sustainable Industrial Design of Textile Structures for Composites" which is co-funded by the European Union. Grant Agreement no. 101079009 Call: HORIZON-WIDERA-2021-ACCESS-03/Twinning

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ID 237
MODULATING TEXTILE PROPERTIES: INTRODUCING A NOVEL CIRCULAR CELLULOSE-BASED BINDER FOR PRINTING TEXTILE FINISHES

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ABSTRACT
This research shows how regenerated cellulose obtained from waste post-consumer textiles was introduced for the first time as a print binder in textile screen printing. The resulting properties are compared to three types of petroleum-based textile processes that hinder a cellulose-based textile material's circularity: puff binder, plastisol, and thermoforming. The results demonstrate significant reduction in temperature for textile finishing, as well as mono- material cellulose-based processes for fabric manipulation, textile print and textile shape on cellulose-based textiles suitable for a circular bioeconomy context.

INTRODUCTION
Regeneration technologies for cellulose-based textiles are heralded as one of the solutions in achieving a circular economy for textiles. These chemical recycling processes are often focused on the like-for-like replacement of environmentally impactful fibres with waste- derived fibres. Low value regenerated cellulose materials that are not suitable for the fibre process remain unconsidered in the commercial drive to up-scale circular chemical recycling technologies (Lou and Hamouda, 2014, p.122). Within this technological landscape, synthetic binders conventionally used in textile finishing techniques such as printing are a barrier to a material's circularity of bio-derived cellulose-based textiles.
Screen printing is the most commonly used process for printing textiles worldwide with more than 90% of printing in textiles conducted with this technology (Sinclair, 2015). This work demonstrates how design practice at the raw material stage of regenerated cellulose materials resulted in introducing regenerated cellulose for the first time as a binder in textile screen printing techniques. The process resulted from a Materials Driven Textile Design (MDTD) methodology (Ribul et al., 2021) and is one of four processes validating the methodology within a framework of future materials circularity for regenerated textiles (Ribul, 2021). Printing a cellulose dissolution onto fabric modulated textile properties in surface manipulation, finish and shape suitable for a circular bioeconomy context of textiles. The mono-material, cellulose-based textile finishing technique can alter a fabric surface to varying degrees and is compared to existing approaches using puff binders, plastisol and thermoforming, which all rely on petroleum-based materials and processes.

RESULTS AND CONCLUSIONS
The haptic and visual qualities in the printed textiles resulting from the technique demonstrate the possibility for a replacement of toxic, petroleum-based materials in puff, plastisol and thermoforming processes. The cellulose-based technique requires much lower process temperatures shown in Table 1. Different print line thickness can alter a fabric surface to

varying degrees as shown in Figure 1.

Table 1 Textile print technique process temperatures

Synthetic binder/material	Process temperature	Cellulose-based binder	Process temperature
Puff binder	160˚C – 170˚C	Fabric manipulation	80˚C
Plastisol	165˚C	Textile finish	80˚C
Thermoforming	190˚C	Textile shape	80˚C

Fig.1 Textile screen print with Screen print with 4.2mm line thickness generates a strong puffed surface effect on the fabric with light pleats at the end of the print line.

The research identified a novel cellulose-binder for textile finishing that uses one hundred per cent waste, non-toxic and chemically recyclable materials, resulting in mono-material textiles with similar visual and haptic properties to these three synthetic processes.

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ID 240
HEMP FOR GARMENT TEXTILE IN NORMANDY: A FULL VALUE CHAIN IS BORN

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ABSTRACT
This work presents the elements to demonstrate the interest of establishing a whole textile hemp industrial value chain alongside the flax one to fulfil the always increasing demand of long line fibres for the manufacturing of fine garment textiles. It is particularly shown that the value chain is now reaching a sufficient degree of maturity to become industrial, with high hemp straw and fibre yields associated to high quality fibres that can guarantee farmers competitive incomes, higher than the ones of traditional crops.

INTRODUCTION
With the development of new textile generations which are more respectful of the environment, the flax value chain is under always increasing demand, but the production territory is globally saturated. It is also important to mention that flax is particularly sensitive to drought (Melleli et al.) and recent dry springs and summers brought worries in the flax community. Hemp, is very much less sensitive to drought and is a very good head of rotation for following crops, particularly for organic farming. It could be implemented within the flax rotation to increase the production of high-quality long line fibres with the objective to use as much as possible the experience and the machinery of the flax value chain.
In the last 6 years, the LCBio cluster coordinated trials with volunteer farmers to reach the objective to recreate a textile value chain locally in Normandy. From 2020 larger field trials were performed with harvesting machines not adapted to industrial conditions. However, these trials were very interesting to establish the conditions of hemp growth and start establishing the right combination of hemp cultivars/time of harvest/time of dew retting. This study reported by Pinsard et al. (2023) also dealt with the importance of using adapted to hemp scutching process parameters at industrial scale (Teillage Nord de Caen) that were previously tested at the laboratory scale by Grégoire et al. (2021).
In 2021 and at a larger scale in 2022, a new harvesting machine, capable to work at an industrial scale was used to complete the value chain with the objective to demonstrate the full potential of hemp to fulfil the demand of long line scutched fibres suitable for fine yarn manufacturing using wet spinning process.

RESULTS AND CONCLUSIONS
The results from the 2021 and 2022 trials using the full chain of equipment (from the field to the extraction of fibres as illustrated in Fig.1) will be presented.

Fig.1: (a): Industrial hemp dedicated harvesting machine, **(b)** Hemp dew retted straw at the scutching input, **(c)** Industrial scutching machine, **(d)** Scutched long hemp fibres, **(e)** Labscale hackling machine, **(f)** Hackled hemp fibres, **(g)** histological cross section of a hemp stem

Particularly, a focus will be given on the quality of the produced hemp fibres such as the thickness of technical hackled fibres, their strength, their degree of maturity etc.…
Technical economical data, consisting of straw yields, long fibre, tow fibres, shive yields as well as the cost of production will also be given with the objective to demonstrate that hemp is an advantageous crop to grow for farmers, with incomes that are positioned on the upper part of their rotation.

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ID 241
SERVICE LIFE PREDICTION OF MODERN MATERIALS

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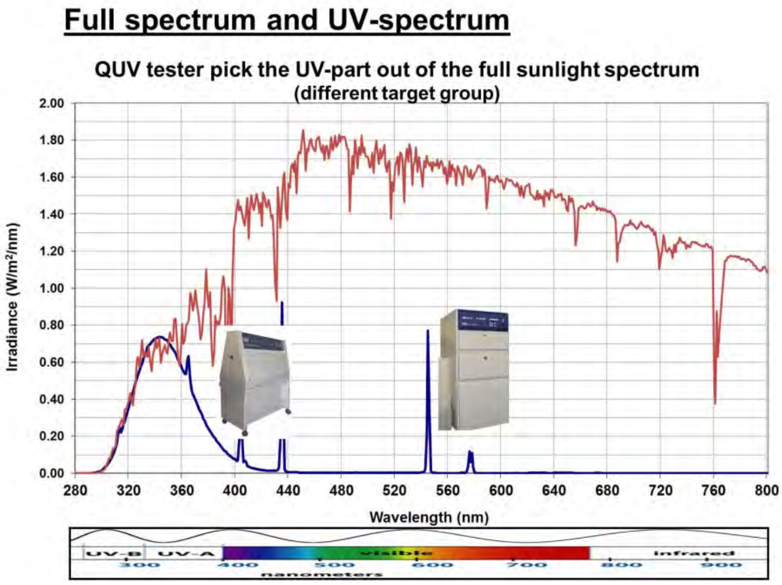
ABSTRACT
This presentation compares the effect of two different standardized test methods (ISO 4892-2 and ISO 4892-3) for the artificial and accelerated ageing of an innovative epoxy/fibre composite provided by DITF. For testing in accordance with ISO 4892-2 a full spectrum Xenon arc tester (Q-SUN Xe-3) is used and testing according to ISO 4892-3 is performed in a fluorescent UV tester (QUV).
For measuring the effect of artificial ageing and for the comparison of the two selected standard test protocols specific mechanical properties, e.g. breaking strength and water absorption of the epoxy/fibre composite has been determined.
Also, this presentation answers frequently asked questions about the different tester technologies and how they can help to predict service life time of an innovative material.

INTRODUCTION
Every year, a huge variety of innovative new materials were presented and very often they are expected to be long-lasting over years or decades. But surprisingly, most of these new materials have never been tested for their stability over time. Durability and service life prediction are the key words if you want to enter today´s global markets with your new products. Therefore, it is important to use time- and moneysaving accelerated test methods to determine the effective service life time of your new product.
Two different internationally accepted test protocols are dominating all other test scenarios for plastic materials, coatings and laminates, ISO 4892-2 and ISO 4892-3. Both test protocols are intended to simulate the relevant weathering factors such as simulated sun light (“solar radiation”), UV radiation, heat, humidity, dew and rain.
Despite the fact, that both test protocols are intended to determine service life time of a polymeric matrix they are using fundamentally different tester technologies, Q-SUN full-spectrum testers and QUV fluorescent UV testers.
Q-Sun Xenon-arc testers are designed to simulate the entire spectral range of natural solar radiation and are known to be the best available technique if the full solar spectrum is required. In a QUV tester, only the short UV range of the natural solar radiation is simulated with fluorescent UV radiation source. For both tester types the ISO 4892 series describes the general requirements and offers a variety of standardized test methods to simulate indoor and/or outdoor use of materials.



RESULTS AND CONCLUSIONS

Xenon arc testers are designed as insulated, closed cabinets to allow full humidity control. Depending on the sample color, the infrared radiation heats up the samples significantly above ambient temperature and condensation (to simulate dew formation on material during night) is not possible. Nevertheless, the filtered Xenon light technology is the best available approach to simulate the full sunlight spectrum.

The open-back design of QUV testers allows a condensation step to simulate dew formation on the material during night time and today´s Fluorescent UV tubes are the best possible simulation of the UV area of natural sunlight. The UV part of the sunlight is the most powerful area with the highest photon energy which is responsible for most material degradation processes of polymeric materials, including wool, cotton and other fibers as natural polymers.

Both test protocols show significant impact on the breaking strength of the epoxy/fibre composite. This loss of material strength can be interpreted as material degradation of the cross-linked polymeric epoxy matrix based on the exposure to UV radiation (300-400nm). Infrared radiation which is part of the Q-SUN technology only does not affect the epoxy/fibre resin significantly, as long as it does not exceed the typical natural level found outdoors.

After the polymeric matrix is broken and open, the wood-based fibre part of the composite is affected and degraded by the ultraviolet radiation. While the water absorption is not measurable prior to the UV exposure the material properties and dimension significantly change at the end of test.

Both test protocols, ISO 4892-2 for Xenon arc and ISO 4892-3 for fluorescent UV allow to determine timeline for material degradation which can be used for service life prediction. For both, the effect is based on the UV exposure but it can be seen that the QUV fluorescent UV technology is faster compared to Q-SUN Xenon arc technology.

ID 242
COTTONISATION OF BY-PRODUCT FIBRES FOR TEXTILE APPLICATIONS

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ABSTRACT
This work compares the yarns obtained after a cottonisation process of different by-products from agro-resources. Different batches of fibres from hemp and linseed flax plants were analysed. Cottonised treatments were carried out at lab and semi-industrial scale. Blends of the by-product material and cotton or viscose were made up to 80/20 ratio. Finally, 60 tex yarns of this ratio have been spun and chemical, morphological, mechanical and knittability tests on fibres and yarns were performed. It demonstrated that the yarns, manufactured using rotor spinning can be used for garment textile purposes.

INTRODUCTION
Residual material from industrial process such as textile or seed oil manufacturing are present in huge quantities and are nowadays transformed in low added-value products. Their fibre high lignin content or their morphology make it difficult to spin through conventional dry or wet spinning techniques. Different process combinations have been tried, such as enzymatic treatment with rotor spinning, allowing spinning blends of residual hemp and cotton but only with low content of hemp (Kozłowski et al. 2013).

To valorize those fibers as high-valuable textile grade materials, a cottonisation process including mechanical, alkaline and oxidizing treatments have been used. It allows reducing the non-cellulosic materials content and keeping a high content of the specified material in the final blend.

Four different types of fibres were analysed: unretted hemp fibres from oil seed industry, retted long hemp fibres, retted medium length hemp fibres and mid retted linseed flax fibres.

At laboratory scale, samples were chemically cottonised and three different blend ratios of the by-product material (BM) with cotton were achieved: 40/60 BM/cotton, 50/50 BM/cotton and 80/20 BM/cotton. Finally, yarns were spun using an open-end rotor spinning machine and tested mechanically. The treated fibers have been chemically characterized to determine the influence of the cottonisation treatment on their final properties according to Tappi Standard T 222 om-2015.

The process was then carried out at a semi-industrial scale processing 5 kg of fibres and the blends were done using viscose to measure the real final content by dissolution of the viscose (ISO 1833). Fibres were spun to obtain yarns with sufficient strength to produce knitted fabrics.

RESULTS AND CONCLUSIONS
A decrease in the lignin content was observed after cottonisation process and the measure of the degree of polymerization shows that the treatments do not change the nature of cellulosic materials. Lignin was removed from 19% to 50% depending on the batch and the treatment. Examples of final counts of the spun yarns from two different batches are presented in the Table 1.

Table 1: Linear mass of spun yarns

Blends	Counts obtained in tex
40/60 linseed flax/cotton	40
50/50 linseed flax/cotton	42
80/20 linseed flax/cotton	66
40/60 retted long hemp/cotton	36
50/50 retted long hemp/cotton	56
80/20 retted long hemp/cotton	57

This study shows that by-products materials can be used for textile grade application. Fine yarns with up to 80% of side-product materials can be spun using a cottonisation process and contribute to a circular economy. The different obtained yarns associated to their morphological and mechanical characteristics will be presented. Their processability using a labscale knitting process will also be presented and their interest for the textile industry discussed.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the funding by the Agence de l'Eau de Seine-Normadie, the Mechanics, Energetics, Civil Engineering and Processes (MEGeP) doctoral school and the Laboratoire Génie de Production.

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ID 243
POTENTIALITIES OF AGAVE SISALANA FIBERS IN THE DESIGN OF BIO-INSPIRED MATERIALS AND ARTIFACTS.

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ABSTRACT
A bioinspired design approach is believed to favor research and eco-performance metrics over creating bioinspired forms and materials. This article explores agave's lightness and strength strategies. A bioinspired process was developed considering the following: biologically based study through microscopic observation; parametric modeling applied to bioinspired surf artifacts and generation of optimized study models, aided by digital fabrication processes and 3D printing (CNC and SLS); and the development of biologically-based composites that combined agave fiber with castor bean resin, with subsequent verification of mechanical properties in flexion and traction tests, where it was verified that the insertion of 20% of 600 μm agave fibers in a castor bean resin matrix increases its resistance in terms of maximum tension. In this way, it was possible to carry out emulations of lightness and strength strategies in structures and composite materials with application in bioinspired surf artifacts.

INTRODUCTION
Through biomimicry, it is possible to learning how to emulate forms, materials and processes based on natural elements to create more sustainable designs (Benyus, 1997; Baumeister, 2014; Bianciardia, A. *et al*. 2023). Biological structures provide a wide range of properties with minimal use and flow of materials and energy, and generate recyclable products (Byrne *et al*. 2018). Computational strategies and principles of natural systems are a design approach that is closely related to biology and digital fabrication. The use of parametric design instruments allows the development of biomimetic designs through understanding the shape and material of biological structures, as complex interrelationships that are incorporated and explored in the generation of sustainable artifacts (Ahmar *et al.* 2013).
In this sense, the work carried out a research about Agave in terms of inspiration in the cell structure and exploration of the fiber as a raw material. The emulation of the Agave strategy was carried out with parametric design, and algorithms were developed with application in bioinspired surfing artifacts as a case study. In the development of the code, parameters for selecting optimized models and analysis of the resistance of the structures of the artifacts were incorporated. In research with bioinspired materials, the potential of Agave fibers associated with bicomponent resin matrices derived from castor oil was explored in the development of 5 biobased composites, with subsequent verification of mechanical properties in bending and tensile tests. Different compositions were developed using two types of castor bean resin: rigid elastomer resin (Resina 3002); and rigid expansive polyurethane resin (Respan). Agave fibers of 106 μm and 600 μm were selected to reinforce the matrixes. Five different samples compositions were defined: Resina 3002 with 20% 106 μm fibers; Respan with 20% 106 μm fibers; Respan with 10% 600 μm fibers; Respan with 20% 600 μm fibers; and Respan with 20% fiber mix (106 μm; 250 μm; 425 μm; 600 μm, with

25% of each one). Samples of natural Agave; paulownia wood; Resina 3002 100%; and Respan 100% were ,also produced for comparison purposes. Five specimens of each sample were tested on mechanical tensile and bending tests were performed in a MultiTest 2.5 - dV Mecmesin single-column universal test equipment with a 2.5 kN load cell, using VectorPro™ software to collect load, displacement and time values.

RESULTS AND CONCLUSIONS

Several outputs were generated with the emulation of the Agave strategy of the generative possibilities of the algorithm applied to surf artifacts (bioinspired internal structure of hollow construction surfboard and fins - figure 1). The surfboard design was generated with Shape3D software, and then the slices were exported to Grasshopper to apply the bioinspired parametric design strategy. The models were created by evolutionary algorithm for selection of optimized models. Prototyping equipment was used, including CNC Router model RMC 3000 Plus - Rhino Máquinas, as well as an Elegoo Mars 2 Pro (SLA) resin printer to carry out experiments.

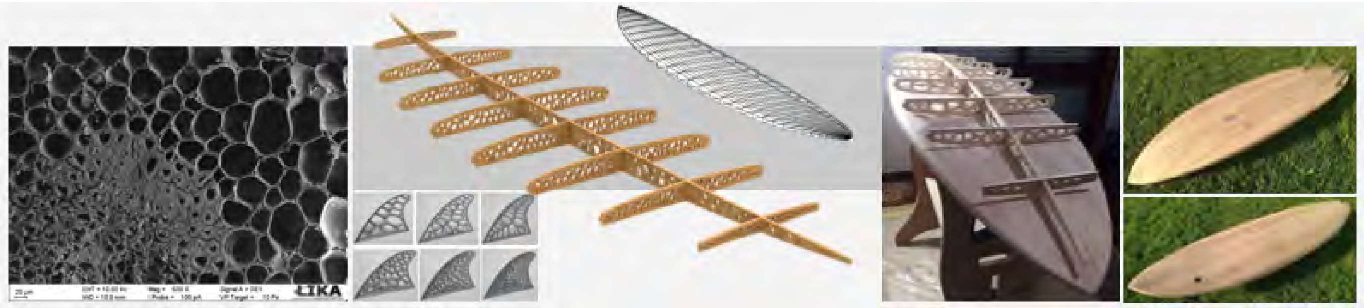

Fig.1 Agave SEM Bioinspiration and outputs surfing artifacts.

Regarding the performance of bioinspired materials, in terms of maximum tension, it was found that the behavior of Agave is similar to that of a polyurethane foam, perhaps due to the internal cavities present in both samples. By inserting 20% of agave fibers in the castor resin matrices, an improvement in the sample's resistance was noticed.

Starting from nature's strategies and following a methodological process of biomimetics using engineering techniques, it was possible to emulate and develop bioinspired systems. The bio-inspired approach to create lightweight resistant artifacts with sustainable materials helps researchers to emulate other strategies from natural systems and biology studies and apply them to develop different industrial projects.

ACKNOWLEDGMENTS

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ID 245
PLANT FIBER TRANSVERSE PROPERTY IDENTIFICATION – INVESTIGATING THE INFLUENCE OF FIBER GEOMETRY THROUGH FINITE ELEMENT ANALYSIS

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ABSTRACT
The Single Fiber Transverse Compression Test (SFTCT) represents an established method to identify the transverse elasticity of fibers. The elastic modulus is identified by inverse methods using analytical models. These models represent the fiber as a perfect cylinder, a geometry that is far from the complex morphology found in plant fibers. This work, compares the results of finite element models of complex fiber geometries to analytical model predictions, providing the boundaries in which analytical models can be used accurately.

INTRODUCTION
To widen the adoption of plant fibers in a larger domain of engineering applications, the characterization of their mechanical properties is critical. While the longitudinal mechanical properties of plant fibers have been extensively studied their transverse properties are essentially unknown. The SFTCT can be used to identify these properties, starting with the transverse elastic modulus ET. However, fibers exhibit non-linear behavior when transversely compressed, even if they are purely elastic. For this reason, direct measurements of ET are not possible. Analytical models are used instead, to provide it through inverse identification.
These models represent the fiber as right circular cylinder. Plant fibers however are characterized by complex geometries that do not match this ideal geometric representation. The limited space inside the plant stems leads to fibers that grow into elliptical or polygonal sections with flatter regions where they are in contact with each other. Plant fibers also possess a central cavity, the lumen. A question can thus be raised: given the morphological complexity of plant fibers, can analytical models with simple geometric representations be used to identify their apparent ET?
To answer this question the transverse compression of plant fibers cross sections is simulated using 2D finite element (FE) models. The impact of their geometric features on the fiber's compressive behavior is discussed. Using, force and displacement data from these FE simulations an apparent transverse elastic modulus is identified by inverse method using the analytical model. By comparing the identified apparent modulus with one imposed in the FE simulation, the error made on ET by the analytical model is evaluated. Ideal representations of the main plant fiber geometric characteristics (lumen, ellipticity, flatness) are first modeled, to study the influence of each geometric feature independently (see Fig. 1 a). Complex fiber cross sections, extracted from microscopy images of plant stems, are then studied, providing a more realistic response resulting from the interaction between numerous geometric features (see Fig. 1 b). For these

complex geometries, neural networks are also trained to predict the analytical model's identification ability.

