NATURAL FIBERS AND BIOCOMPOSITES

A Sustainable Solution

EDITORS

Henry A. Colorado Lopera,
Sergio Neves Monteiro,
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Preface

The environmental impact of conventional engineered composite materials is a pressing concern since they comprise synthetic resins and fibers despite their exceptional mechanical properties. As the demand for high-performance materials continues to grow, it is imperative to explore sustainable alternatives that can meet the stringent requirements of various industries while minimizing their ecological footprint. Natural fibers and biocomposites represent a promising alternative for addressing the environmental challenges associated with conventional composites. These materials are derived from renewable biological sources, such as plants, animals, and microorganisms, providing many advantages such as reduced carbon footprint, lower energy consumption, and improved biodegradability. Natural fibers exhibit remarkable mechanical properties, including high strength-to-weight ratio, stiffness, and toughness. When combined with suitable bio-based matrices, these fibers can form biocomposites that exhibit comparable or even superior performance to conventional composites in certain applications. The transition from conventional composites to natural fiber biocomposites is challenging and influenced by growth conditions, harvesting methods, and processing techniques, as well as interfacing mechanisms to achieve the desired mechanical and structural performance metrics. Hence, the motivation and objective of this effort.

These proceedings offer a comprehensive overview of recent advancements in the processing and characterizing of natural fibers and biocomposites, highlighting their potential as sustainable solutions to mitigate environmental challenges. Natural fibers, derived from renewable sources, possess exceptional properties, biodegradability, and low cost, making them a compelling alternative to synthetic fibers in composite reinforcement and other applications. The book underscores the growing research and industrial interest in natural fibers to reduce the carbon footprint associated with traditional synthetic materials, given their ability to absorb carbon dioxide

during growth. The imperative to develop sustainable, biodegradable, or biocompatible solutions has spurred applications in diverse fields, including sports, transportation, armor, medicine, infrastructure, construction and building materials, and architecture.

Henry A. Colorado Lopera
Sergio Neves Monteiro
George Youssef

Contents

About the Editors

Henry A. Colorado Lopera is a Full Professor in the Mechanical and Materials Engineering Departments of the Universidad de Antioquia at Medellin, Colombia. He is also affiliated with the Materials Department from the State University of Northern Rio de Janeiro, Brazil and has been a Visitor Professor in the Military Institute of Engineering and a Fulbright Visitor Scholar in the Mechanical and Aerospace Engineering Department of the University of California San Diego from 2023 to 2024. His main areas of research are composite materials, natural fibers, green materials, and additive manufacturing. Dr. Lopera obtained a BS and M.Sc. in Mechanical Engineering from Universidad Nacional de Colombia in 2005; and M.Sc. and Ph.D. in Materials Science and Engineering from the University of California Los Angeles in 2010 and 2013, respectively. Henry has active collaborations in several institutions in Brazil, Chile, China, and USA. He is also working as Associate Editor for the journals *Heliyon*, *Engineered Science*, and *ES Materials & Manufacturing*; and Editorial Board for *Advanced Composites and Hybrid Materials*, and for the *American Ceramics Society Bulletin* (2023–2026). He is also a Global Ambassador for the American Ceramics Society.

Sergio Neves Monteiro graduated as a metallurgical engineer (1966) at the Federal University of Rio de Janeiro (UFRJ) Brazil. He received his M.Sc. (1967) and Ph.D. (1972) from the University of Florida, USA, followed by a 1975 course in energy at the Brazilian War College, and a post doctorate (1976) at the University of Stuttgart, Germany. In 1968, he joined the Metallurgy Department of UFRJ as full professor of the post-graduation program in engineering (COPPE). He was elected as head of department (1978), coordinator of COPPE (1982), Under-Rector for Research (1983), and was invited as Under-Secretary of Science for the State of Rio de Janeiro (1985), and Under-Secretary of the College Education for the Federal Government (1989). He retired in 1993 from the UFRJ and joined the State University of North Rio de Janeiro (UENF), where he retired in 2012. He is now a voluntary professor at the Military Institute of Engineering (IME), Rio de Janeiro. Dr. Monteiro has published more than 2,300 articles in journals and conference proceedings with more than 13,000 citations in Scopus (H index 57) and 20,000 in Google Scholar (H index 72). He has been honored with several awards including the ASM Fellowship, the Brazilian Army Medal, the Humbertus Colpaert Medal, and several TMS awards, like the Leadership Award (2020). He is a top researcher (1A) of the Brazilian Council for Scientific and Technological Development (CNPq) and Emeritus Scientist of State of Rio de Janeiro (FAPERJ). He was president of the Superior Council of the State of Rio de Janeiro Research Foundation, FAPERJ (2012), and coordinator of the Engineering Area of this foundation (2016). Dr. Monteiro has also served as president of the Brazilian Association for Metallurgy, Materials and Mining (ABM, 2017–2019), as a consultant for the main Brazilian R&D agencies, and as a member of the editorial board of five international journals as well as associate editor-in-chief of the *Journal of Materials Research and Technology*. He is the author of 130 patents and a top world researcher in "Natural Fiber Composites" and "Ballistic Armor", Scopus 2020. He has contributed to the Graduate Program of Materials Science and Engineering to reach the top position in the Brazilian Federal Agency for Support and Evaluation of Graduate Education (CAPES) institutional rank.