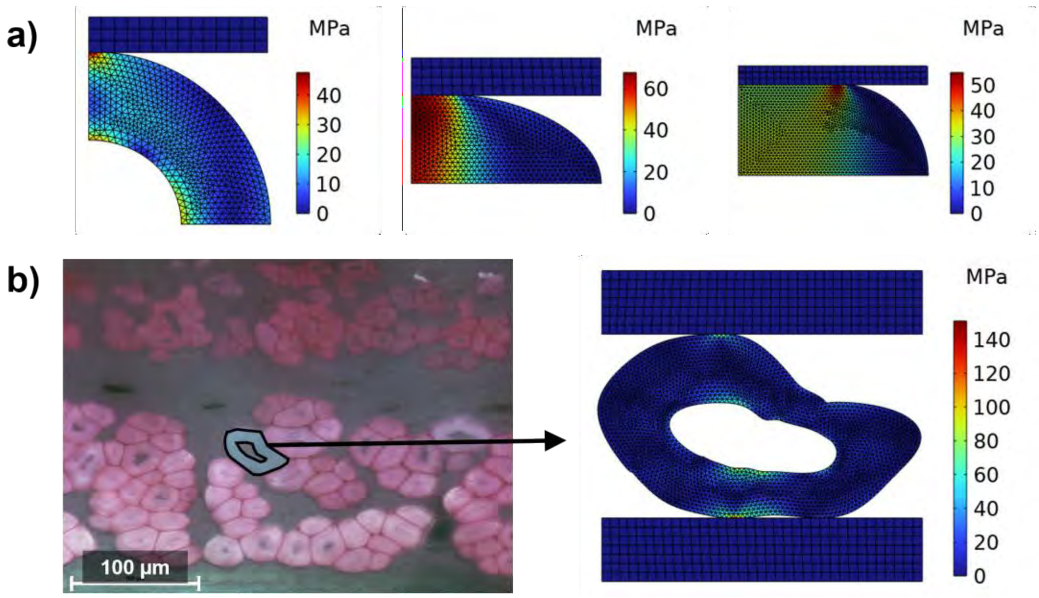

Fig.1 a) simulation of ideal geometric representation **b)** extraction and simulation of complex fiber geometry

RESULTS AND CONCLUSIONS

Concerning the study of the ideal geometric representations, the lumen is shown to lead to an underestimation of ET by the analytical model, while for elliptical and flat section geometries, an overestimation of ET occurs. For fiber geometries issued from microscopy images (20 fibers were extracted in total), the analytical model made on average an underestimation of 7% on ET with a standard deviation of 33%. Neural networks trained on these simulation results predicted the error made by the analytical model with an accuracy of 2.5%.

Overall, given the complexity of the plant fiber geometry, analytical models are shown to identify the apparent transverse elastic modulus of plant fibers with deviations that are within those typically found on the characterization of other plant fiber properties (Baley, 2014). Finite element models of complex transverse compression are also developed, along with neural networks trained on their results. In the future, these simulations and networks could eventually replace analytical models, to provide even more accurate ET identification abilities.

ACKNOWLEDGMENTS

This project has received funding from the European Union's Horizon 2020 research and innovation program under grant agreement No 771134. The project NETFIB was carried out under the ERA-NET Cofund SusCrop (Grant N°771134), being part of the Joint Programming Initiative on Agriculture, Food Security and Climate Change (FACCE-JPI). This work has also been supported by EIPHI Graduate School under grant agreement "ANR-17-EURE-0002".

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ID 247
PLANT FIBER INTERFACE ADHESION CHARACTERIZATION THROUGH PEELING TESTS: INFLUENCE OF PLANT VARIETY, RETTING AND EXTRACTION PROCESSES

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ABSTRACT
This workemploys a custom micro-mechatronic peeling experimental setup to characterize the adhesion properties at the interface of plant fibers within a bundle. The adhesion between different plant varieties, retting levels and extraction processes is compared. The fracture mechanisms taking place during the separation of a fiber from its bundle are also categorized and studied.

INTRODUCTION
The use of plant fibers as reinforcements in composite materials instead of synthetic fibers, represents an alternative with significant environmental benefits. However, in order to use plant fibers as composite reinforcements, a series of steps is necessary to isolate them from the rest of the plant tissues and separate them from fiber bundles to single fibers. This fiber individualization is critical to the final composite's performance (Charlet 2011).
From the growing and harvesting to the processing stages of plant fiber production many different parameters can impact the adhesion between elementary fibers and affect their individualization. Plant variety, maturity level, retting duration and conditions, mechanical extraction processes (scutching, hackling) are such parameters (Bourmaud 2018). Despite its importance however, no quantitative data exists on inter-fiber adhesion. Organoleptic and subjective criteria are used instead by workers, to evaluate if a given plant will result in good fiber individualization (Bourmaud 2018).
In this work, a custom micro-mechatronic experimental setup is used to peel plant fibers from their parent bundle using a combination of linear actuators and a microgripper (see Fig. 1 a. and b.). The force required to peel the fiber is measured by a force sensor and the inter-fiber adhesion can be evaluated through an analytical model (Kendall 1975). Through the use of an overhead microscope the fiber peeling is monitored and different fracture mechanisms are identified (see Fig. 1 c.). Their impact on the peeling force is also evaluated.
With the use of this setup the influence of plant variety and retting level on inter-fiber adhesion is studied for hemp fibers, while the influence of extraction processes (breaking unit or hammer mill) is studied for nettle.

RESULTS AND CONCLUSIONS
Fig 2 illustrates a typical plant fiber peeling result. The average peeling force can be identified allowing for the calculation of an average adhesion energy. Changes in peeling force can be observed that are often relied to fracture mechanisms at the fiber interface. Fiber kink bands or microfibril bridging (see Fig. 1 c.).

are the most common source, attesting to unique adhesion mechanisms at the plant fiber interface. The force required to break these interface features is also quantified.

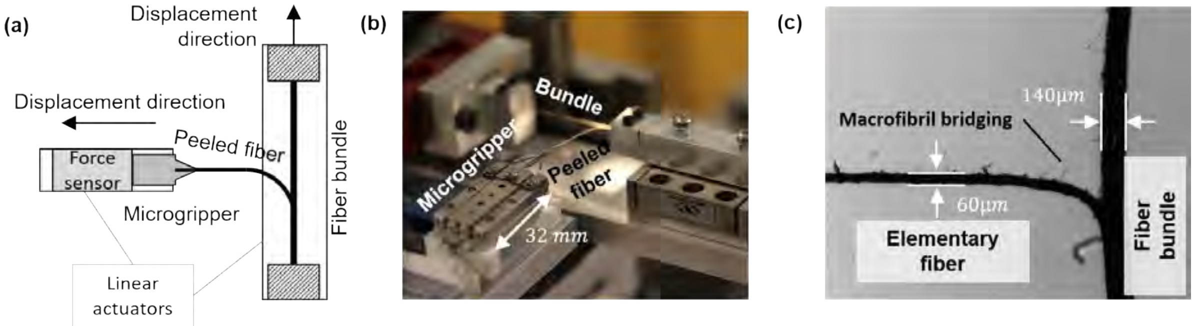

Fig. 1 a) peeling test schematic view, **b)** peeling setup close-up view, **c)** peeling zone observation by overhead microscope.

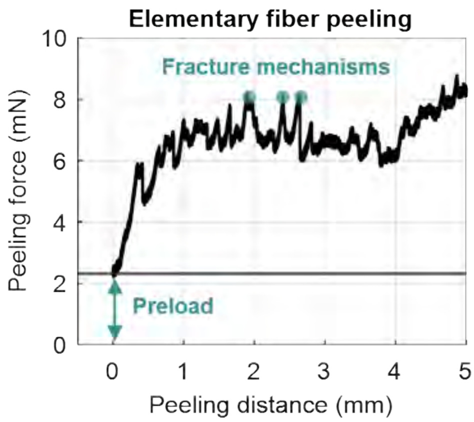

Fig.2 Typical example of plant fiber peeling test.

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ID 249
CHARACTERIZATION OF NATURAL FIBER ROPES FOR POTENTIAL APPLICATIONS AS REINFORCEMENT IN CONCRETE

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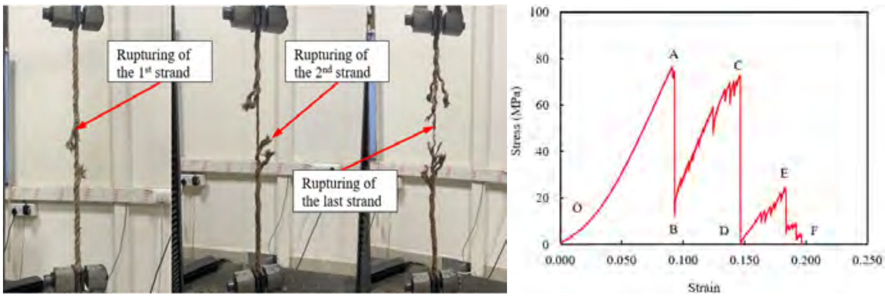
ABSTRACT
This research work focuses on the characterization of plant-based natural fiber ropes under ambient and elevated temperatures for potential application of the ropes as reinforcement material in concrete structures. Various plant-based natural fiber ropes are treated using NaOH solution and hot water, and the influences of these treatment methods on the physical, thermal, and mechanical properties, and chemical compositions of the ropes are experimentally investigated. Further, the plant-based natural fiber ropes are exposed to different elevated temperatures, and the effects of the elevated temperatures on the physical and mechanical properties of the ropes are studied. The identified characteristics of the ropes are essential in assessing the potential application of the ropes as a reinforcement material in concrete structures.

INTRODUCTION
Steel reinforcement is produced through high resource- and energy-intensive processes from non-renewable resources. Moreover, the production process results in emission of greenhouse gases, primarily CO_2, into the atmosphere, which has adverse implication in air pollution and climate change (Mahboob et al., 2021). However, the use of plant-based natural fiber ropes as a reinforcement material in concrete structures could offer many advantages due to their renewability, biodegradability, lower cost, abundant availability, lower energy for extraction, and lightweight. Recent studies have reported investigation on the use of banana fiber bars as a replacement of the conventional steel reinforcement in concrete beams and found that the banana fibers help to improve the flexural strength of the beams (Elbehiry et al., 2021; Elbehiry et al., 2020). Despite the potential merits of using the natural fiber ropes, there is lack of adequate research on the characterization of their physico-mechanical properties in the context of application as reinforcement.
In this study, various experiments are conducted to characterize different types of plant-based natural fiber ropes. The ropes are treated using NaOH solution and hot water treatment methods, and the influence of the treatments on the pertinent properties of the ropes are studied. Further, the ropes are exposed to 100, 200, and 300 ºC elevated temperatures, and the effects of the elevated temperatures on the physical and mechanical properties of the ropes are investigated.

RESULTS AND CONCLUSIONS
The results of the tensile strength tests are shown in Figure 1. The non-linear behavior of the stress-strain curve of the rope before the peak stress is one of the salient features observed in Figure 1(b). Moreover,

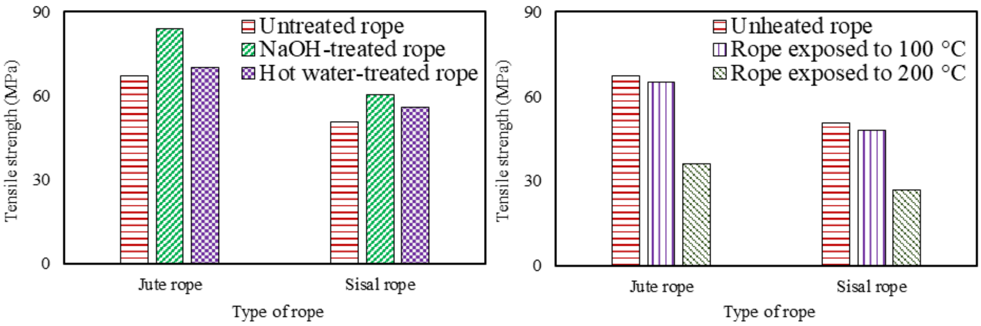
the stress-strain curve from point “A” to “F” indicates the effect of the successive rupturing of the different strands in the ropes. During loading of the rope, the stresses in the strands of the rope are not the same, and the strand with the maximum stress ruptures first. Consequently, the tensile strength of the ropes could potentially be enhanced by enabling the occurrence of a relatively uniform stress in the different strands.



(a) **(b)**

Figure 1: Tensile strength test results showing: **(a)** rupturing and **(b)** stress strain curve of the typical rope.

Figure 2(a) and 2(b), respectively, present the effects of various treatment methods and elevated temperatures on the tensile strength of the ropes. Here, NaOH solution and hot water treatment methods have been studied, whereas the ropes are exposed to elevated temperatures of 100 and 200 ºC.



(a) **(b)**

Figure 2: Effects of: **(a)** treatments and **(b)** elevated temperatures exposure on tensile strength of the ropes.

The results of this experimental investigation show that treatments and elevated temperature exposure have substantial effect on the tensile strength properties of the ropes. Moreover, effects of the treatments on the pertinent properties, such as water absorption capacity of the ropes, are also investigated.

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ID 250
NATURAL FIBRE REINFORCED COMPOSITES UNDER CYCLIC LOAD - IMPLICATIONS FOR DESIGN PROCESS OF LOAD BEARING PARTS

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ABSTRACT
Natural fibre reinforced thermosets are currently used in many lightweight design constructions. Due to their lower emission footprint in resourcing and manufacturing they promise a more sustainable product. However, material properties vary greatly depending on manufacturing method and processing. This paper gives a rough outline to the steps from material characterization and processing to choosing the right application, simulation and manufacturing of a natural fibre part. It sheds light onto the viscoelasticity of the fibre thermoset composite and by choosing a highly weight sensitive demonstrator part for the aero industry proves, that natural fibres are not always a more sustainable solution.
Keywords: Natural fibres, lightweight design, viscoelasticity, sustainability

INTRODUCTION
In a series of developments currently underway at the Institute for Textile Technology of the RWTH Aachen University high end structural applications for the mobility sector are developed using flax fibres. The parts are optimized and manufactured with the aim to prove or disprove their sustainability through a carbon footprint analysis over their life cycle. This process is undergone by designing and producing three separate demonstrators for the mobility sectors of air, rail and road travel. Within this paper the process from material characterization towards building a landing gear for an ultra-light plane, as in Figure 1, is discussed.
In the mobility sectors "the right material in the right place" is the premises for lightweight construction, which aims to optimize material application to save resources and energy. Fibre-reinforced composites (FRP) are predestined for this objective in the mobility sector due to their high specific strength, but here, too, resources and energy can be saved by selecting the right reinforcing materials and processes to manufacture them. The sustainability of the currently predominant materials of glass and carbon fibre reinforced plastics (GFRP and CFRP) is justified by the overall lifecycle balance and the savings in fuel emissions compared to metal counterparts.
In the mobility sectors "the right material in the right place" is the premises for lightweight construction, which aims to optimize material application to save resources and energy. Fibre-reinforced composites (FRP) are predestined for this objective in the mobility sector due to their high specific strength, but here, too, resources and energy can be saved by selecting the right reinforcing materials and processes to manufacture them. The sustainability of the currently predominant materials of glass and carbon fibre reinforced plastics (GFRP and CFRP) is justified by the overall lifecycle balance and the savings in fuel emissions compared to metal counterparts.

Natural fibre reinforced plastics (NFRP), however, offer the potential of a sustainable balance in every sub-cycle from production, through use, until the end-of-life. Thus the sustainability of NFRP stays valid regardless of the energy source used for powering the mobility application. Particularly with the progressive market entry of electric drive systems, the amount of emissions during the lifetime is reduced and thus the influence of this cycle on the overall balance. In the long term, therefore, the focus is not only on vehicle weight but also on particularly efficient manufacturing processes and resources.
While the production of one kilogram of carbon fibres emits 29.08 kg of CO2, this value for flax fibres is only 0.66 kg (Song, 2009). However, NFRP has decisive disadvantages. Due to their poorer mechanical properties, more material must be used for components than for the same component made of CFRP. The common properties like tensile strength, modulus and elongation of natural fibres are provided on all datasheets of the manufacturers. However, the performance in a composite structure varies and have more complex requirements for accurate characterization. Mechanical properties of matrices and natural fibres are particularly time dependent and develop permanent strain resulting in a complex material behaviour (Nordin, 2003). Another current focus of research is the improvement of vibration and damping behaviour without adding mass vibrations by adding viscoelastic layers (Khalfi, 2013, Gascon-Pérez, 2015).
In this paper first the material characteristics of NFRP with flax and thermoset matrix are classified and a suitable application for those properties in the aerospace industry is found. The design process is described and the carbon footprint analysis concludes the report.

RESULTS AND CONCLUSIONS

The viscoelastic behaviour of NFRP under cyclic load using a thermoset matrix has been shown. Furthermore, the resulting material characteristics were used to choose a fitting application. The landing gear was presented and the load cases simulated. Finally, with the landing gear as a demonstrative example, it was shown that NFRP as a material for structural parts under high load in the extremely weight sensitive aero industry does not yield a more sustainable product.

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ID 252

TO STUDIES THE INFLUENCE OF FABRIC HANDLE PROPERTIES ON SOYABEAN, POLYESTER, WOOL FIBRE BLENDS DEVELOP APPARELS WEAR

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ABSTRACT

Fabric handling, which also affects surface and mechanical properties, is a major aspect impacting the quality of fabric and apparel. A fabric hand or handle gives a sense of the texture of the fabric and depicts how the fabric feels in the human hand. There are also other psychological sensations that are felt, such as stiffness, softness, or hardness, as well as wetness or dryness. The goal of this study is to examine the fabric handling characteristics of twelve garment wears made with diverse raw materials Soyabean, polyester, wool, and blends but using identical fabrication techniques. On these fabrics, a comparison analysis was conducted. GSM, draping, crease recovery, stiffness (bending length), and fabric feel factor are all examined along with other properties.

INTRODUCTION

Fabric handle, together with the surface and mechanical properties, is a very important characteristic affecting the quality of cloths and garment. A fabric hand or handle depicts the way a fabric feels when it is touched by a human hand and gives an indication of texture of the fabric. Various psychological feelings such as stiffness, softness or hardness, wet or dry are also perceived. The handle properties of fabrics are related to the easiness of body motion and load generated in fabric during body movement, so that a study of clothing physical comfort is essential for the fabric's low stress mechanical properties. A lot of research has been carried out by many researchers on fabric handle properties which show that these are concerned with the judgment of harshness, smoothness, thickness, pliability, roughness etc. To increase the properties of resultant yarn type and to reduce the cost of the raw material blending of different fibres is done which is a common practice in the spinning industries. The percentage of fibres in the blend have effect on various properties and besides it, the properties of blended yarns are affected by the properties of the constituent fibres and their compatibility. Further, it is observed that the stronger fibres have to be blended at least by a certain percentage in order to increase the tensile properties.

Table 1 Specimen Fabrics

S.No.	Types fabric	EPI	PPI	GSM	Weave
1	100% Soyabean	58	50	195	Plain
2	100% Polyester	60	58	175	Plain
3	100% Wool	58	60	173	Plain
4	Soyabean/Polyester (50/50)	60	52	210	Plain
5	Soyabean/Polyester (65/35)	60	52	198	Plain
6	Soyabean/Polyester (35/65)	63	53	190	Plain
7	Soyabean/Wool (50/50)	62	52	185	Plain
8	Soyabean/Wool (65/35)	58	60	195	Plain
9	Soyabean/Wool (35/65)	58	60	189	Plain
10	Polyester/Wool (50/50)	60	56	167	Plain
11	Polyester /Wool (65/35)	60	58	196	Plain
12	Polyester /Wool (35/65)	60	56	175	Plain

RESULTS AND CONCLUSIONS

The fibre resilience value affects the drape coefficient, less the fibre resiliency less the drape coefficient wool fibre has excellent resiliency property and polyester also shows crispy and resilient behaviour. Soyabean fibre is less resilient then wool so blend of polyester and Soyabean shows less drape coefficient then other blends. Drape coefficient is directly proportion to bending length hence, more the bending length the drape coefficient or vice versa. According to results that 100% wool exhibits the highest value of crease recovery in warp & weft direction while 100% polyester shows lowest value of crease recovery in warp & weft direction. According to data found that, 100% polyester shows highest value of bending length while Soyabean/Polyester (65/35) shows lowest value of bending length. Bending length determines the flexibility of the fabric. Fabrics with high bending length are stiffer. Because of inherent nature of fibre soya is softer then polyester and wool. The fibre resilience value affects the bending length, less the fibre resiliency less the bending length. Soya fibre is less resilience than wool so Soyabean/polyester shows less bending length than 100% polyester.

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ID 256
ZERO WASTE ECO-PRINTING ON SILK SCARVES

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ABSTRACT
With the Covid-19 pandemic, users' lifestyles, clothing preferences and changes in their sensitivity to nature have been observed. More natural, more comfortable, nature-compatible activities and products demand is increasing. Especially in daily life, the needs for clothes have started to decrease, customer expectations are more focused on comfortable and nature based clothing regarding slow fashion. Eco-print process was developed and applied on comfortable cut wool-cashmere-containing knitwear, wool-cotton blended knitted surfaces and silk woven surfaces. The most easily applicable product category is selected as silk scarves. For the scarf and foulard products, studies will be carried out on the layouts in harmony with the colors and patterns in the shirt, trousers and dress category.

INTRODUCTION
When we look at world brands, the recycling trend is often associated with the concept of sustainability. We come across with an innovative approach to product designs not only with the recycling method but also with the upcycle method a new novel method is provided. In this journey, the raw materials and production processes of the clothes are protected without disturbing the ecological balance.
In Eco-print technique crucial stages are extraction, post treatments, coloring and the coloring materials (plant/fruit/vegetable/natural waste) (İşmal, 2016). It transfers the colors to the fabric with the help of pre-mordant technique. The product is the only example of each type of printing made with eco print. A new selection and placement of plants for each edition should be prepared. For this reason, it is very difficult to produce in series. However, it consumes less energy and water compared to serial printing techniques. Since coloring will be done by natural methods, tests have been carried out for wet and dry rubbing fastnesses. Dry cleaning fastness has been done instead of washing fastness. The feeling of pattern designs is as if the sunlight reflecting behind the tulle, it can be defined as the shadow of the plant falling on the plant and creating a projection to the ground.
Recipe optimization for color darkness, brightness, that is appropriate for obtaining the best results in terms of pattern clarity and it is very important to determine the amount of the substance and the application conditions (temperature, time, the way of using the plants, the way of binding, the type of the pipe on which the fabric is wrapped, fixation, etc.) (Özen and İşmal, 2021).
The mordanting procedure is one of the crucial steps in coloring materials. To ensure that the fat, oil, starch, and dirt left over from the weaving process are removed and the dye can be absorbed by the cloth directly, the fabric to be dyed is first mordanted (Nurmasitah, 2022).
In this study, mordanting was applied on silk woven surfaces, plant materials, and accompanying cloth (blanket fabric) separately. Firstly, silk base fabric was washed with water and without detergent. After drying, in order to open up and swell the fabric structure for better color penetration during printing, silk fabric was mordanted using tannin extracted from pomegranate peel, sage or green tea. For mordanting 100

grams of fabric, 20 grams of pomegranate peel is placed in 5 liters of water at boil, boiled for three minutes, pomegranate peel is removed and the fabric to be mordanted is placed in the solution for five minutes. Secondly, plant materials were mordanted using iron sulfate solution. For plant material to be applied on 100 grams of fabric, Iron sulfate solution was prepared by addition of 2 grams of Iron sulfate into 500 mL of water, dispersion of iron sulfate in the solution followed by totaling the solution to two liters with cold water. Plant materials selected are dipped in this solution and left to dry at room temperature. The print design composition was formed by placement of mordanted leaves and flowers, tree bark, and plant roots (i.e. Eucalyptus, black pepper, deer berry, sawtooth lion's claw, fern, cottonseed, pomegranate flower, thuja) upon the silk fabric. Thirdly, an accompanying cloth has been mordanted with 1g/L Iron sulfate solution and put on, forming the third layer at the top. The three-layer composite was rolled and tightly tied with a thin rope followed by boiling for three hours. For transfer and fixation of the colors from the plant materials on the fabrics, the three-layered structure goes through a hot calendar or hot iron at 160ºC. The most important part is that this process is a zero-waste process. The natural waste has been printed on both the scarf and the accompanying cloth. The accompanying cloth was used in manufacturing a satchel. Images of eco-printed silk scarf and satchel were presented in Figure 1.

RESULTS AND CONCLUSIONS

The scarves are tested for color fastness according to ISO 105-X12 for rubbing fastness, and ISO 105 -D01 for dry cleaning fastness. The results from the dry and wet color fastness to crocking were presented in Table 1.

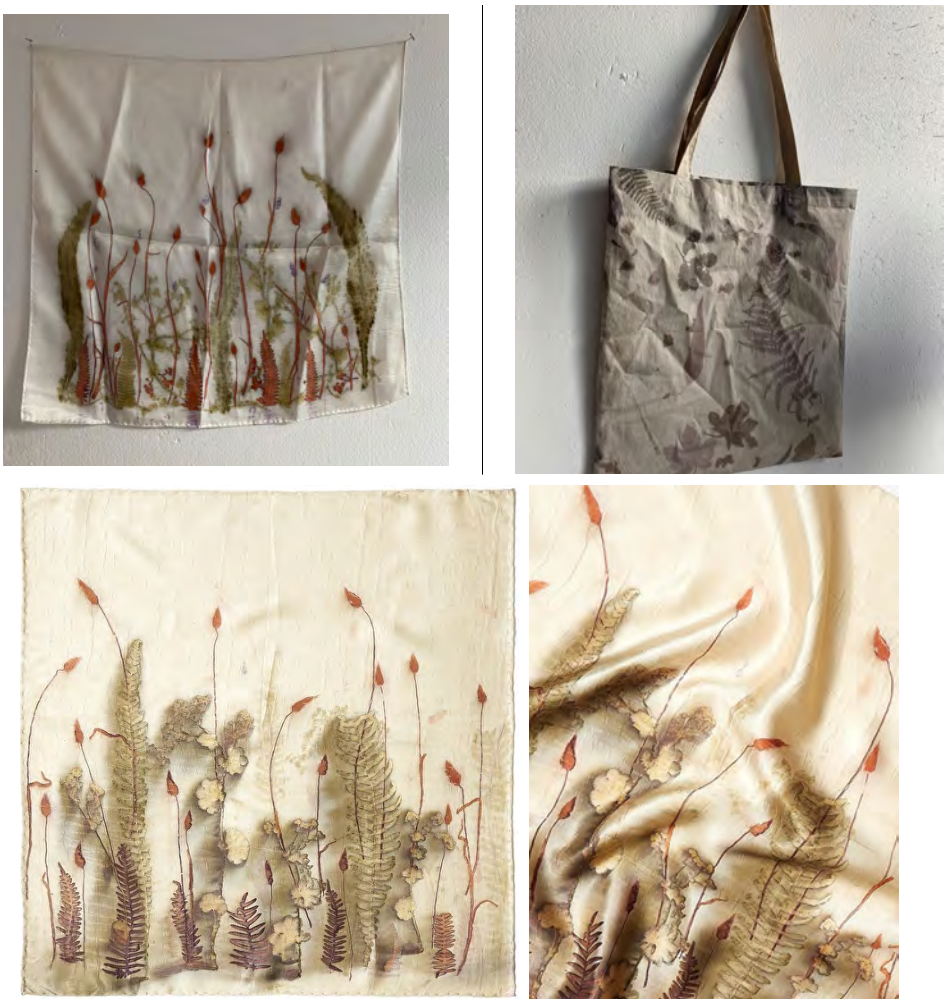

Fig.1 Images of Eco-Printed Scarf and Satchel

Table 1 Color Fastness to Crocking Test Results

Scarf	Staining	Color Change
Wet	4	4/5
Dry	4/5	5

As can be seen in Table 1, both dry and wet crocking fastness values are very high for eco-printing on silk material.
Dry cleaning test results were given in Table 2.

Table 2 Color Fastness to Dry-Cleaning

Scarf	Staining	Color Change
	5	5

As given in Table 2, the eco-print on scarves have reached perfect fastness performance against dry cleaning.
Zero-Waste Eco-Printing on silk was shown to offer high fastness values demanded for mass production by fulfilling the idea of bringing together a natural fiber fabric with natural colorants in harmony.

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ID 259
IMPROVING PROPERTIES OF THERMOPLASTIC STARCH FILMS WITH CELLULOSE NANOCRYSTALS FROM DIFFERENT SOURCES

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ABSTRACT
In this work, thermoplastic starch (TPS) was modified with cellulose nanocrystals (CNCs) extracted from bleached cotton and flax by sulfuric acid hydrolysis. Rectangular films with a thickness of about 70 µm were cast from the TPS-CNC suspensions. Cellulose nanocrystals as reinforcement were used in a wide concentration range (up to 50 %) in thermoplastic starch-based composite films. Structure and interactions were modified using different types (glycerol and sorbitol) and different amounts (30 and 40%) of plasticizers. Visible spectroscopy, Haze index measurements, and scanning electron microscopy were used to characterize the structure of the composites. The effect of CNC content on vapor permeability was also investigated. Significant differences were observed in the properties of TPS composites containing CNC from flax or cotton fibers as a function of composition. The differences were mainly due to the aggregation ability of the two types of CNC.

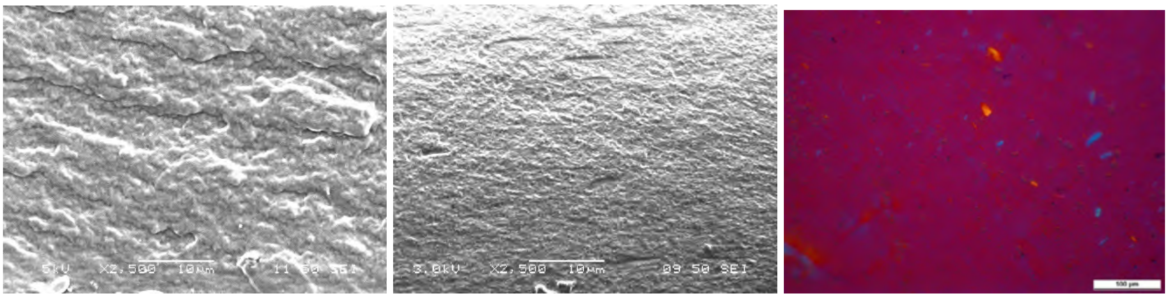
INTRODUCTION
Starch is a semicrystalline biopolymer that is readily available, inexpensive, and biodegradable. Despite being one of the most promising biopolymers, granular starch shows limited processability that makes the use of plasticizers such as glycerol, sorbitol, or water necessary. Starch granules are destroyed while applying plasticizers and specific conditions resulting in thermoplastic starch (TPS) showing a hydrophilic character and inadequate mechanical properties. However, when natural fibers, nanoclays, or other biodegradable polymers are incorporated, the properties of TPS can be significantly improved.
The effect of cellulose nanocrystals on the properties of TPS films has been widely investigated. In general, the CNCs improve the mechanical properties of the starch and reduce its water sensitivity. Published results generally show that the effect of CNCs is positive and that cellulose nanocrystals contribute significantly to improving the properties of TPS films. However, despite the numerous results, many questions remain unclear as the results are often contradictory.
In addition, the interactions are usually not quantitatively evaluated because of (1) the narrow concentration range of CNCs investigated and the fact that (2) the type and concentration of the plasticizer are not varied in the research. Furthermore, many publications do not specify the cellulose source and extraction

method used for producing CNCs, although these substantially impact the properties of CNCs.

RESULTS AND CONCLUSIONS

Significant differences were observed in the properties of TPS composites containing CNC from flax or cotton as a function of composition. The differences were mainly due to the aggregation ability of the two types of CNC. Due to the high aggregation of flax-CNC (Fig.1), a given amount of CNC induced a much more significant change in the haziness for TPS-flax-CNC films than for TPS-cotton-CNC films. Furthermore, while the strengthening effect of cotton-CNC was valid over a wide composition range, the flax-CNC increased the strength of TPS only in a narrow concentration range.

Nevertheless, the reinforcing effect of the two types of CNCs was influenced by similar interactions. In both cases, the lower plasticizer content and the use of sorbitol as plasticizer provided favorable conditions to achieve a remarkable reinforcing effect in TPS. The results highlight that precise knowledge of the properties and structure of CNCs used is essential for interpreting the properties of polymer/CNC composites and the compositional dependence of the properties.



a: TPS **b:** TPS-CNC **c:** TPS-CNC

Fig.1 SEM **(a, b)** and POM **(c)** micrographs – evidence of flax-CNCs aggregation

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ID 261
ECOLOGICAL PRINTING ON NATURAL FIBER FABRICS

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ABSTRACT
This study focuses on use of organic dyestuffs and an ecological thickening agent for printing in acidic conditions on luxurious fiber fabrics made of silk, cotton/silk and wool. Four ecological printing recipes were prepared using natural dyes, namely Sun Yellow made from marigold flowers (Tagetes Patula plant), Eco Saffron from Bixa Orellana flowers, Eco Beige from walnut shells, and Olive Green from blooming mulberry bushes (Moraceae family) as well as guar gum, non-toxic auxiliaries and water. Printing pastes were tested for stability prior to printing. Following application of the print paste and curing, printed samples were cold and warm washed, and dried. Finally, samples were tested for fastness.

INTRODUCTION
Due to growing environmental concerns and public awareness, using organic dyestuffs to color textile materials is becoming more popular (Gulrajani, 1999). Studies on the coloring of fabrics made of natural fibers tend to concentrate on dyeing (Davulcu, Benli, Şen Bahtiyari, 2014 and Tutak, Korkmaz, 2012), and there aren't many that address printing (Patel, Chattopadhyay, 2009). Since organic dyestuffs and environmentally friendly thickening agents were used in this work, our goal was to provide good color yield with acceptable fastness on silk, wool, and cotton/silk fiber fabrics that were ecologically printed.
Throughout the investigation, twill woven textiles with unit weights of around 50 g/m^2 of 100% wool, 100% silk, and 54% cotton/46% silk were utilized. Throughout the study, natural dyes were employed, including Sun Yellow made from marigold flowers (Tagetes Patula plant), Eco Saffron from Bixa Orellana flowers, Eco Beige from walnut shells, and Olive Green from blooming mulberry bushes (Moraceae family). The agent applied for thickening was guar gum. Printing pastes were prepared in water media using natural dyes, ecological thickening agents and non-toxic auxiliaries, and conditioned for three hours prior to print paste preparation. Change in color of print pastes were observed after ten days for stability. The printing was done using the screen printing method, and finished with fixation at 102 °C for 45 minutes. After printing, distilled water without a surfactant was used to wash the residual ink off. A two-step washing off was carried starting with cold washing for 15 minutes followed by washing at 40°C for 10 minutes. Finally, printed samples were dried at 130 °C for 20 minutes. According to ISO 105-E01:2013 - Colour fastness to water, samples of printed cloth were subjected to color fastness testing.

RESULTS AND CONCLUSIONS
Figure 1 displays wool and silk fabrics with eco-friendly printing. Figure 1 clearly shows that a high color yield was attained without haloing.

Fig.1 Ecological printed silk and wool fabrics.

Results of water fastness tests on ecologically printed silk and wool fabrics are shown in Table 1. As a result of the fastness test, eco-printing with acceptable to high fastness was reached.

Table 1 Color fastness to water test results

SUN YELLOW	ECO SAFFRON	ECO BEIGE	OLIVE GREEN
2/3	3/4	4	3/4

In this study, natural fiber fabrics were printed with ecological recipes comprising natural dyes as colorant and guar gum as thickener. It is shown that luxurious fiber fabrics can be printed using organic dyestuffs in acidic conditions with ecological thickening agents. High color yield and fastness performance were achieved without haloing. Moreover, the waste water contains auxiliaries, natural colorants and ecological thickening agents all of which are non-toxic for the environment. Further tests should be performed to analyze the effects of external agencies such as detergent washing and sunlight on the performance of ecological printed products.

ACKNOWLEDGMENTS

The authors would like to thank Vakko Esenyurt Design Center for their support throughout the study.

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ID 263
INVESTIGATING THERMO-PHYSIOLOGICAL COMFORT PROPERTIES IN MOHAIR SOCKS.

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ABSTRACT
This work compares the thermo-physiological comfort properties of medical socks, comprising of mohair fibres or yarn in different structures, with alternatives in the market. A variety of techniques was used to evaluate thermal and moisture management properties, including the Alambeta, Permetest, Thermal foot manikin and thermochromic iButtons. The results emphasize the advantages of the use of natural protein fibres to aid in the therapeutic support for symptoms of diabetes, circulatory problems, sweaty feet, etc. The study also illustrates the suitability of the various test methods for the effective prediction of the in-use performance of socks.

INTRODUCTION
Mohair, produced from the Angora goat, is regarded as one of the most luxurious and best quality natural fibres. Its unique combination of characteristics, such as lustre, colour reflection, resilience, fineness, exceptional wavy crimp structure, etc. leads to performance properties that made it popular in a wide range of applications in knitwear, suiting, blankets, upholstery, carpets, curtaining, etc. Lately, mohair blends have also become popular in socks – for leisure- as well as sports- and medical applications (Fan LJ & Hunter L, 2009; Botha A.F. & Hunter L., 2013). The shift from traditional apparel and household items towards novel applications such as the above, emphasizes the need for research on the performance of mohair in fabric structures (Gericke, *et al.*, 2022).
The objective of this study was to do a comprehensive quantitative evaluation of the comfort-related performance properties of medical socks containing mohair. In vitro as well as in vivo test methods were used to compare results and determine which tests or instruments would prove to be the best suitable to predict performance. Instruments include the Alambeta and Permetest to measure the thermal resistance (Rct) and moisture vapour resistance (Ret) of fabric samples in the laboratory. The Rct of the full sock was determined on the Thermal Foot Manikin (Rogina-Car *et al.*, 2019, 2020; Fung *et al.*, 2020).
The comfort factor experienced by the wearer while wearing the sock over a given time, however, is dependent on the conditions developing in the micro-climate between the fabric and the skin. The fabric that the shoe that covers the sock is made of, will interfere with the release of heat as well as moisture from the skin to the environment. To take this into account, further evaluations were performed with the use of thermocron iButtons during wear trials to determine the temperature and humidity conditions inside the micro-climate. These small, stand-alone dataloggers have been proven to work effectively in the related trial where the conditions inside the micro-climate were investigated (Splendore *et al.*, 2011; Gericke, Militký, *et al.*, 2022).

RESULTS AND CONCLUSIONS
Analyses of the thermal resistance results measured on the fabric samples versus those measured on the full sock indicated a trend towards correlations between the two sets of data, but not enough to base any

conclusions on. Results are shown in Table 1

Table 1 Thermal properties of measurements on single-layer fabrics as well as on full socks

Sock code	Fabric Description	Fabric Thickness (mm)	Fabric Thermal Conductivity (W m^{-1} K^{-1})	Fabric Thermal Resistance (W^{-1}m^{2} K)	Full sock Thermal Resistance (W^{-1}m^{2} K)
S-MW	Mohair-wool1	4.1	0.045	0.098	0.0461
S-MWV	Mohair-wool2	3.5	0.046	0.063	0.0207
S-MWV	Bamboo-viscose	3.9	0.079	0.054	
S-PET	Recycled polyester	4.6	0.067	0.007	0.0502
Instrument			Alambeta		Thermal Manikin

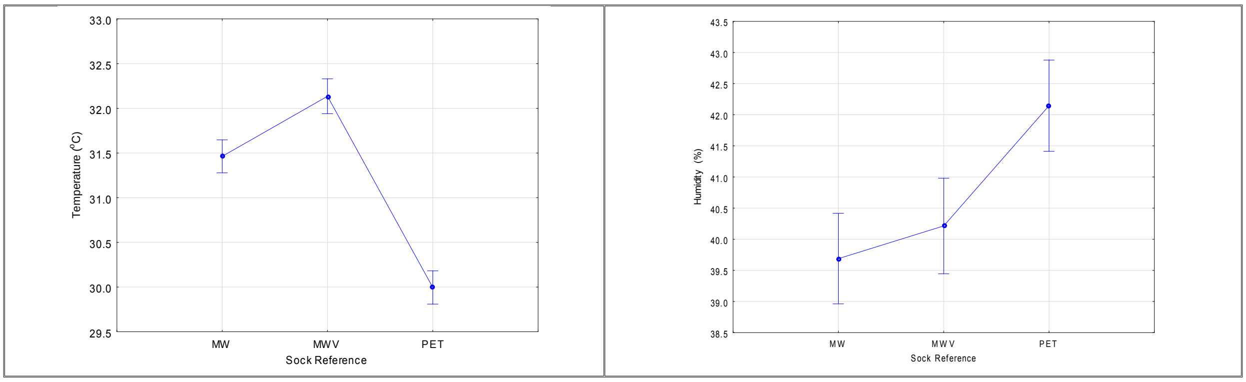

Fig.1 (a) Temperature (°C) and **(b)** humidity (%) as measured inside the micro-climate between the sock fabric and the skin

This study shows that, surprisingly, regarding temperature, the conditions inside the micro-climate were not a direct reflection of the thermal resistance of the fabrics that the socks were made of. The humidity inside the micro-climate, however, showed the effect of the mohair/wool fibres on the dissipation of moisture was significant and the natural moisture management properties of the protein fibres were evident.

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ID 264
MICROPARTICLE COATING FOR BONDING IMPROVEMENT OF NATURAL FIBER TEXTILE-REINFORCE MORTARS (NFTRM)

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ABSTRACT
The complexity of the bond behavior of natural fiber textile-reinforce mortars (NFTRM) surrounds shear stress transfer mechanisms and their impact on tensile performance. This research aimed at bonding improvement through silica coating on two commercial jute textiles after the desizing process. Characterization tests for textiles, matrix, and composite were evaluated, bending test of composite differentiated the effect of fabric treatments. Findings showed similar yarns, but different tensile properties of woven fabrics, after silica, only opened jute fabric indicated its effect on strain and stress gains.
Keywords: bonding, natural fiber, jute textile, mortar, NFTRM.

INTRODUCTION
In the last years, solutions for strong bonding between textiles and cementitious matrix have been sought. Traditional textiles provide a large diameter of yarns, hindering matrix impregnation and leading to possible slip between inner, named telescope effect (Trochoutsou et al., 2021) because only the external surface of the bundle interacts and transfers stresses (Alatawna et al., 2021; Fidelis et al., 2016). In addressing this problem, initiatives are still being studied to achieve better tensile properties of the composite by polymer impregnation or/and inorganic particle coatings (Nadiv et al, 2017).
This study aimed to evaluate the interactions and differences between jute textile woven patterns and mortar matrix, before and after the micro-silica coating process. Two plain jute woven fabrics were desized by an alkaline and oxidation bath, after that, the micro-silica coating was applied. Short eucalyptus pulp, CPV ARI cement - Type III, and limestone constituted the matrix. Slurry-dewatering molding method was used to sheet production. Matrix composition (X-ray fluorescence spectrometer and thermogravimetric analyses); textile characterizations aimed to identify the structure of yarns (mechanical tensile properties, count, and torsion) and fabrics (mechanical tensile properties, mass per area and, covered factor); additional tests (FTIR, Optical micrography) were employed to verify the degree of silica impregnation; and bending capacity of NFTRM boards evaluated the coating effects of textiles for the matrix reinforcement in 8 days of thermal cure.

RESULTS AND CONCLUSIONS

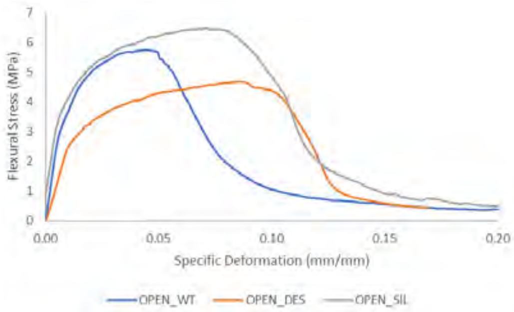
Results pointed out jute woven fabrics were made by similar yarns (count yarn and twist) around 400 tex and single-ply jute yarns had the “Z” direction, an average of 172 twist/m. Fabric structures demonstrated great differences, being CJF presented higher mass per area, covered factor, and tensile strength at break (258 ± 15 g/m^2, 0.5, and 391 ± 13 N – weft/ 510 ± 30 N warp, respectively), while opened jute fabric – OJF (192 ± 8 g/m^2, 0.44, and 174 ± 33 N weft/432 ± 62 N warp, respectively.

In general, alkaline and oxidative baths were applied for starch and impurities removal, as a consequence, reduced tensile stress, and fabrics become more hydrophilic to standardize the silica coating impregnation. Just this loss of tensile properties can be observed at the bending test of composite sheets (Fig. 1b, orange line), using as reinforcement the desized fabric (OJF), which decays the flexural stress but grows the strain. With silica addition, mechanical properties were recovered (Fig.1a and 1b) and enlarged (from 4.97 ± 0.41 MPa to 6.81 ± 1.25 MPa of modulus de rupture), which could be confirmed at ANOVA analysis and the Tukey test was performed at a 5% level of significance, being silica had effect only to OJF, obtaining advantages of MOR, MOE, and SE, except LOP.

	MOR (MPa)	LOP (MPa)	MOE (GPa)	SE (kJ.m^{-2})
OJF_WT	5.75 ± 0.63 b	3.02 ± 0.64	2.29 ± 0.35 a	2.72 ± 0.59 bc
OJF_DES	4.97 ± 0.41 c	2.67 ± 0.1	1.52 ± 0.4 b	3.21 ± 0.51 b
OJF_SIL	6.81 ± 1.25 a	3.37 ± 0.95	2.36 ± 0.63 a	4.28 ± 0.91 a
CJF_WT	5.37 ± 0.71 b	2.7 ± 0.41	1.41 ± 0.23 bc	3.23 ± 0.77 b
CJF_DES	5.76 ± 0.65 b	3.11 ± 0.59	1.72 ± 0.33 b	3.09 ± 0.74 b
CJF_SIL	5.07 ± 0.54 bc	2.7 ± 0.3	1.58 ± 0.13 b	3 ± 0.65 b

*The letters in the same column represent the statistical difference from the Tukey test ($p < 0.05$)

(a)



(b)

Fig.1 Mechanical results of composites: (a) Bending test results of opened jute fabric (OJF) and closed jute fabric (CJF) without treatment, desized and silica coating: modulus of rupture (MOR), limit of proportionality (LOP), modulus elastic (MOE), and specific energy (SE), (b) Graph of bending test results of opened jute fabrics.

As result, even though the yarns are similar, the effect of closed jute fabric was not the same, it probably is associated with low interaction of cementitious matrix inside yarns and led to premature failure. Nevertheless, silica coating impregnation could enhance the bonding of jute reinforcement and mortar of thick yarns, remarkably employing opened jute fabric (OJF) reinforcement.

ACKNOWLEDGMENTS

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ID 266
ENGINEERED HYBRID BRAIDED TEXTILES FOR COMPOSITES

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ABSTRACT
Braided textiles can achieve a higher modulus of elasticity as compared to traditional yarns. This study aimed to develop braided textiles from sources already used in civil construction, such as PES/glass core, and compare them with PES/sisal core, according to tensile strength, tenacity, yarn count, thickness, and angle braids. Results indicated modulus of elasticity values for PES/glass rod values increased as much as yarn count, but its elongation was limited. In this way, PES/sisal core (47/53%) rod presented strain capacity and energy absorption, PES/glass core had the highest values of modulus of elasticity and tensile strength, which are indicative of potential use for composite reinforcement.
Keywords: braided, hybrid textiles, sisal, glass, polyester.

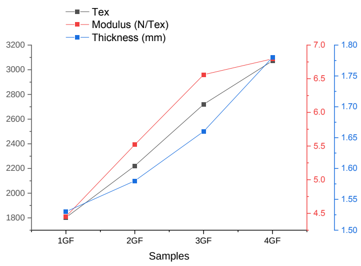
INTRODUCTION
Engineered textile materials have been developed to achieve high performance on structures, such as braid textiles, which permit flexibility and high tensile strength in the longitudinal direction, allowing the use in composite materials, ropes, and biomedical applications (Freire et al., 2021). In general, the braided structure depends on the braid angle, diameter, and pick count. A slower production affects the angle, promoting tight spacing and stiffer (Hamouda et al., 2022). Rods made from braided polyester and glass textiles were employed in meshes to control the damage in the masonry infills, reducing the cracking of mortar and presenting lateral drift of 2.5%, increments of 51% and 39% on the energy dissipation capacity (Akhoundi et al., 2018).
This study aimed to develop hybrid braided textiles, comparing traditional raw materials for reinforcement of composites for structural applications, PES/glass core (four samples, Tab.1a) and PES/sisal core (three samples Tab. 1a). A bidirectional braiding machine with 16 rows (polyester HT, 45 Tex) and core (Advantex T30 - glass filaments, 410 Tex; or sisal cord, 1100 Tex) was used, maintaining constant low velocity (90 m/h). Tensile strength (ISO-2062:2009), tenacity, thickness, count number, and, braid angles were determined.

RESULTS AND CONCLUSIONS
According to developed braided rods, with the increase of yarn count (1802-3072 Tex, Tab.1a) of PES/glass core rods the modulus of elasticity (4.45-6.8 N/Tex, Fig.1b) and tensile strength at maximum force (467-756 N) also grew up. However, due to the stiffness of glass fiber and the high elongation capacity

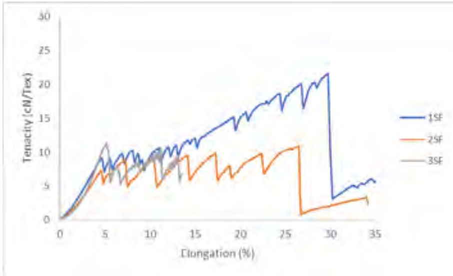
of polyester, only 1GF could achieve more than 20% of elongation and the others were lower, the break was almost after elastic behavior, achieving only 13% - 2GF, 8.5% - 3GF, and 7% - 4GF). The other rods made from PES/sisal core presented decrescent values of modulus of elasticity Tex (3SF - 2.12 ± 0.33 N/Tex, 2SF - 1.76 ± 0.14 N/Tex, and 1SF - 1.29 ± 0.13 N/Tex) concerning yarn count (2580-5150 Tex). The tenacity curve of sample 1SF (43% Sisal, 57% PES) provided the best performance of elongation at break (29.17 %), tenacity (21.5 cN), and maximum load (449 N) (blue curve, Fig.1c), but lower modulus of elasticity (2.12 N/Tex) than all other PES/glass core rods. It is very important to note, this last sample could achieve the highest tensile strength at almost maximum elongation, even with multiple failures of the sisal cord core that could support the load and followed the elongation capacity of polyester filament.

Sample	Yarn Count (Tex)	Composition: External (%)	Composition: Core (%)
1GF	1802	PES (78)	glass (22)
2GF	2221	PES (63)	glass (37)
3GF	2720	PES (55)	glass (45)
4GF	3072	PES (47)	glass (53)
1SF	2580	PES (57)	sisal (43)
2SFF	4090	PES (46)	sisal (54)
3SFF	5150	PES (36)	sisal (64)

(a)



(b)



(c)

Table 1 (a) presenting yarn count (Tex), composition of PES/glass and PES/sisal rods of developed samples; **Fig.1 (b)** Proportionality of yarn count (Tex), modulus of elasticity (N/Tex), and thickness (mm) of PES/glass core rods; **(c)** Tenacity (cN/Tex) versus elongation (%) of PES/sisal core rods;

The angles of braided rods were measured at initial, elastic, and before tensile test failure. The initial state of rods: PES/sisal core (1SF, 2SF, and 3SF) presented the major difference between them (36°, 42°, and 46°), probably influenced by increased thickness (2.41 mm, 2.84 mm, 3.37 mm); while PES/glass core (1GF, 2GF, 3GF, and 4GF) showed similar findings of angles (26°, 29.5°, 30.6°, 31°) and thickness also following low range (1.53 mm, 1.58 mm, 1.66 mm, 1.78mm).

Finally, PES/glass core samples reached the highest values of modulus of elasticity and tensile strength, however, the lowest elongation values. The sample 1SF – SISAL/PES core presented the growth of strength values throughout the rod elongation (Fig.1c), showing the best values of strain capacity and energy absorption, which are indicative of potential application for composite reinforcement.

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ID 267
EXPERIMENTAL COMPARISON BETWEEN NATURAL AND SYNTHETIC FIBERS WITHIN AN OPTIMIZED COMPONENT FRAMEWORK

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ABSTRACT
This work aims at evaluating experimentally six different fibers in a topologically optimized component. The selected fibers are carbon, E-glass, viscose, flax, hemp, and basalt fibers. Tailored fiber placement was used to manufacture the textile preforms, which were infused with a partly biogenic epoxy resin. The objective of this study is to evaluate and compare the absolute and specific mechanical performance of synthetic and natural fibers within a component framework as a base for improving assessments of endless-fiber reinforced composite material used ecologically. Furthermore, manufacturing aspects regarding the different fibers are also considered in this work.

INTRODUCTION
Carbon fibers are usually used as reinforced plastics due to their exceptional mechanical properties. However, their manufacturing has, in general, a large carbon footprint (Hermansson, 2022). On the other hand, many natural fibers are available in the market, despite not being used as reinforcement when structural performance is crucial for several reasons: problems in resin-fiber interface, low strength properties, moisture absorption, great variance in performance, etc. (Spinacé et al., 2009; Faruk et al., 2014; Dong, 2017). Still, some natural fibers can achieve mechanical performances (stiffness) comparable to glass fibers (Deerai et al., 2021).

Many studies have considered comparisons in terms of evaluating simple tests on coupons (Spinacé et al., 2009; Faruk et al., 2014; Deerai et al., 2021). In the authors' knowledge, little to none has been performed in terms of a component level. The objective of this work is to apply six different fibers, both natural and synthetic, as reinforcement to an optimized component, originally considered as a carbon-fiber reinforced polymer (Albers A et al., 2023), and study their processibility and performance mechanically. The configuration of the specimen improves the capability of the fiber-reinforced polymer and, therefore, the comparison is straightforward. The fibers chosen are carbon, E-glass, viscose, flax, hemp, and basalt. The Tailored Fiber Placement (TFP) technology (Mattheij, 1998; Bittrich, 2019) was used to manufacture the specimens for testing. This technology, as shown in this work, can deal with many fibrous materials by embroidery means.

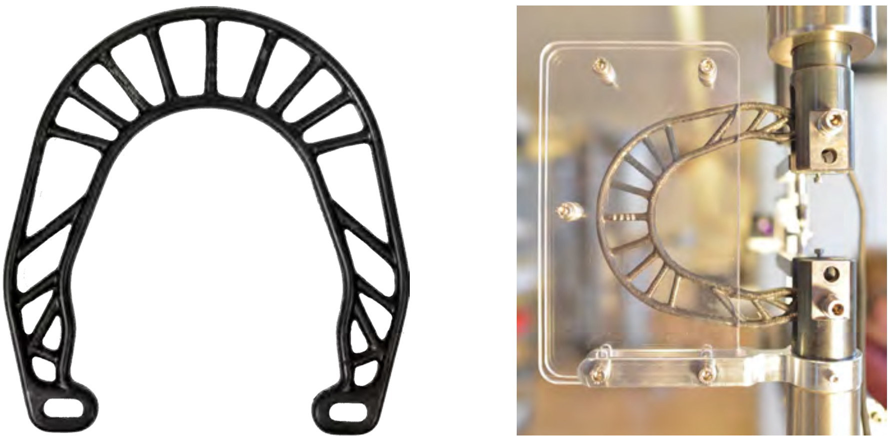
TEST AND SPECIMENS PREPARATION
The TFP technology is used to produce the preforms of the component. Fig. 1(a) shows the component's preform of carbon being made. In essence, a rotating delivery eye stitches the rovings onto a base material in predetermined places. One can observe that any fibrous material that can be made as a roving could, in

principle, be used in this manufacturing process. More information about the process can be found in Mattheij, 1998. The preforms are then cut (Fig. 1(b)) and placed inside the molds, which are made of silicone. 3D-printed molds are produced to cast the silicone with a particular thickness profile of the component (Fig. 1(c)). For the infusion, the partly biogenical epoxy resin SR InfuGreen® 810 (with the hardener SD 8822) was used. According to the manufacturer, it is produced with 38 % of carbon from plants and has a lower impact when compared to standard epoxy systems.

(a) (b) (c)

Fig. 1 – (a) Manufacturing of the brake booster preform by TFP (carbon roving), **(b)** the partially pre-cut preform, and **(c)** top and bottom of the silicone mold made by casting of 3D-printed models .

The component considered for the mechanical testing is shown in Fig. 2a. It is essentially a topology-optimized counterpart of a bicycle brake booster. Fig. 2b shows the mechanical test. The geometry favors the preferable compression and tensile loading of members, resulting in a good mechanism for exploiting the fibers' mechanical performance.



(a) (b)

Fig. 2 - (a) Brake booster and **(b)** testing procedure.

The fibers considered in this work are presented in Table 1. The cross-section is defined through the density and the linear density (* means that these values have been determined by the manufacturer). This information is of importance given the fact that the thickness profile of the component will change depending on the fineness of applied fibers. As the main idea was to have a "fair" comparison between the different materials, the thickness was used as a parameter to be the closest between the specimens.

Table 1 - Properties of the fibers in the work.

Fiber type	Family	Density [g/cm³]	Linear Density [tex]	Cross-section [mm²]
Carbon		1.770	800.00	0.4520
E-Glass	Synthetic	2.550	112.95	0.1416
Basalt		2.804	397.00	0.0443
Viscose		1.500*	123.12	0.0821
Hemp	Natural	1.500*	1250.00	0.6870
Flax		1.500*	1030.53	0.8333

Figure 3 – Brake booster specimens of each material: hemp, E-glass, viscose, basalt, flax, and carbon (from top to bottom - left to right).

As a result, three different molds were prepared in accordance with the differences between the sample's cross-sections. One mold was defined to the synthetic fibers. Basalt and E-glass rovings were rewound 3 and 10 times, respectively, in order to increase the roving's cross-sections used for TFP. Still, around 9 % difference is expected in terms of the thickness of the specimens when comparing basalt to carbon/glass components. Similarly to E-glass, viscose had to be rewound 10 times to get to a roving fineness comparable to hemp. However, no possible comparison can be made between the flax and hemp/viscose model. Consequently, flax has its own mold.

RESULTS AND CONCLUSIONS

The experimental campaign is still going on. They will be finished by the end of February. For each fiber type, 5 specimens are to be manufactured. Furthermore, coupons level experiments are also planned, aiming at obtaining the mechanical properties of the fibers alone and infiltrated.

Regarding the manufacture of the specimens, since the natural fibers have no sizing, the handling for positioning inside the molds was somewhat more complex. Moreover, given some friction between the roving and the TFP specific delivery eye, stitching fibers like viscose was more laborious. This has shown that approaches commonly used for carbon/glass fiber might be modified to the respective natural fibers, like mold tolerances and dimensions, for example.

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ID 268
APPLICATION OF NIOBIUM OXIDE: FUNCTIONALISED FLAT FABRICS FOR SMART TEXTILES

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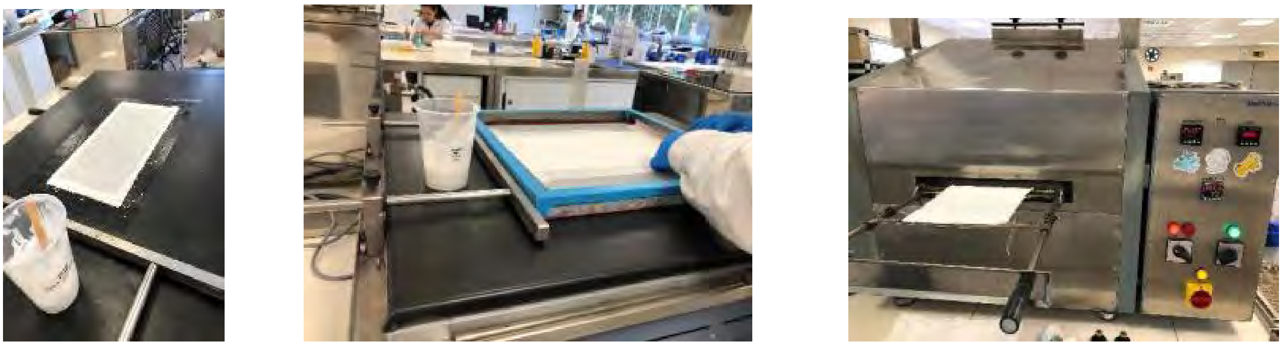
ABSTRACT
In the textile sector, research is developed for intelligent fabrics, which can respond to various functionalities. In general, metals are applied in the manufacture of chemical fibers and in the finishes that we apply to the textile surface. The objective of this study was the application of three niobium oxides on the textile surface with analysis of the properties of the new fabrics and identification of adherence. The methodology consisted of the application of niobium oxides (1G, 2P, and 3H) with acrylic resin on the surface of 2 fabrics (cotton and flax) and identification tests of impregnated Nb2O5 such as Raman, X-ray, UV-Vis, optical microscopy and TGA. Results indicate that there was impregnation of Nb2O5 in the fabrics. More studies for analyzing the fabric's functionalities and processes/optimization of impregnation, and durability, among others, are necessary.

INTRODUCTION
Textile finishing processes can provide fabrics with new functions and properties (Ferreira et al., 2014). Among the most used nanomaterials (NM), nanosilver (NPAg) stands out, which has antimicrobial and fungicidal action (Santana et al., 2015). The element Niobium is in the group of refractory metals due to its high melting point, 2468°C. Niobium and niobium compound films can be obtained by different techniques and provide different properties depending on the application technique used (Geríbola, 2014; Nico et al., 2016). The aim of this study was the application of three niobium oxides on the textile surface and the identification of adherence. The methodology consisted of the application of niobium oxides (1G, 2P, and 3H) with acrylic resin on the surface of 2 fabrics (cotton and flax) and identification tests of impregnated Nb2O5 by: Raman, X-ray, UV-Vis, optical microscopy and TGA.

RESULTS AND CONCLUSIONS
The application of niobium oxide (3 samples of chemical purity: 1G (≥ 99.8%), 2P (≥ 98.0%), and 3H(≥ 98.5%)) in the screen-printed process was carried out as follows: 1) wash the fabric; 2) prepare the paste

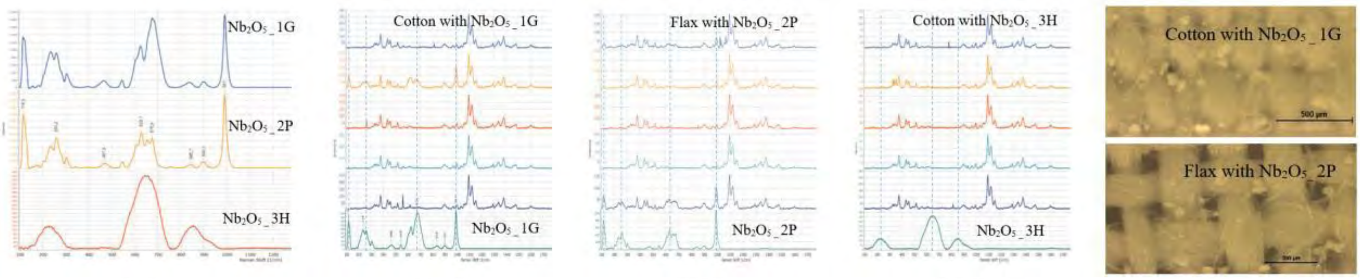
for application with Styrene Acrylic Resin and the niobium compounds, for 15 minutes in the motor agitator (18rpm); 3) applications of the pastes with Nb2O5 on the electromagnetic table; 4) place in two moments for drying the paste and fixation of the paste on the fabrics in the laboratory stenter frame (Fig.1).



(a) (b) (c)

Fig.1 Nb2O5 application process **(a)** Electromagnet table, **(b)** Frame on the fabric on the electromagnet table, and **(c)** Stenter frame 2 times in 2 min (drying and polymerization).

Fig.2 shows the Raman test and optical microscopy results



(a) (b) (c) (d) (e)

Fig 2. Raman spectra (a) Original Niobium Oxides (b) Cotton impregnated by Nb2O5 1G, (c) Flax impregnated by Nb2O5 2P, (c) Cotton impregnated by Nb2O5 3H, and (e) Optical Microscopy of Nb2O5 impregnated on the textile surface.

In the Raman and X-ray tests, the Nb2O5 1G and Nb2O5 2P in the base of both, cotton and flax fabrics, show similar peaks that demonstrate the presence of Nb2O5 in the impregnated fabrics. In the application of Nb2O5 3H, the same vibrational peaks that niobium identifies in the Raman and X-ray tests were not found, this is because it is changed amorphous by short-range atomic ordering (Skrodczky et al., 2019). In the optical microscopy images (Fig. 2e), Nb2O5 particles were identified in all impregnated fabric, regardless of the base and the compound. The TGA with the impregnated fabrics did not show any differences with the fabric without impregnation, due to its point of degradation of celluloses and lignins, which predominate concerning the niobium oxides that are degraded from 1500°C. UV-Vis shows an ultraviolet absorption curve after treated fabrics, unlike untreated fabrics, which show reflectance in the ultraviolet wave range. After these processes, the impregnation of niobium oxides on the textile surfaces was achieved, except for the 3H sample (amorphous). There is a need to analyze the fabric's functionalities and study new processes/optimization of impregnation, and durability, among others. These functionalized fabrics have high potential in different areas of applicability.

ACKNOWLEDGMENTS

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EFFECTS OF FIQUE FIBER TYPE AND ORIENTATION ON THE MECHANICAL PROPERTIES OF EPOXY-FIQUE BIOCOMPOSITES

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ABSTRACT

This work compares the tensile and flexural mechanical properties of biocomposites from a epoxy matrix and Three types of Fique arrangements: Fique powder, industrial nonwoven Fique Fiber mat and a unidirectional fique fiber mat. The mechanical tests performed allowed to identify significant differences on the mechanical properties of the manufactured biocomposites. Furthermore, it was found that fique powder acts as a filler for the epoxy matrix while the fique fiber mats reinforce the polymeric matrix.

INTRODUCTION

A natural fiber extracted from Fique plants (Furcraea andina), native of the Andean region of South America, has recently shown potential for the manufacture of biocomposites for engineering applications (Muñoz-Velez, 2018). Recent research has shown that this fiber can compete with other known natural fibers to produce reinforced biocomposites (Hidalgo-Salazar, 2018). Therefore, in this work a mechanical characterization of biocomposites was carried out from different types of fique (Fique powder, industrial nonwoven Fique Fiber mat and a unidirectional fique fiber mat) with an epoxy matrix. The effect of the type of fique arrangement used in the tensile and flexural mechanical properties was evaluated. This would allow appreciating the real possibilities of this natural fiber to be used as reinforcement for polymer matrix composites. Because the aim of this study is to compare the mechanical characteristics of Epoxy-Fique biocomposites, the tensile and flexural tests were performed at 23 °C with an INSTRON universal testing machine model 3366 according to the ASTM D 638-14 and D 790-17 respectively (Hidalgo-Salazar, 2018). All the results were taken as the average value of five samples. All the biocomposites were manufactured using resin film infusion technique and the Epoxy-Fique weight fraction was 70/30 (% wt/wt).

RESULTS AND CONCLUSIONS

Figure 1 shows the different biocomposites produced, it is observed that resin film infusion technique allows to manufacture homogeneous sheets with well-distributed Fique Fibers The influence of Fique Fibers addition on the Epoxy mechanical properties was evaluated and presented in Table 1. This study shows that there are substantial differences on the mechanical properties of different biocomposites. In general terms, the addition of fique fibers improved the mechanical properties of the epoxy matrix. Among the biocomposites it is observed that the fique powder acts as a filler, while the best results were obtained with the non-woven and unidirectional fique mats.

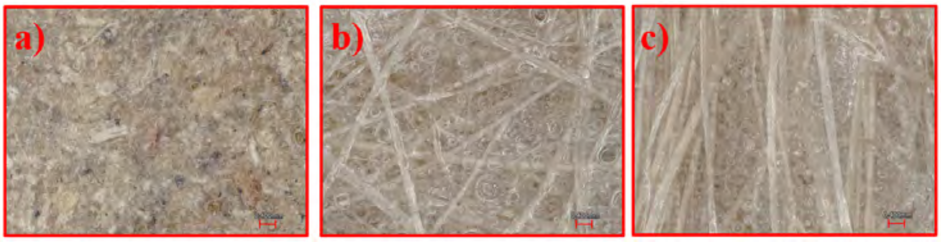

Fig.1 The biocomposites made from epoxy resin and **a)** fique powder, **b)** industrial nonwoven Fique Fiber mat and **c)** unidirectional fique fiber mat

Table 1 Tensile and flexural properties of EP and EP-Fique biocomposites

Sample	Tensile and Flexural Properties*				
	Tensile properties			Flexural properties	
	Modulus (MPa)	Strength (MPa)	Deformation at break (%)	Modulus (MPa)	Strength (MPa)
EP	34 ± 10[a]	9.7 ± 3.3[a]	90 ± 15.3[a]	83 ± 24[a]	2.3 ± 0.6[a]
EP-Fique Powder	925 ± 200[b]	8.8 ± 0.6[a]	2.8 ± 1.1[b]	594 ±85[b]	12.4 ± 1.2[b]
EP-Fique non-woven	1074 ± 198[b]	16.6 ± 1.7[b]	3.8 ± 0.8[b]	390 ± 65[b]	12.9 ± 0.2[b]
EP-Fique unidirectional	2000 ± 414[c]	36.6 ± 4.3[c]	9 ± 0.8[c]	1014 ± 159[c]	21.2 ± 4.6[c]

a–b) Different letters in the same column indicate significative differences ($p < 0.05$).
*Mean of five replications ± standard deviation.

Usually, biocomposites with higher aspect ratio (L/D) may contribute to higher reinforcing efficacy because the contact between the reinforcing elements and the matrix occur over a larger surface. Among the Fique mats, the unidirectional mat exhibit the better mechanical properties. Previous studies suggested that at an orientation of 0 °(parallel to the applied load) biocomposites possessed a much longer fiber structure due to minimal fiber breakage that aiding in strengthening the biocomposite structure by a homogeneous distribution of the load (Radzuan, 2020). Finally, this work opens the possibility of considering non-woven Fique fibers as a reinforcement material with a high potential for the manufacture of thermoset biocomposites for automotive and building applications.

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ID 273
LINEN BEDDING: CHARACTERIZATION AND COMFORT PROPERTIES

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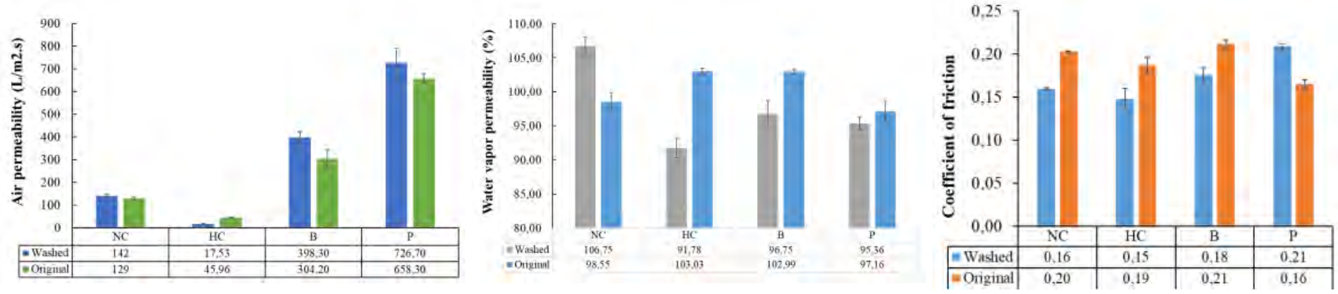
ABSTRACT
The quality of linen bedding is perceived in the comfort of contact and use of the product. Therefore, the thermophysiological and sensorial properties of the fabrics become crucial for the success of these products in the market. This study aimed to carry out the analysis of bedding linen made of plain fabrics (cotton and flax), based on the evaluation of thermal properties, air, and water vapor permeability properties, coefficient of friction of textiles, and others, to understand the comfort standard in bedding linen. It is concluded that linen fabric "P" presents greater air permeability, while cotton fabric "NC" shows better results in water vapor permeability, lower friction coefficient, and higher threads per inch, being an identifier for comfort in bedding linen.

INTRODUCTION
Choosing home textile products, the four main factors that consumers evaluate when purchasing bedding linen are: size, softness, durability, and brand, remaining the most important and unchanged over the years. The most popular factors are the value added of the product and the 100% cotton fabric, or a natural fiber such as flax or silk. Consumers are increasingly aware of the importance of thread count in fabrics concerning the quality of the final product (Das, 2010; Laktim, 2018). Researchers investigated that when people slept in comfortable beds, their average skin temperature was higher than when they slept in an uncomfortable bed, and their lower body skin temperature, sleep efficiency, and percentage of deep sleep were also higher (Chanda et al., 2020; Sundaresan et al., 2016). The thermal insulation and air transfer values for bedding linen make by flax fabrics are higher than those of cotton fabrics with similar fabric weight. The 100% flax fabric has high moisture transfer, compared to cotton fabrics. The high-water absorption capability of flax fabrics provides a temperature regulation of the human body that is a decisive factor for comfort. Flax fibers dry very quickly the use of cotton for bedding products is common in the market (Bilen, 2021; Longhi & Merino, 2020). The ability of a fabric to transport water vapor is an important determinant of physiological comfort and the low moisture passing ability of fabric causes an uncomfortable sensation (Bilen, 2021).
The aim of this study is the analysis of the fabrics' properties and their relation to linen bedding comfort. The fabric samples, all from "Trousseau" linen bedding brand, are named: HC (100 % Cotton); P (100% Flax); NC (100 % Cotton); and B (100 % Cotton). The test methods, which are related to comfort, for fabrics and threads, were: thread count per inch, count number, weight, fabric thickness, air and water vapor

permeability, optical microscopy, pilling formation, and coefficient of friction of textiles (Frictorq).

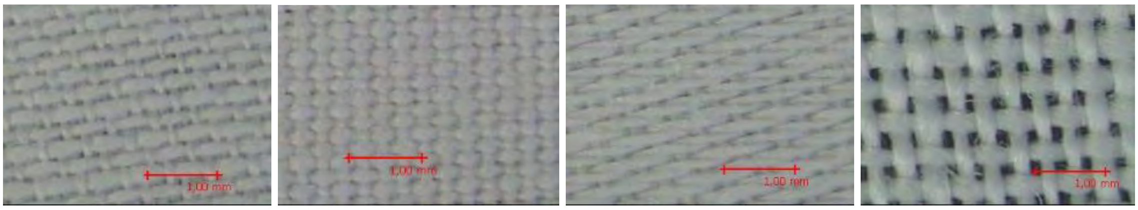
RESULTS AND CONCLUSIONS

Fig.1 shows significant differences between the original product and after washing in terms of air and water vapor permeability test, and Frictorq test for samples before and after washing. Fig. 2 presents the optical microscopy of samples.



(a) (b) (c)

Fig.1 Realized tests of NC, HC, B, and P: (a) Air permeability, (b) water vapor permeability, (c) Frictorq test (coefficient of friction).



NC HC B P

Fig.2 Optical microscopy (1mm).

Fabric "P" (100% flax) has the highest air permeability (726.70 L/m2/s) (Fig.1a) compared to other cotton fabrics. Optical microscopy (Fig. 2) confirms the greater opening in the interweaving of the "P" fabric, confirming greater air permeability, including a lower count of threads per inch (105/inch). The "NC" cotton fabric (106.75 g/m²*day) showed the highest value among the others in terms of permeability to water vapor after washing, the coefficient of friction (Fig.1c), showing one of the lowest values (0.16), providing better touch, but has low air permeability, due to the greater count of threads per inch (290/inch).

It could be concluded that the "P" fabric has greater air permeability, related to the more open ligament, while the "NC" fabric shows better properties in the other tests performed and, according to the literature, the permeability to water vapor is one of the main requirements in the identification of comfort, being an identifier of the best quality among those analysed.

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ID 275
TEXTILE VEGETAL FIBERS CHARACTERIZATION BY FTIR AND MULTIVARIATE STATISTICAL ANALYSIS

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ABSTRACT
The characterization and identification of textile fibers from various materials are important for industrialization, recycling, recognition of objects in museum collections, forensics, and development of new materials such as composites. This characterization demands mainly the identification of the physical and chemical properties of the material. This study aimed at the characterization and differentiation of seven different textile vegetal fibers employing the non-destructive Fourier Transform Infrared (FTIR) analytical technique in conjunction with multivariate statistics PCA (Principal Component Analysis). The results indicated that, even with very similar chemical compositions, the materials were discriminated against and separated into groups. In this way, these findings are promising and are allowing to confirm the strategy of using the multivariate statistical analysis associated with the FTIR data allows the identification of different materials constituted of vegetal textile fibers.

INTRODUCTION
The characterization of textile materials by non-invasive analytical techniques is an advantage for several areas such as forensic science, the recycling industry, and museology. In textile recycling, the correct identification of the fibers is necessary to guarantee the quality of the final product. The characterization of vegetable fibers allows for knowing their main constituents (cellulose, hemicellulose, and lignin). However, the identification and discrimination between different vegetable textile fibers using only the FTIR technique is difficult due to the great similarity of the chemical composition of the fibers; even after alkaline treatment processes, the spectra of the fibers remain very similar (Saito, 2021).

However, the use of multivariate statistical treatment on FTIR spectra allows for distinguishing textile fibers of different chemical compositions (Makela, 2021; Quintero Balbas et al., 2022; Saito et al., 2021; Zapata et al., 2022). In this way, infrared spectroscopy associated with the PCA treatment has allowed distinguishing different fibers and fiber blends in tapestries composed of cotton, silk, wool, cotton with wool, cotton with silk, and silk with wool (Quintero Balbas et al., 2022).

The present study aimed to analyze 7 different vegetal textile fibers: 1 and 2) cotton (Gossypium hebaceum) and kapok (Chorisia speciosa), seed fibers mostly composed of cellulose; 3, 4 and 5) jute (Corchorus capsularis), flax (Linum usitatissimum) and hemp (Cannabis sativa), bast fibers; 6 and 7) tucum (Astrocaryum chambira) and tururi (Manicaria saccifera), Amazonian palm fibers, respectively from leaves and fruit sacs; employing FTIR analysis (Brucker, Alpha model equipment, ATR with ZnSe crystal, 4000 – 600 cm-1 range, 4 cm-1 resolution, 64 scans).

Data compression without information loss is possible because to the extraction of spectral features using the PCA (Principal Components Analysis) statistical technique, which also reduces the database's dimensionality. The data were initially normalized using only the conventional normalization (standardscaler), and then the functions were applied to produce the main components. The PCA statistical test was carried out in the Python language using the Jupyter Lab program.

RESULTS AND CONCLUSIONS

The FTIR analysis of the group of seven different fibers with very similar chemical composition (cotton, kapok, jute, flax, hemp, tucum, and tururi) did not enable to distinguish between them only by recording the spectra and its direct comparison (Fig.1a). However, these spectra information analyzed by PCA statistics, generated new data, which grouped differently according to the origin fiber. This was more evident for tururi and kapok fibers. (Fig. 1b).

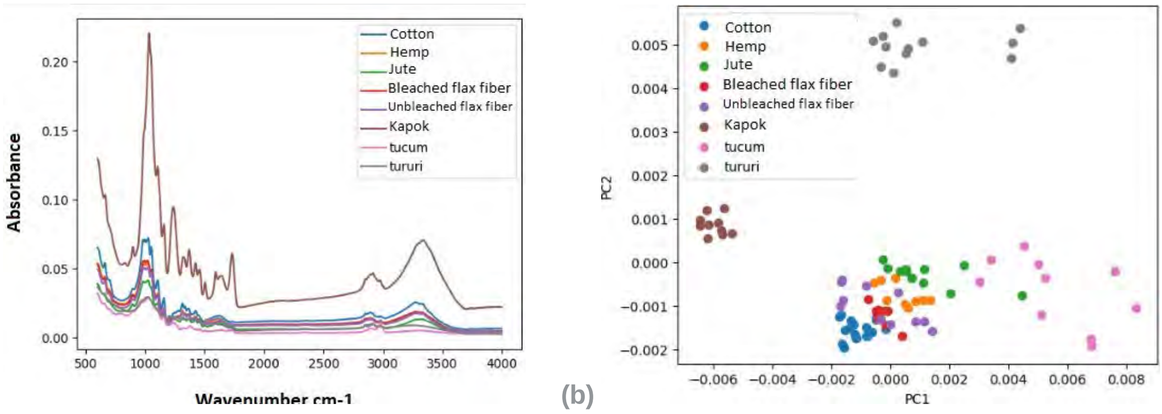

Fig.1 (a) FTIR absorption spectra, ATR mode, of seven textile fibers (cotton, kapok, jute, flax, hemp, tucum, and tururi); **(b)** PCA statistical analysis for the FTIR spectra data, grouping differently according to the origin fiber.

The treatment with multivariate statistics was efficient to distinguish textile fibers of similar chemical composition, which would be little or almost impossible to distinguish only by analyzing the spectra without statistical treatment. Thus, the association of multivariate statistical analysis to the data obtained by FTIR has shown to be potentially promising for the identification and differentiation of textile fibers with very similar chemical composition, such as vegetable fibers, regenerated fibers, and others.

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ID 279
NANOCELLULOSE PRODUCED FROM LIGNOCELLULOSIC CROPS AS REINFORCEMENT AGENTS IN BIOBASED FILMS: DEVELOPMENT AND CHARACTERIZATION

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ABSTRACT
The aim of this work was to optimize and test nanocellulose obtained from two different lignocellulosic biomass (sorghum, *Sorghum bicolor* (L.) Moench and sunn hemp, *Crotalaria juncea* L.) as reinforcement agents in chitosan films. Nanocellulose (NC) was produced via an alkaline pre-treatment approach applied into the different biomasses, followed by acid hydrolysis. The nanoparticles obtained from sorghum and sunn hemp were incorporated in chitosan films at a rate of 2.5% w/w, and the resultant bionanocomposites (Sorghum NC films and sunn hemp NC films) were fully characterized. The results confirmed that sorghum NC, improved the strength and stiffness of the chitosan biopolymer and that sunn hemp NC improved the plasticity of the chitosan polymer. Hence, results indicate that those lignocellulosic crops may afford a source of NC for the production of bionanocomposites.

INTRODUCTION
Lignocellulosic feedstocks are rich in cellulose, hemicellulose, and lignin which can give rise to innovative value-added products. Cellulose can be isolated from the lignocellulosic fibers and depolymerized to nanocellulose (NC), a nano-bio-based material with applicability in diverse technological areas (Pires et al., 2019a). Yet, the recalcitrance of the lignocellulosic material, requires pre-treatments, which can be more (e.g. use of ionic liquids) or less sustainable (chemical processes). The insertion of these scattered particles, as reinforcement agents, into bio-based polymers, is seen as a promising option to surpass the shortcomings associated with bioplastics (mechanical, thermal and barrier properties)(Pires et al., 2019b). Therefore, the aim of this work was to optimize the NC production from two different lignocellulosic biomass (sorghum, *Sorghum bicolor* (L.) Moench and sunn hemp, *Crotalaria juncea* L.) and to test it as reinforcement agents in chitosan films. From sorghum, two different NC were prepared, from inner and outer layer, as the original stems were divided in these two parts. Nanocellulose was produced via an alkaline pre-treatment approach applied to the two different lignocellulosic biomasses, followed by acid hydrolysis. In the alkali pre-treatment process optimization, some parameters were tested, namely time of reaction and temperature. The produced nanocelluloses were incorporated in chitosan at the rate of 2.5% w/w and

the bionanocomposites were characterized: mechanical properties, thickness, optical properties (opacity and transparency), surface color, and solubility and swelling degree. Commercial nanocellulose at the same rate was also tested in the chitosan films for comparison. Pristine chitosan film was the control.

RESULTS AND CONCLUSIONS

Taking into consideration a scale up of the process, the time of 3h and temperature of 60ºC were chosen as optimum for the process. Bionanocomposites made from sorghum and sunn hemp NC were slightly more saturated and opaque than the pristine chitosan films. In terms of solubility, no differences were observed among pristine films of chitosan and films reinforced with sunn hemp and sorghum NC. Films reinforced with sorghum and sunn hemp NC presented a higher swelling degree than pristine chitosan films - incorporation of those NC's in the chitosan matrix created a porous material with free -OH and free -NH3 from chitosan, and also from the NC, where the water molecules can infiltrate. The higher swelling degree attributed to the incorporation of NC from sunn hemp and sorghum, may be interesting for the production of hydrogels - interaction between chitosan and the NC's was not hydrophobic. The introduction of sorghum NC did not result in a significant change of the water vapour permeability (WVP), compared with pristine chitosan, and the insertion of sunn hemp NC increased the WVP. Those results obtained with sunn hemp NC and sorghum NC, can be explained by the hydrophilicity of the NC and of the bionanocomposites produced with those NC's. This hydrophilicity thus help the transport of water molecules through the film. Sunn hemp NC films also showed a slightly higher thickness than sorghum NC films and pristine chitosan films. Further, the results confirmed that sorghum NC, improved the strength and stiffness of the chitosan biopolymer and that sunn hemp NC improved the plasticity of the chitosan polymer (Table 1). Hence, results indicate that those lignocellulosic crops may afford a source of NC for the production of bionanocomposites. Considering the application of those bionanocomposites by the food packaging industry, sorghum NC - chitosan films showed more promising results than sunn hemp NC – chitosan films.

Table 1 Mechanical properties of the different chitosan films

	Tensile Strength (MPa)	Elastic Modulus (MPa)	Elongation at Break (%)
Chitosan + Inner Sorghum	35.1 ± 1.8	1722 ± 88	17.1 ± 1.9
Chitosan + Outer Sorghum	36.7 ± 3.1	1565 ± 110	19.1 ± 3.7
Chitosan + Sunn Hemp	32.2 ± 1.8	1356 ± 121	29.9 ± 3.2
Chitosan + NC commercial	37.2 ± 2.3	2035 ± 107	10.9 ± 2.2
Chitosan	34.6 ± 3.7	1415 ± 94	27.9 ± 1.6

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ID 282
FIBROUS BIOCOMPOSITES FROM NETTLE (GIRARDINIA DIVERSIFOLIA) AND POLY(LACTIC ACID) FIBERS FOR AUTOMOTIVE DASHBOARD PANEL APPLICATION

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ABSTRACT
This work deals with fibrous biocomposites prepared by using nettle and poly(lactic acid) fibers and employing carding and compression-moudling processes. The role of carding process in determining the tensile strength of the biocomposites was analyzed. The tensile, bending and impact properties of the biocomposites were found to increase initially with the increase of nettle fiber content till 50 % by weight and decrease afterwards. The thermogravimetric analysis inferred that the biocomposites were thermally enough stable and their thermal stability increased with the increase of nettle fiber content. The dynamic mechanical analysis suggested that the biocomposites were extremely good in terms of storage modulus, loss modulus, and damping factor. Further, the biocomposites exhibited excellent biodegradability and their biodegradability increased with the increase of nettle fiber content. Overall, the biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) showed high potential for automotive dashboard panel application.

INTRODUCTION
Fibrous biocomposites, consisting of biofibers reinforced with biomatrix, is a bioproduct that is derived from renewable sources, stable in its desired lifetime, and fully biodegradable after disposal in composting environment. They are non-toxic, relatively cheap, easily processable, and fully biodegradable, they offer high strength-to-weight ratio, and they can be recycled to reduce the carbon footprint of materials. A biocomposite prepared from nettle fibers as reinforcement and poly(lactic acid) as matrix. In this work, the tensile strength, Young's modulus, and flexural modulus of the biocomposites were determined. It was concluded that nettle had in principle the same potential as other bast fibers to act as reinforcement in poly(lactic acid). However, a large difference was reported for the mechanical properties of nettle fibers as compared to those of biocomposites. There was no attempt made to improve the mechanical properties of the biocomposites by chemical treatment on nettle fibers. Also, the performance of the biocomposites was not evaluated in terms of dynamic mechanical analysis, thermogravimetric analysis, and biodegradability. It is necessary to examine these behaviors of biocomposites when the intended application is automotives.

RESULTS AND CONCLUSIONS

A series of fibrous biocomposites, prepared by using alkali-treated nettle fibers and poly(lactic acid) fibers in different weight proportions and employing carding and compression-moudling processes, was evaluated for static and dynamic mechanical properties, thermogravimetric behavior, and biodegradability. The role of carding process in determining the tensile strength of the biocomposites was analyzed. It was observed that at higher cylinder speed, the tensile strength of the biocomposites increased, but at the cost of higher width-wise shrinkage of fiberwebs. The increase in tensile strength at higher cylinder speed was attributed to the preferential alignment of more fibers along the longitudinal axis (direction of applied load) of the biocomposites. The tensile, bending, and impact properties of the biocomposites initially increased with the increase of nettle fiber content and decreased afterwards. The maxima of these properties were obtained at equal weight proportion of nettle and poly(lactic acid) fibers. The thermogravimetric analysis inferred that the biocomposites were thermally enough stable and their thermal stability increased with the increase of nettle fiber content. The dynamic mechanical analysis suggested that the biocomposites were extremely good in terms of dynamic mechanical properties. Further, the biocomposites exhibited excellent biodegradability and their biodegradability increased with the increase of nettle fiber content. Overall, the biocomposite prepared with equal weight proportion of nettle and poly(lactic acid) showed high potential for automotive dashboard panel application.

(a) (b) (c)

Microscopic images of tensile failure of biocomposites prepared with 10 % **(a)**, 50 % **(b)**, and 90 % **(c)** nettle fibers by weight

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ID 283
FUNCTIONALIZATION OF BIOBASED POLYMERS WITH NANOCELLULOSE AND ESSENTIAL OILS: DEVELOPMENT AND CHARACTERIZATION

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ABSTRACT
The aim of this work was to develop bionanocomposites based on chitosan incorporated with nanocellulose as a reinforcement agent and two different essential oils (*Aloysia citrodora* and *Cymbopogon citratus*) and to evaluate its bioactivity via *in vitro* assays. Films were produced by casting and commercial nanocellulose (NC) and *Aloysia citrodora* EO (ACEO) or *Cymbopogon citratus* EO (CCEO) were incorporated. The release process of the active compounds present in the bio-based polymer was monitored by a migration assay in different simulants (water, ethanol 10%, ethanol 50% and ethanol 95%). Total phenolic compounds and their antioxidant activity were measured at the simulants over time. The antioxidant activity of the active compounds was maintained even after the diffusion process, being higher with ACEO than with CCEO. The diffusion coefficient of ACEO was higher than the CCEO, and the nanocellulose entrapped the phenolic compounds, retarding its release. In conclusion, the incorporation of EOs conferred antioxidant activity to the bio-based polymer. Functionalization of the films is being studied also in terms of their antimicrobial activity.

INTRODUCTION
Bioplastics are considered an environmentally friendly alternative to the non-biodegradable materials from non-renewable resources. They can contribute to reduce the global waste disposal problem by reducing the consumption and the accumulation in the environment of traditional petroleum-based polymers. Biopolymers, such as chitosan, alginates or proteins, have been investigated to substitute synthetic polymers. Essential oils (EOs) are extracted from a variety of crops, e.g. rosemary, ginger, thyme, to mention a few, and are rich in active compounds, such as phenolic acids and terpenes, which confer them biological activities (antimicrobial and antioxidant). Thus, EOs have the potential to substitute synthetic additives, and when incorporated into polymers, may enhance their activity and functionality. The rich content in phenolic compounds and terpenes confer to EOs remarkable bioactivity, namely antimicrobial properties against microorganisms (through destabilizing the phospholipid bilayer of the cell membrane, enzyme system and genetic material of bacteria) (Souza et al., 2019) and antioxidant activity (by stabilizing free-radical and reactive oxygen species - ROS, chelating metals and blocking UV-light) (Souza et al., 2018). Therefore, the aim of this work was to develop bionanocomposites based on chitosan incorporated with nanocellulose

as reinforcement agent and with two different essential oils (*Aloysia citrodora* and *Cymbopogon citratus*) and to evaluate its bioactivity via *in vitro* assays. Films were produced by casting, from the chitosan film form solution (FFS) (1.5% w/v). Commercial NC was incorporated at the level of 2.5% (w/w chitosan) for mechanical reinforcement. *Aloysia citrodora* EO (ACEO) or *Cymbopogon citratus* EO (CCEO) were incorporated in the proportion of 1% (v/v FFS). Films were casted in glass molds and naturally dried. Films without EO and NC were used as control. The release process of the active compounds present in the film was monitored by a migration assay in different simulants (water, ethanol 10%, ethanol 50% and ethanol 95%). Total phenolic compounds and their antioxidant activity were measured at the simulants over time (10 days).

RESULTS AND CONCLUSIONS

Results show that the phenolic compound's diffusion to the simulant media was higher with ACEO than with CCEO. The antioxidant activity of the active compounds was maintained even after the diffusion process, being higher with ACEO than with CCEO. The diffusion coefficient of ACEO was higher than the CCEO, and the nanocellulose entrapped the phenolic compounds, retarding its release. Higher antioxidant activity was also noticed in simulants with low amount of ethanol (10%). In conclusion, the incorporation of EOs conferred antioxidant activity to the bio-based polymers. Functionalization of the films is being studied also in terms of their antimicrobial activity.

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ID 286
ENVIRONMENTALLY BENIGN ALGINATE EXTRACTION AND FIBRES SPINNING FROM DIFFERENT EUROPEAN BROWN ALGAE SPECIES USING NATURAL CROSSLINKERS

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ABSTRACT
To support the growing demand of natural fibres extensively in various applications, sustainable alternatives for their extraction and fibre formation are needed. Four different Sodium alginate species from European seaweed- Saccharina latissima (SAC), Laminaria digitata (LAM), Sacchoriza polyschides (SACC), and Himanthalia spp. (HIM) using sustainable protocol were extracted and were investigated to produce fibre. After extraction (3% w/v biomass) using citric acid based sustainable protocol, crude alginate represented 61-65 % of the dry biomass weight for SAC and LAM, and 34-41 % for SACC and HIM when experiments were performed at lab scale (1.5 g of starting material). Interestingly, decreased yields to 26-30 % was observed when scaling-up extraction (60 g of starting material). SAC and LAM alginates had the highest molecular weights and M/G (mannuronic acid/guluronic acid) ratios when compared to those from SACC and HIM (MW: 302 and 362 kDa, M/G:1.98 and 2.23, vs 268 and 168 kDa, 1.83 and 1.86). To study the spinnability and mechanical properties, the four types of alginates were cross-linked with $CaCl_2$, only SAC and LAM alginates were spinnable. These fibres showed no clumps or cracks and were uniform under stretching action and presented a similar Young's modulus (2.4 and 2.0 GPa). We have demonstrated successful spinning of alginate extracted from S. latissima and L. digitata into functional fibres cross-linked with $CaCl_2$. The mechanical tests performed allowed to identify significant differences on the mechanical properties of the fibres. Future work involves the cross-linking of these fibres using natural cross-linkers to tune the mechanical properties of the fibres for varied applications.

INTRODUCTION
Seaweeds are composed of more than 50% of carbohydrates and polysaccharides (Rioux & Turgeon, 2015). Structurally,a linear copolymer block of alginates consist of 1,4-linked β-d-mannuronic acid (ManA) and α-l-guluronic acid (GluA), that can be arranged in homopolymeric (GG and MM) and heteropolymeric (MG) blocks. Alginate based materials encompass a wide range of end applications. Fibres produced of these polysaccharides are currently used in cosmetic sector, medical, skin care, and hygienic products(Qin et al., 2017).

This study aimed to investigate the sustainable protocol for alginate extraction from brown seaweeds that can be farmed in European coastal waters, and their potential use to manufacture functional fibres. To achieve this goal, four different brown seaweed species, Saccharina latissima, Laminaria digitata, Sacchoriza polyschides, and Himanthalia spp., were extracted and analysed . The extracted alginate fibres were checked for their spinnability and the spinnable fibres were produced via wet spinning, and their mechanical properties were assessed. Characterization of alginate fibres obtained from different brown algae allowed to distinguish the most appropriate brown seaweed feedstock for the manufacturing of functional fibres.

RESULTS AND CONCLUSIONS

The results determined from NMR in Table 1 indicate that M (FM) was the main component of alginates in all four species. Values of η parameter lower than 1 indicates abundant homopolymeric block. SAC, LAM, SACC and HIM alginates were characterized by ManA/GulA ratios of 1.18–2.32 and η < 1 with abundance of F_{MM} blocks (Silva et al., 2023).

Table 1: Composition of extracted alginates determined by NMR. FG: guluronic acid ratio; FM: mannuronic/guluronic acid ratio; M/G: mannuronic/guluronic acid ratio; FMM: mannuronic homopolymeric blocks, FGG: guluronic homopolymeric blocks; FMG: heteropolymeric blocks mannuronic/guluronic acid; FGM: heteropolymeric blocks guluronic/mannuronic acid; η: relative abundance of homopolymeric blocks MM and GG (η = FGM/[FM + FG]), η < 1 with abundance of homomannuronic blocks.

Sample	FG	FM	M/G	FMM	FGG	FGM=FMG	η
SAC	0.35	0.65	1.89	0.25	0.10	0.56	0.10
LAM	0.30	0.70	2.32	0.29	0.01	0.69	0.01
SACC	0.46	0.54	1.18	0.42	0.04	0.50	0.04
HIM	0.34	0.66	1.95	0.28	0.06	0.60	0.06

All four alginates showed coagulation ability in the $CaCl_2$ bath, but only SAC and LAM alginates were spinnable (Table 2). Alginates from SAC and LAM showed coagulation ability and good spinnability These results correlate with the content of GulA and ManA, the ManA/GulA ratio, and MW obtained for the four species (Table 1). It is well known that the viscosity increases with the molecular weight (Jiao et al., 2019).

Table 2: Coagulation and spinnability of extracted alginates.

Type of alginate	Coagulation	Spinnability
SAC	□	Good
LAM	□	Good
SACC	□	Poor
HIM	□	Poor

The stress/strain curves obtained from the tensile test are presented in Table 3. Fibres produced from LAM and SAC alginates showed a linear response, followed by plastic deformation and net failure. A similar behavior has been previously observed for other biopolymer fibres (Foroughi et al., 2011; Li et al., 2013; Sahari, J., Sapuan, S. M., Ismarrubie, Z. N., & Rahman, 2012). The stretching action caused by the wet-spinning process produces fibres with high molecular alignment (Qin, 2008). As a result of this improved molecular alignment, the mechanical properties of SAC and LAM alginate fibres such as the Young's modulus and tensile strength (Table 3) were better than other biopolymers such as gum arabic, starch film, polyhydroxybutyrate, and carboxymethyl cellulose (which are not in fibrous form) (Aji et al., 2020; Marinho et al., 2015) (Figure 1).

Table 3: Mechanical properties of alginate-derived fibres.

Fibre type	Average Youngs Modulus (GPa)	Average Tensile strength (MPa)	Average Strain (%)	Average Diameter (mm)
LAM	2.0 (±0.3)	26.4 (±5)	12.6 (±3)	0.122 (±0.011)
SAC	2.4 (±0.19)	32.8 (±7.7)	9.4 (±1.6)	0.124 (±0.013)

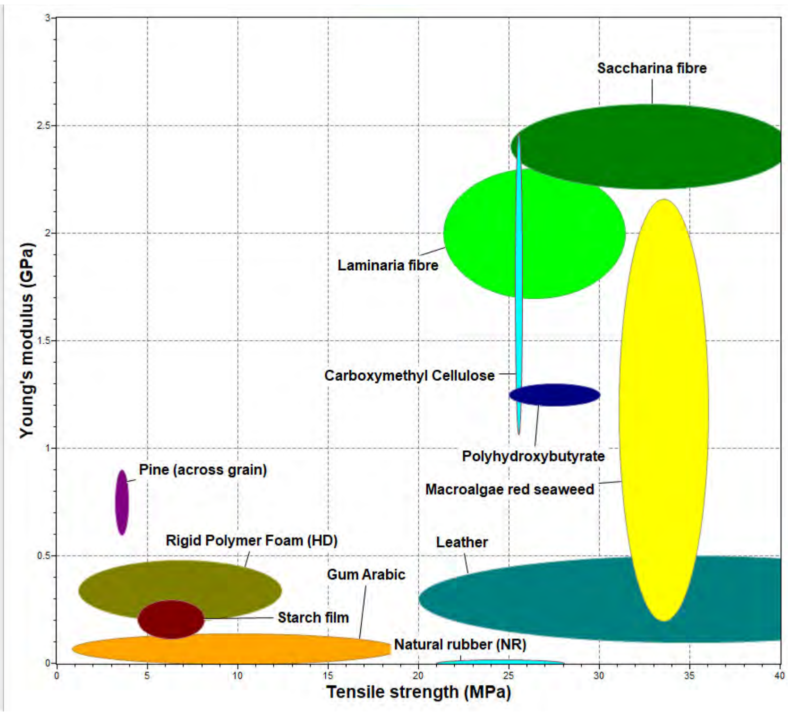

Figure 1: Comparison of the Young's Modulus and tensile strength of Laminaria and Saccharina alginate with different polymers

This study shows that alginates extracted from different species of brown algae show potential of European farming for seaweeds. Qualities of the derived fibres were affected by the different G/M ratio, physical and mechanical properties of the fibres. Variation of their mechanical properties would allow a better control of their structural integrity and biodegradability to expand their uses in the manufacturing of textile and other industries. Future work includes the use of different natural cross-linking agents to enhance tensile strength of alginate fibres.

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ID 287
MICROPLASTIC SUSTAINABLE REMEDIATION BY HYDROGEL OF NATURAL BACTERIAL FIBERS

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ABSTRACT
Currently, there is a growing environmental concern related to microplastics (MPs), that comes with the global increase in plastic production. Microplastic morphotypes such as beads and films have been a source of environmental concern for a long time, however, microfibers (MFs) are also an important source of microplastic contamination. Natural extracellular polymers, secreted by bacterial species, such as bacterial cellulose (BC) have been proven to have potential in a variety of fields. So, in this work, the bioflocculant activity of bacterial cellulose hydrogel (BCH) was tested in order to remove MPs and MFs from contaminated waters. The BCH exposed to MPs- and MFs- highly contaminated waters (2g/L) showed very high flocculation rates of 88.6% and 99.6%, respectively. The MPs and MFs were highly retained and adsorbed in the biopolymer network. The effective bioflocculant activity is most likely caused by both the BC's microporous nature and the morphological characteristics of the pollutants. These results show that BCH is an effective bioflocculant and a potential replacement for synthetic flocculants in wastewater remediation processes, offering a high-performing, environmentally friendly, and sustainable option.

INTRODUCTION
Microplastics (MPs) comprise of plastic particles with a diameter inferior to 5 nm (Arthur et al., 2009). Moreover, they can adopt different shapes like beads, fibers (MFs), foam, and fragments (Bayo et al., 2020; Vethaak et al., 2021). Microfibers (MFs) are a type of MPs and they have been identified in all aquatic environments around the globe (Singh et al., 2020). The microfibers are often the result of the breakdown of larger synthetic textile items, such as clothing and carpets. MPs and MFs are particularly concerning because of their ability to accumulate in the food chain. Many marine organisms, from plankton to fish, mistake these particles for food, leading to the ingestion of plastic by animals that humans consume. This can have harmful effects on both human health and the health of ecosystems. The presence of MPs and MFs in the environment has led to growing concern and research into potential solutions for their removal. In recent years, the development of green technologies for the removal of MPs has been extensively studied. Bacterial cellulose (BC), a natural extracellular polymer produced by bacterial species belonging to the Komagataeibacter genus, has shown applicability and further promise in a wide range of fields (Mendonça et al., 2023). Therefore, in this work, the potential of BC hydrogels as bioflocculants and hetero-aggregators of MPs and MFs was evaluated, towards their prospective removal from contaminated WWTP (waste

water treatment plant) waters.

RESULTS AND CONCLUSIONS

Using the response surface methodology (RSM), BCH revealed a very high MPs and MFs removal rate, up to 88.6% (Figure 1A) and 99.6% (Figure 1B), respectively. Both contaminants were retained in the hydrogels' network, with a substantial reduction in the number of particles outside the hydrogel. The effective MPs and MFs removal activity is likely caused primary by the BC's microporous nature (which may lead to adsorption and electrostatic interactions) and by the morphological characteristics of the contaminants. Therefore, BCH shows to be an effective bioflocculant.

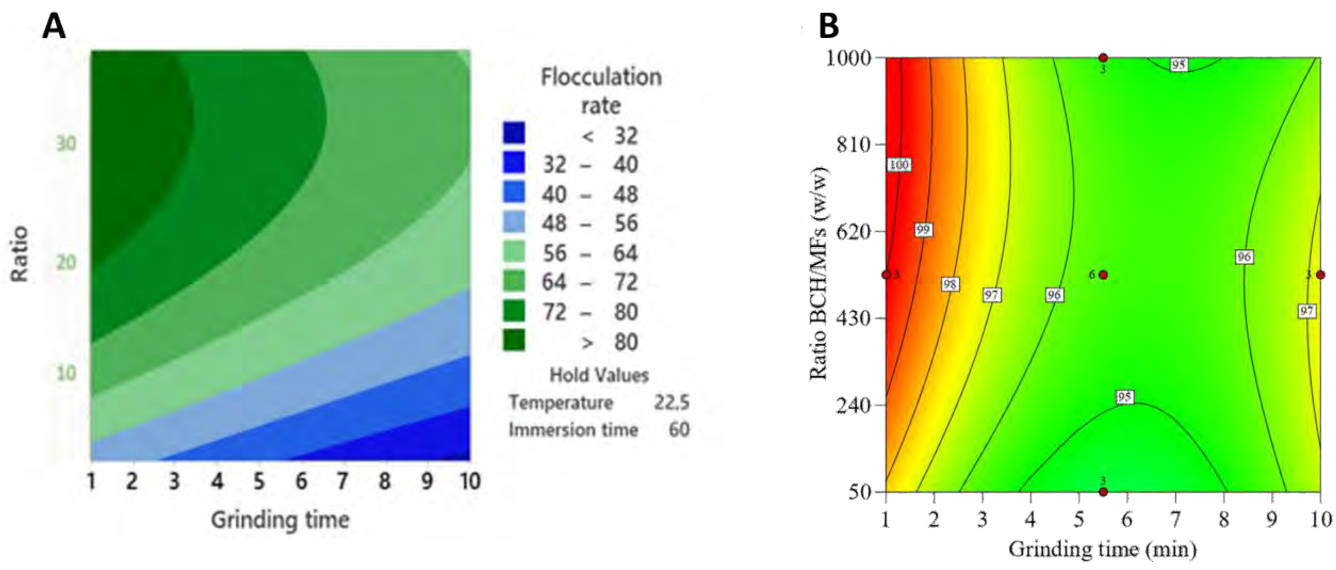

Figure1. Contour plot: flocculation rate of BCH and (A) MPs and (B) MFs.

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ID 288
NATURAL MICROBIAL FIBRES FOR EMERGENT CONTAMINANTS REMOVAL

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ABSTRACT
Nowadays, microplastics (MPs) and synthetic microfibers (MFs) are becoming more prevalent in the environment, which leads to an accumulation in the food chain due to their consumption by many ecologically relevant organisms. Following the need to develop sustainable solutions for the removal of these contaminants, bacterial cellulose (BC) biopolymers were building. BC membranes present distinctive characteristics, such as a hydrated ultrafine three-dimensional nanofibril structure and high porosity, for biotechnological applications, in a wide range of fields. Diligently, BC biopolymer membranes were used to evaluate their viability for the removal of both MPs and MFs from highly contaminated waters. Results show that BC membranes are able not only to capture MPs and MFs, displayed by very high removal efficiencies but also firmly imprison the micropolymeric particles on and within the network. Thus, it is clear from this study that BC biopolymers have massive potential as a viable, and biosustainable solution to membrane-based water treatment practices.

INTRODUCTION
As important as microplastic morphotypes such as beads and films are to the environment, microfibers (MFs) are among the most important sources of microplastic contamination, since they represent the highest abundance of plastic in marine environments, wastewater affluents and are found as the most abundant MPs in the animal's stomachs (Acharya et al., 2021; Walkinshaw et al., 2020).
Bacterial cellulose (BC) is a naturally occurring extracellular polymer that is formed as a byproduct of bacterial metabolic activities (Blanco Parte et al., 2020; Rangaswamy et al., 2015).
Several scientific and industrial interests have been generated by this biopolymer because of its wide range of morphologies, physicochemical characteristics, and applications (Wang et al., 2019). As a result of the necessity to create long-term solutions to remove pollutants from contaminated waters, BC biopolymer membranes were evaluated for their viability in removing both MPs and MFs from severely contaminated waters.

RESULTS AND CONCLUSIONS
The removal rate of MPs and MFs by BC was shown to be exceptionally high - up to 99%. Furthermore, the contaminant particles were trapped within the biopolymers network (Figure 1), and the amount of contaminants remaining in the contaminated water was considerably decreased. The microporous structure of BC is able to filter out MPs and MFs efficiently as a result of adsorption and electrostatic interactions, as well as by the morphological attributes of the pollutants.

Figure 1. Representative top-view SEM image of **(A)** MPs and **(B)** MFs, adsorbed and embedded in the BC membrane surface.

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ID 289
MICROBIAL FIBERS AS A BIOFLOCULANT TO HARVEST AND STORAGE VEHICLE FOR LIVING MICROALGAL CELLS

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ABSTRACT
The industrial-scale harvesting and stockpiling of living microalgal cells can be challenging due to various factors. The use of cellulose-producing bacteria in the production of hydrogels has been studied extensively in recent years in many domains. These hydrogels have a propensity to create dense flocs that enable the net capture and bridging of microalgae cells into the network, subsequently flocculating and settling them. By exploring the scalability of bacterial cellulose for harvesting microalgae cells and keeping them growing in a scaffold, we have discovered that different marine and freshwater microalgae can be remaining active. The growth and survival of the bacterial cellulose scaffold were tested under different abiotic conditions, demonstrating that bacterial cellulose hydrogel is a viable storage vehicle for living microalgal cells. Overall, the use of BC microbial polymers holds great promise for a sustainable and eco-friendly future to harvest and stock living microalgal cells.

INTRODUCTION
Bacterial cellulose (BC) is a smart and powerful natural biopolymer. Due to its inherent purity, biodegradability, biocompatibility, and non-cytotoxicity, BC is an incredibly versatile biomaterial that has a commercial interest (Gregory et al, 2021). Its environmentally friendly biosynthesis produced by Komagataeibacter saccharivorans, makes it an interesting biopolymer to be applied in different fields, from bioremediation to biomedicine applications (Faria et al, 2022; Swingler et al, 2021).
The commercialization of microalgae as a food source has gained popularity in recent years, driven by its high nutritional value and potential health benefits. Microalgae are rich in protein, fiber, vitamins, and minerals, making them an excellent source of nutrients for human consumption. In addition to its nutritional benefits, the commercialization of microalgae as a food source has several environmental benefits: microalgae are highly efficient at photosynthesis, making them an excellent candidate for sustainable food production. Additionally, the cultivation of microalgae requires minimal water and land resources. However, there are still challenges associated with the commercialization of living microalgae as a food source. Harvesting and stockpiling living microalgal cells pose several challenges. Harvesting microalgal biomass from the growth medium has been a difficult step during microalgal biomass production due to its negatively charged surface area and small size (Yang et al, 2021). Commonly, synthetic organic and inorganic floc-

culants were used, and BC comes as a sustainable alternative due to its unique physicochemical properties and the non-toxic and high wettability of BC can allow microalgal cells to grow within the BC hydrogel. A significant challenger is preserving the viability of living microalgal cells. Microalgae require specific conditions to remain viable, including low temperatures, stable environments, and adequate nutrient levels. However, maintaining these conditions for an extended period can be challenging, particularly in large-scale operations, which can result in a high mortality rate. This work shows news perspectives when BC is used as a scaffold or flocculant agent.

RESULTS AND CONCLUSIONS

RSM design model was employed to maximize and optimize the harvesting of microalgae cells: grinding time of 5.5 min; immersion time of 60 min.; BC concentration of 1000 mg and agitation time of 15 min. At these conditions, BC hydrogels show a powerful flocculant activity harvesting about 98.7% of microalgae cells from a culture medium (Figure 1A). In addition, the non-toxicity, high swelling, and wetting capacity of the BC mesh allows microalgae cells to grow inside the cellulose hydrogel (Figure 1B), keeping the microalgae active/viable.

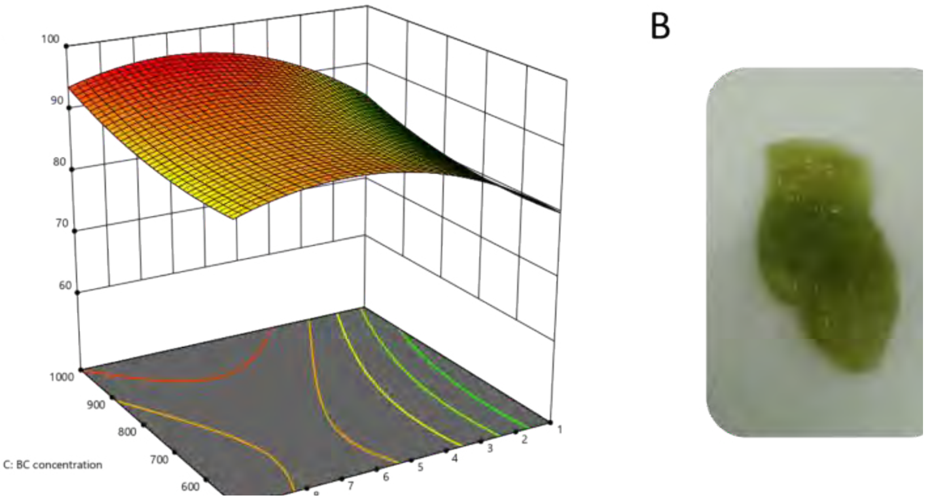

Figure 1. (A) 3D Response surface plot representing the response of the process to BC concentration and BC grinding time; **(B)** stockpiling living microalgal cells in the BC hydrogel.

ACKNOWLEDGMENTS

The financial support has been provided by the Foundation for Science and Technology (FCT) with a PhD grant (BD/6615/2020), through CIIMAR (UIDB/04423/2020 + UIDP/04423/2020) and by the European Territorial Cooperation Programme PCT-MAC 2014-2020 through project REBECA-CCT (MAC/1.1.B/269).

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ID 296
INFLUENCE OF THE USE OF DIFFERENT PLASTICIZERS ON THE MECHANICAL, PHYSICAL AND THERMAL PROPERTIES OF A BIOPOLYMER

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ABSTRACT
This work compares the influence of using two plasticizers on the production of starch-based biopolymer. Glycerol and distilled water were mixed with potato starch and a biopolymer (PBAT) to produce five different samples that were tested in tensile, FTIR, DSC and TGA tests. The mechanical test results show that the sample made with 20% glycerol has better mechanical properties than the samples with distilled water as a plasticizer and the PBAT sample.

INTRODUCTION
Synthetic polymers (produced from petroleum), like PET, PVC, PE, PP, PS and PA, are the most widely used materials globally because of the attractive characteristics inherent to them. However, the excessive usage of these materials has led to enormous environmental problems since, upon their decomposition, they generate microplastics, which can have devastating effects on living beings (Siracusa, 2008). In addition, they take hundreds of years to degrade. With the increasing environmental awareness, an urgent need was triggered to reduce the dependence on synthetic and replace them with bio-based (biopolymers) or biodegradable polymers. Biopolymers are obtained from natural resources and can be produced synthetically or by living organisms. Several alternatives have been explored for the synthesis of biopolymers. One of the most relevant options is the development of starch-based biopolymers since starch is a renewable material of natural origin, vastly abundant, has an accessible cost and can degrade completely in nature in the form of non-toxic or polluting compounds (Ghanbarzadeh, 2011). Due to the characteristics of starch, it is necessary to combine it with plasticizers to improve the polymer's properties, that is, to form thermoplastic starch, TPS. The TPS is blended with other biopolymers like PBAT (Liu, 2009).
A total of 5 samples were produced in a twin-screw corotating extruder with a temperature profile between 165-145°C (die), mixing potato starch, two different plasticizers (glycerol and distilled water) and PBAT. The amount of plasticizer used in the TPS was 10 and 20 % w/w. A ratio of 60:40 (TPS: PBAT), in terms of % of the weight, was used to produce the samples. Each sample was characterized in mechanical (tensile tests (ISO 527)), thermal (DSC (ASTM D3418) and TGA (ASTM E1131)) and chemical tests (FTIR (ASTM E1252)), according to the adequate standards.

RESULTS AND CONCLUSIONS

The results from the tensile tests are shown in Table 1. The results show that the sample made with 20% glycerol has better mechanical properties than the ones made with distilled water as a plasticizer. The FTIR test results in Fig.1 show that all samples have very similar chemical spectrums. The only difference between the samples with and without TPS is the band between 3600 and 3200cm^{-1} which corresponds to the vibration of the hydroxyl groups in starch, glycerol, and distilled water. The thermal analysis, also presented in Fig.1, shows the samples with TPS have a lower thermal resistance (250°C) than the PBAT sample (350°C) because the starch and the plasticizers start degrading.

Table 1 Tension test results.

Sample	Maximum Stress (MPa)	Maximum Deformation (%)	Young's Modulus (GPa)
PBAT (reference)	1,86 ± 0,18	14,09 ± 2,52	0,018 ± 0,01
10% glycerol	1,25 ± 0,17	7,61 ± 1,35	0,029 ± 0,01
20% glycerol	2,49 ± 0,23	3,87 ± 0,76	0,055 ± 0,01
10% distilled water	2,22 ± 0,40	3,59 ± 0,94	0,042 ± 0,01
20% distilled water	1,75 ± 0,02	3,74 ± 0,58	0,036 ± 0,01

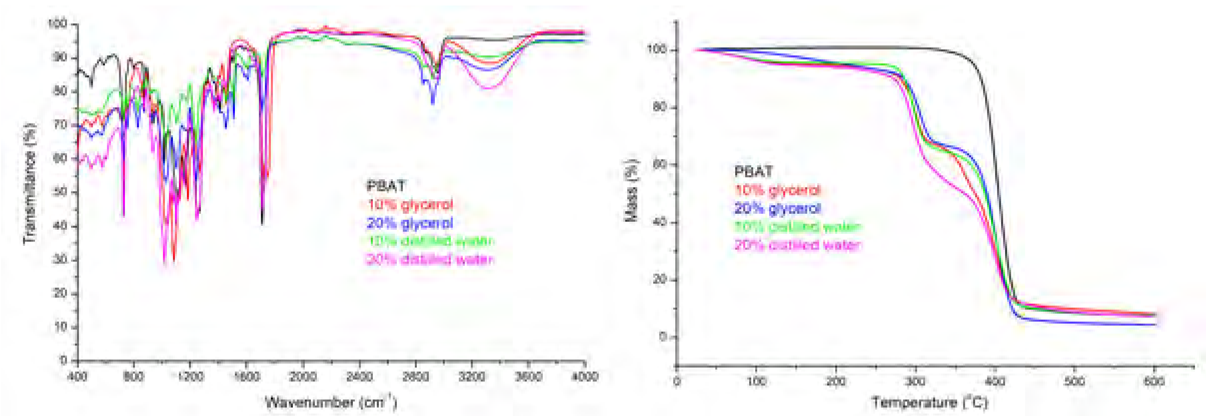

Fig. 1 FTIR test results (left) and TGA test results (right).

This study shows that potato starch is an interesting choice to develop starch-based biopolymers. Using starch combined with a biopolymer (PBAT), it's possible to produce a plastic with better mechanical properties than the biopolymer itself while reducing the cost of production since starch is a much cheaper polymer than PBAT, for instance.

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ID 297
STUDY OF THE ACOUSTIC PERFORMANCE OF COMPOSITES DEVELOPED WITH ORGANIC WASTE

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ABSTRACT
This work compares the acoustic performance of composites made with two different organic wastes (olive stone and corn cob). A total of twenty samples were produced using the two residues, varying the size of the residue particle, the density of the composite and its thickness. The acoustic performance tests, performed in an impedance tube, showed that the olive stone composite had better sound absorption in all frequencies than the corn cob composite, despite having a higher density and a less porous microstructure. The resin may have encapsulated the corn particles, which led to the blockage of the porous microstructure of the residue.

INTRODUCTION
Sustainability and environmental problems are increasingly relevant issues for society, which is reflected, at a global level, in the scope of scientific studies and product development. Within this theme arises the problem of agricultural waste generated in the production of olive oil, which creates enormous amounts of solid waste composed mainly of olive pits, or from the production of corn, which produces millions of tons of solid waste, with 20% of this total representing corn cobs (Bovo, 2022). These agricultural residues are typically used as fuel to generate electricity or heat. However, they can be used to produce activated carbon to absorb polluting agents in liquid and gaseous media and as a fertilizing agent or animal feed. However, these wastes can potentially be used in value-added products such as acoustic or thermal insulation panels in the construction sector (Farag, 2020). A previous study (Faustino, 2012) showed that corn cob has interesting acoustic behaviour for sound insulation purposes in buildings, showing incredible similarities with the sound insulation behaviour of cork since the corn cob has a porous and light microstructure. A study (Borrell, 2020) showed that the microstructure of the olive stone is rough and irregular, with small surface cavities. These cavities in the internal structure of the material guarantee the absorption of sound waves at certain frequencies.

The composites used in this work were produced in a hot compression moulding machine mixing the organic wastes (olive stone and corn cob) with a resin containing high bio-based content (SR GreenPoxy 56). A total of 20 samples were produced using the two residues, varying the size of the residue particle (1 and 2 mm for the olive stone and 6 and 20 mm for the corn cob), the density of the composite (between 0.45 and 0.55 $kg.m^{-3}$ for the corn cob and 0.7 and 1 $kg.m^{-3}$ for the olive stone) and its thickness (10, 20 and 30 mm). A ratio of 90:10 (corn cob or olive stone particles: resin), in terms of % of the weight, was used to produce the composites. The acoustic performance of the composites was performed in an impedance tube, according to the standard EN ISO 10534-2:2001, to evaluate the sound absorption of the materials.

RESULTS AND CONCLUSIONS

The acoustic results for the olive stone and corn cob composite with the best sound absorption are shown in Fig. 1. The olive stone composite shows better sound absorption in all frequencies than the corn cob composite, despite having a higher density and a less porous microstructure. These two factors should, in theory, give the olive stone composite the worst sound absorption capability than the corn cob composite. The resin may have encapsulated the corn waste, coating the porous microstructure of the particles, which led to a lower-than-expected sound absorption capacity.

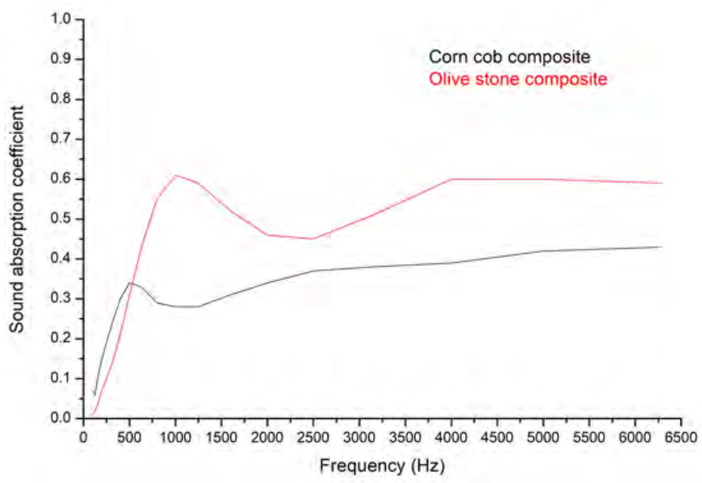

Fig.1 Acoustic performance test results.

This study demonstrated that the two types of waste used demonstrate potential for sound absorption applications. The lower-than-expected acoustic capabilities of maize residue should be studied in SEM tests.

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ID 299
UNVEILING THE POTENTIAL OF WOVEN FABRIC YARN ORIENTATION FOR ENHANCED PRESSURE SENSOR DESIGN: AN EXPERIMENTAL STUDY

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ABSTRACT
This research explored how fabric orientation affects woven pressure sensors, aiming to improve the design process for the development of textile piezoresistive sensors. The experimental setup involved 100% cotton-based fabrics functionalised through screen printing with a polyurethane-based paste with six different concentrations of graphene nanoplatelets (GNPs). The functionalised fabrics were then assembled to the textile electrodes, with variations in the yarn orientation (warpwise and weftwise), resulting in 12 unique samples. The electrical resistance of the active part of the sensor was measured and recorded. Additionally, the sensors were tested for piezoresistive behaviour to evaluate their sensitivity (S) and voltage coefficient of variation (CV%). The results indicate that the fabric orientation has a significant effect on the performance of the pressure sensors.

INTRODUCTION
In an intrinsically anisotropic material, such as a smart textile, the contact between the fibres in different directions can lead to peculiar deformation performance and, consequently, alter the path of current conduction. Although the force/pressure applied onto a textile substrate through a uniaxial compression is constant and independent of the yarn direction, it should be noted that such mechanical action causes a geometric change in the substrate, which can be identified by the reduction in its lateral thickness. In line with this, a recent study indicated that pressure sensors made of knitted fabric have different sensitivities in the walewise and coursewise directions (Xie et al., 2019). Therefore, we performed an experiment to evaluate this behaviour in woven fabric structures. The Fig. 1 exemplifies how the sensors were assembled in our study. The sensitivity (S) and CV% characterization methods were based on a previously published protocol (Arruda et al., 2022).

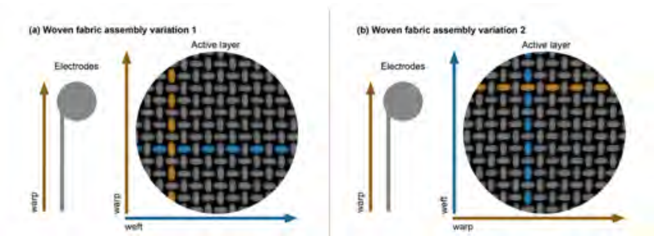

Fig.1 (a) the electrodes and the active layer are positioned in the same direction, both in the longitudinal position of the substrates; **(b)** the electrodes and the active layer are positioned in different directions, with the electrodes in the longitudinal position of the textile substrates and the active layer parallel to the substrates' transversal position. The copper colour represents the warpwise direction, and the blue colour the weftwise one.

RESULTS AND CONCLUSIONS

The results from the piezoresistive tests are comprised by the voltage CV% (Fig. 2) and by their (S) (Fig.3). A lower CV% was achieved for the weftwise direction (dashed line), and a higher (S) for all woven sensors whose active material was positioned in the warpwise direction.

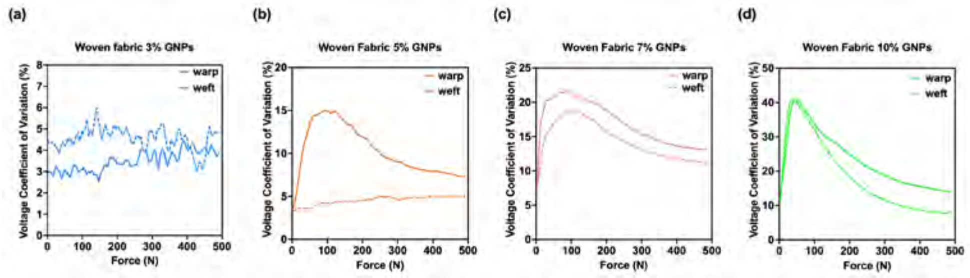

Fig.2 Voltage CV% for woven fabric doped with different % GNPs, whose active material was assembled towards the warpwise and towards the weftwise directions.

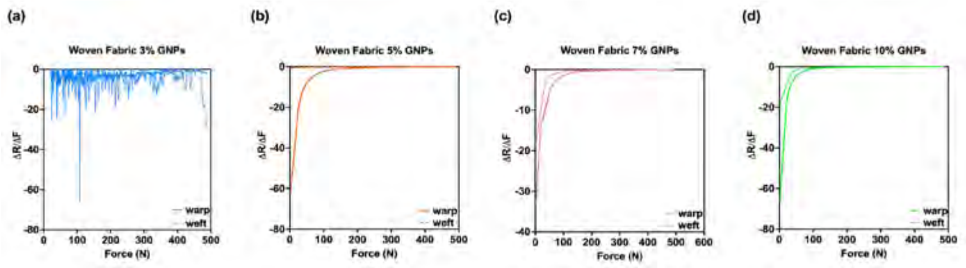

Fig.3 Sensitivity (S) of woven fabric doped with different % GNPs, whose active material was assembled towards the warpwise, and towards the weftwise directions.

The piezoresistive tests showed that the geometric deformation of the substrates and the direction of the yarns generated different electrical responses. In the weftwise direction, the friction phenomenon caused a lower CV%. Crimp ratios and superior crossing zone per unit area provided by its warpwise density contributed to a higher sensitivity in woven fabrics, in the warpwise direction.

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ID 303
ANTIBACTERIAL/ANTISEPTIC WOUND DRESSINGS BASED ON CASEIN/PVA ELECTROSPUN NANOFIBERS

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ABSTRACT
This work aimed to produce effective wound dressings with antibacterial/antiseptic properties based on casein/PVA nanofibers. The combination of these two polymers in a fibrous arrangement allows obtaining dressings able to protect the wound site and keep a suitable moisture level with a structure similar to the extracellular matrix (ECM). The dressings were functionalized with antibacterial/antiseptic agents, namely polyhexanide, the commercial antiseptic Octiset® (octenidine+phenoxyethanol), and zinc oxide, to prevent wound infection. The obtained systems were characterized regarding physical properties (e.g., surface morphology/topography), drug release behaviour, and biological properties (antibacterial activity, irritability, cytotoxicity, hemocompatibility). The material containing zinc oxide was the most interesting since it presented a structure closest to the ECM and had the highest antimicrobial activity.

INTRODUCTION
Dry dressings are ineffective for treating chronic wounds in diabetic patients as they do not adapt to the wound microenvironment and may cause further damage by adhering to the wound. Wet dressings are already used, but their efficacy is still limited, and new alternatives have been pursued. New strategies are being explored, such as the use of bioactive platforms that mimic or restore the function of the skin. Electrospun nanofiber membranes have raised particular interest since they have been shown to improve drug loading rates, absorb excess liquid, and replicate the normal function of the ECM, enhancing wound healing [1,2]. Casein is a natural polymer that is biocompatible, biodegradable, stable, non-toxic, and cost-effective, and can improve wound healing by reducing cytokines involved in the inflammatory process [1,2]. The incorporation of antibacterial/antiseptic agents can further improve the performance of the dressings, preventing/treating the infection.

RESULTS AND CONCLUSIONS
Samples containing Octiset® and Polyhexanide showed a controlled release for 24 hours. All the formulations showed antimicrobial activity with a reduction in microbial growth (30-75%), no irritation in the HET-

CAM test, no cytotoxicity (> 70% cell viability), and good hemocompatibility (< 2%), demonstrating their potential for wound care applications. The ZnO formulation was the most promising since it presented a nanofibrous structure similar to the ECM and had the highest antimicrobial activity against Staphylococcus aureus (Fig. 1).

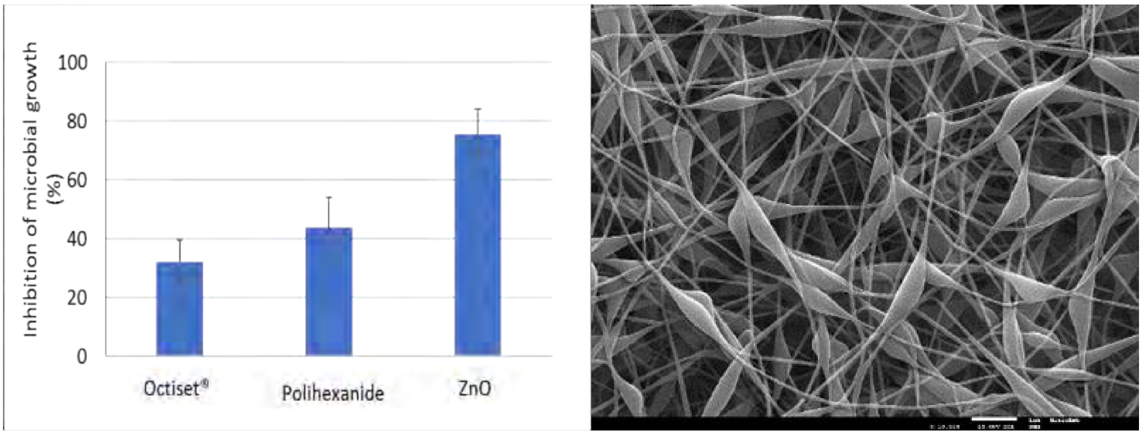

Fig.1 Antibacterial activity of drug functionalized casein/PVA nanofibers (left) and morphology of those containing ZnO (right)

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ID 305
ENVIRONMENTALLY FRIENDLY AND STRONG COMPOSITES MADE WITH NANOCELLULOSE-BASED LONG FILAMENT AND LIGNIN-BASED RESINS

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ABSTRACT
White pollution has caused extensive damage to the environment, thus making sustainable development necessary. Bioresources are attractive candidates for sustainable materials. Nanocellulose obtained from wood is a very promising environmentally friendly material owing to its high mechanical properties, and lightweight, renewable behaviors. This presentation, nanocellulose is utilized for preparing strong and tough filament and the filament is applied to environmentally friendly and strong structural composites. Not only the filament but also the resin should be environmentally friendly. So, we developed bio-based resins by deriving lignin, another side product of wood pulping: lignin-derived epoxies and lignin blended resins. Biobased high-strength resins are synthesized from low-cost and abundant sustainable resources of starch, citric acid and lignin through esterification. By integrating the resin into nanocellulose-based filament mats through hand-layup, hot-pressing and post-curing, an all-green composite was conceived with excellent flexural strength and stiffness. The high-strength and all-green composites can be used for automotive and aerospace structures.

INTRODUCTION
Advanced structural applications such as aerospace and automotive are heading towards sustainable technologies like natural fiber-reinforced green composites (NFRGCs) due to their green premiums. NFRGCs have two main constituents, natural fibers and biobased resins. Natural fibers such as hemp, jute, sisal, coir, bamboo, banana, kapok, henequen, cellulose, and others have been extensively studied for their reinforcement properties [Kopparthy, Netravali, 2021]. These are lightweight, non-abrasive, non-toxic, low-cost, renewable, and biodegradable.
Cellulose nanofibers (CNFs) possess unique characteristics such as biodegradability, biocompatibility, flexibility, lightweight, and a high aspect ratio, rendering them suitable for a wide range of applications. Although the isolation of CNFs is quite simple, its size is too small, limiting its applications for fibers and composites. Thus, extending it to a large-scale continuous filament, the so-called CNF-based long filament (CLF), production is challenging. Recently, wet-spinning techniques have become the most popular methods for spinning filaments due to their versatility and reliability. A custom-designed wet-spinning (pilot) system consisting of three bobbin winders, a coagulation bath, and a washing bath to produce CLF continuously [Kim et al., 2021]. The system shows great potential and has gained recognition since it offers continuous production of CNF by adopting a facile wet-spinning process. Furthermore, the influence of the

alternating current electric field on CNF alignment was investigated by adopting the same wet-spinning setup with a few modifications [Panicker et al., 2022].

Currently, most commercial composite materials comprise petroleum-based resins as matrices, which are reprotoxic and endocrine disruptor. Biobased epoxy resins have gained much attention due to renewable, abundant resources and green premium benefits [Kumar et al., 2021]. Biobased resins, including soy protein resins, wheat gluten protein resins, cardanol resins, linseed oil resins, lignin-derived resins, eugenol-based epoxy resins, and vanillin-based resins have been studied for coatings, flame retardancy, packaging, and structural applications.

This research aims to develop high-performance green composites mainly comprised of all biobased constituents. Figure 1 represents the fabrication process of the proposed green composite. The study embodies the combination of tough and strong CLFs and non-toxic, thermally stable biobased resins. CLFs are fabricated by wet spinning CNF suspension. Biobased resins are synthesized by mixing lignin with polyvinyl alcohol or starch, and citric acid as a green crosslinker. Biobased epoxies are synthesized from the lignin-derived vanillin, and a thermoset resin was prepared after mixing a hardener, for example, vanillin epoxy (VE), vanillin derived epoxy (VDE) and vanillin alcohol epoxy (VAE). Low-cost and green CLF-reinforced green composites are manufactured using vacuum-assisted resin transfer molding (VARTM) and a compression molding technique.

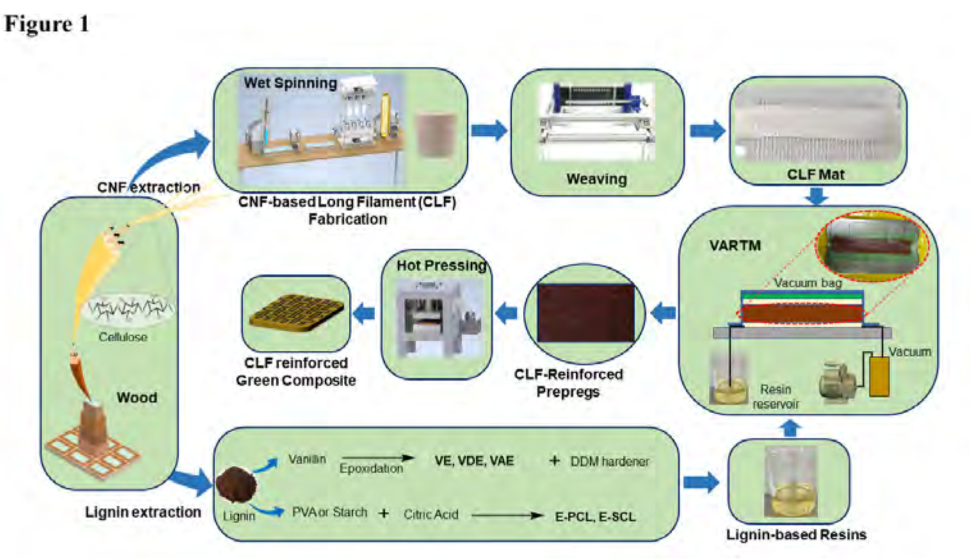

Fig.1 The fabrication process of high-performance CLF-reinforced green composite mainly comprised of all biobased constituents - CLFs fabricated using the wet spinning method and biobased resins.

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INFLUENCE OF WEAVE AND DYEING WITH POMEGRANATE PEEL EXTRACT OF COTTON FABRICS ON UV PROTECTION

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ABSTRACT

This paper presents the design of a cotton fabric that provides the highest ultraviolet (UV) protection by introducing a natural dyeing and different types of twill weaves. In addition to UV protection, the fabric has high antioxidant activity and good air permeability, which is crucial for the comfort of wearing fabrics, especially in summer. The effect of the weave can be seen in the air permeability, which is highest in the weft-faced (3/1) twill weave and lowest in the double-faced (2/2) twill weave, by up to 15%. The latter fabric when dyed, provides the highest UPF protection.

INTRODUCTION

Increasingly strict environmental regulations and legislations in the field of environmental protection are already promoting the use of natural ingredients in the global market. Approximately 1.62 million tons of pomegranate waste are generated annually by the juice industry worldwide (Magangana, 2020). The plant waste is an environmental and economic burden but can be used for bio-based solutions for the safe and sustainable design of functional and protective textiles (Verbič, 2021). When developing such textiles, it is important to plan their construction parameters, which can significantly affect the final properties (Kostajnšek, 2021). The aim of our research was to establish which of four different types of twill weaves provides highest UV protection and the best comfort properties, and how dyeing with natural dye from pomegranate peels (PG) affects the antioxidant activity and UV protection of the final fabric. Four woven white cotton warp (8 x 2 tex) and weft (24 tex) samples were produced at a setting of density 20 warp/cm and 26 weft/cm. The fabrics were made from four different types of twill weaves (weft-faced (1/3), double-faced (2/2), warp-faced (3/1) and weft-faced (1/3) broken twill). The cotton fabrics were dyed with an aqueous extract of PG (40 g/l) at 60 °C for 60 min and then washed. The samples were analyzed for their antioxidant activity (DPPH method), UV protection properties (UV/VIS), and front and back color (Datacolor). The physical and permeability properties of the fabrics were studied: warp and weft density, mass per unit area, thickness, air permeability, the size and distribution of pores were determined using the Jakšić airflow method.

RESULTS AND CONCLUSIONS

The results of the colour measurements of the front and back of the fabric, the calculated UV protection factor (UPF), and the antioxidant activity (AA) of undyed and dyed cotton fabrics are shown in Table 1. The UPF is strongly influenced by the dyeing and less by the weave. The differences in weave affect the UPF when the cotton fabric is dyed, but not when it is not dyed. The *Double-D* sample has the highest UPF, but the same (and extremely high) AA compared to the other samples. The sample is not the darkest, so the UPF is not related to the colour, but rather to the weave.

Table 1 CIELAB values of undyed and dyed (D) samples, UPF and AA (%)

	Front			Back				
Sample	**CIE L***	**CIE a***	**CIE b***	**CIE L***	**CIE a***	**CIE b***	**UPF**	**AA(%)**
Weft	94.66	3.04	-10.02	94.69	3.37	-11.31	3.11	22.55
Double	94.79	3.23	-10.70	94.66	3.25	-10.69	3.12	23.91
Warp	94.82	3.44	-11.34	94.60	3.12	-10.01	3.21	21.45
Broken	94.40	3.10	-9.78	94.46	3.14	-9.91	3.17	24.66
Weft-D	81.69	3.45	18.03	81.14	3.59	18.13	31.08	96.05
Double-D	80.84	3.58	19.17	81.06	3.56	18.03	35.96	96.12
Warp-D	79.84	3.78	19.58	80.14	3.79	18.82	32.19	96.11
Broken-D	81.14	3.58	18.64	80.61	3.62	19.17	31.05	96.07

Table 2 lists the physical properties and air permeability properties of the undyed and dyed samples. A high negative correlation was found between air permeability (-0.8), area fraction (-0.74) and UPF. The effect of weave is evident in the air permeability, which is highest for weft twill weave and lowest for double twill weave, by up to 15%.

Table 2 Physical properties of undyed and dyed cotton fabrics

Sample	**Thickness** (mm)	**Density** (yarns/cm)		**Mass** (g/m^2)	**Air permeability** (cm^3/cm^2s)	**Buble point** (µm)	**Pore diameter** (µm)	**Area fraction** (%)
		warp	weft					
Weft	0.499	22.50	31.25	111.68	84.58	105	26.29	26.06
Double	0.496	22.38	31.38	114.52	80.23	93	22.3	36.11
Warp	0.507	22.38	31.63	112.06	76.00	93	23.78	28.87
Broken	0.507	22.13	31.75	112.46	83.16	98	24.26	19.73
Weft-D	0.497	22.13	31.50	115.96	77.12	111	22.35	20.16
Double-D	0.509	22.13	31.50	114.98	67.98	92	16.91	19.52
Warp-D	0.525	22.38	31.50	117.02	72.93	98	27.38	13.26
Broken-D	0.504	22.00	31.50	116.64	75.10	139	27.94	15.75

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FUNCTIONAL FINISHING OF FIBRE-BASED SUBSTRATES USING ZINC OXIDE-NATURAL POLYMERIC COMPOSITE FOR ANTIMICROBIAL FILTER APPLICATIONS

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ABSTRACT
In this work, non-woven fibrous substrates were functionalized via dip-pad-dry with zinc oxide nanoparticles (ZnO NPs) using sizing ingredients as binders, comparing a natural with a synthetic compound, both being classically used for this end. Functionalized fabrics were characterised by their structural parameters, mechanical behaviour, permeability, moisture management, thermal properties and particle retention ability. The most promising functionalized fabrics were selected to act as an active filter barrier, and so their antimicrobial properties were evaluated. Upon the confirmation of their antimicrobial performance, different combinations of functionalised and non-functionalised substrates were studied considering their application in air filtration, mainly in face masks, based on their particle retention efficiency and breathability performance. Herein, biopolymer-based functional coatings were discussed as a sustainable strategy to incorporate zinc oxide nanoparticles into non-woven fabrics.

INTRODUCTION
All recent virus' outbreaks rely on airborne transmission, for which efficient filtering systems are needed to control the spread of these respiratory pathogens. For these reasons, the development of an innovative filter capable of repelling or degrading viruses and bacteria is one of the recent trends. Besides its use in air filters, this filter can be incorporated within a multilayered system for the production of an active respiratory protection mask. This solution would greatly improve general healthcare, not just to face future threats such as possible pandemic situations but also, for example, for hospital common use. Additionally, new materials are being sought to replace the commonly used filters for personal protection, in order to enhance comfort besides ensuring efficient filtering.

Regarding antimicrobial properties, distinct metallic oxides have been used as antimicrobial coatings. Among the inorganic oxides, zinc oxide (ZnO), is not only stable under harsh conditions but also regarded as safe materials for humans and animals (Ma, 2016). In fact, ZnO nanoparticles (NPs) can be used as part of multifunctional organic/inorganic nanocomposites with distinct applications, including textiles and

healthcare (Antunes, 2022; Ma, 2016).
A typical textile mill produces around 60,000 square meters of fabric per year (Jahan, 2022). In particular, the process of sizing, which comes before weaving or spinning, endows the produced fibres with additional strength (Bisschops, 2003). Sizing ingredients often used include polyvinyl alcohol and other natural polymers, such as starch and carboxymethyl cellulose (Jahan, 2022). On another hand, ZnO NPs have been incorporated in different biopolymeric matrices, as chitosan, starch, and functionalized cellulose, exhibiting strong antibacterial activity, dielectric properties, and UV absorbance (Ma, 2016). In the present study, a natural-based strategy for ZnO NP functionalization of non-woven fibrous substrates is proposed, using sizing ingredients, to provide an alternative impregnation strategy for active biological protection.

RESULTS AND CONCLUSIONS

The successful incorporation of ZnO NPs in the functionalised samples was confirmed both morphologically by SEM-EDS and chemically by FTIR-ATR and UV-Vis spectroscopy. Moreover, the antibacterial activity given by these NPs was proven against reference strains of Staphylococcus aureus (S. aureus ATCC 6538) and Escherichia coli (E. coli CECT 434), two representative of Gram-positive and Gram negative pathogenic bacteria, respectively, whereas the antiviral activity was shown for the bacteriophage MS2 as a surrogate of the SARS-CoV-2 virus.
This study demonstrates the successful development of fibre-based filters with natural-based antimicrobial functionalization by using minimum concentrations of ZnO NPs (<1.2% w/v), which can be promising as a sustainable strategy towards the production of environmentally friendly filtering systems, such as face masks.

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