Felipe Perisse Duarte Lopes has a degree in Materials and Metallurgical Engineering from the Darcy Ribeiro State University of Northern Fluminense (2009), a master's degree in Materials Engineering and Science from the Darcy Ribeiro State University of Northern Fluminense (2011), and a Ph.D. in Materials Science from the Military Institute of Engineering (2013) completed in a sandwich program in partnership with the University of California, San Diego. He also completed a post-doctorate at the Advanced Materials Laboratory at UENF (2018–2023). Dr. Lopes is currently an Associate Professor at LAMAV/UENF. He has experience in materials and metallurgical engineering, with an emphasis on mechanical properties of polymeric composites, metals, and alloys, working mainly on the following topics: polymeric composites, natural fibers, mechanical properties, thermal properties, failure analysis, corrosion and inspection of equipment in offshore units. Dr. Lopes worked for over 10 years as an inspection and maintenance engineer in the oil and gas industry around the world, in countries such as USA, Norway, England, Spain, Singapore, and Mexico.

Carlos Castano Londono is a tenured Associate Professor of the Department of Mechanical and Nuclear Engineering at Virginia Commonwealth University (VCU). Dr. Castano received a B.S. in Engineering Physics and an M.S. in Engineering from the National University of Colombia. He worked in R&D for a few years in the coating industry before obtaining his Ph.D. in Materials Science and Engineering at Missouri S&T. He joined VCU in 2014, holding appointments as Postdoctoral Fellow, Research Associate at the Nanomaterials Core Characterization Center, and Assistant Professor before tenured. Dr. Castano has received funding from different sources including NSF, DOE, and NRC. He has also received numerous recognitions, including the NSF Career Award, honorary ALPHA SIGMA MU member, Roberto Rocca Fellow, VCU NIRA, and was inducted into the National Academy of Inventors Chapter at VCU. Dr. Castano's research group focuses on tailoring the surface properties of materials for applications in various fields including catalysis, sensors, solar fuels, sustainable materials, functional materials, and materials under extreme environments.

Marc A. Meyers is a graduate (Mechanical Engineering) of the Federal University of Minas Gerais and received his doctorate at the University of Denver. He held university positions at the Military Institute of Engineering (Rio de Janeiro), South Dakota School of Mines and Technology, and New Mexico Institute of Mining and Technology, prior to joining the University of California, San Diego, where he is a Distinguished Professor.

Marc Meyers' leadership and visionary effort at unifying dispersed activities through the fundamental physics and chemistry principles relevant to high strain rate deformation have given rise to the field of dynamic behavior of materials. The principles developed have been relevant for designing high rate forming and fabrication processes as well as material failure during impact or explosive loading applications

Although his area of specialization is the dynamic behavior of materials, he has contributed to nanocrystalline materials, synthesis and processing, and mechanical properties in general, with an emphasis on fundamental mechanisms. In recent years he has turned his curiosity and effort to biological, biomedical, and bioinspired materials and how they derive their mechanical properties in terms of their hierarchical structure/architecture. His work in this area, whether associated with abalone shells, toucan beaks, fish scales or teeth, among many other biological materials, has been an inspiration and has generated world-wide interest in terms of the complex structure/property relationships that they portray and the complicated nano/micro-mechanisms that underlie their mechanical performance.

He been recognized by accolades in the USA, Europe, China, and Brazil. In the USA, he received awards from the three societies in which he is most active: TMS, APS, and ASM International; he is a fellow of all three. In the field of dynamic behavior of materials, he is the recipient of the top APS Award, the George Duvall Shock Compression Award, and of the European Dymat Society, the Rinehart Award. TMS has bestowed on him its most prestigious awards: The Mehl Award, Morris Cohen Award, Julia & Johannes Weertman Educator Award, and Leadership Award. He was recognized by the highest German Materials Society (DGM) award, the Heyn Medal. In Germany, he is also the

recipient of the Humboldt Senior Scientist Award. The Luxembourg Academy (Institut Grand Ducal) gave him the Grand Prix en Sciences and elevated him to honorary member. He is a member of the Brazilian Academy of Sciences. He is also the recipient of the Acta Materialia Materials and Society (Hollomon) Award and Gold medal (2025).

George Youssef is a Professor of Mechanical Engineering at San Diego State University. He earned his Ph.D. from the University of California, Los Angeles, in 2010. He focuses his research on the experimental solid mechanics of non-traditional materials. His work delves into the fundamental behavior of polymers, composites, and intelligent materials, exploring their response to diverse loading, operating, and environmental conditions. Dr. Youssef's research contributes to critical areas such as shock-tolerant materials, interfacial strength in heterogeneous structures, polymer damage and failure, biomechanics, and strain-mediated composite multiferroics. The National Science Foundation, the Department of Defense, and private industry have supported his work. Over the past decade, Dr. Youssef has been recognized for his achievements by organizations, including the San Fernando Engineers Council, the Society of Automotive Engineers, the San Diego Engineers Council, and San Diego State University.

Daniel Salazar is an electronic engineer from the Universidad del Quindío (2007) who received his Ph.D. in Physics and Mathematics from the Universidad de Castilla-La Mancha in 2012 under the supervision of Prof. Marco Antonio Lopez de la Torre. He started his research on nanostructured and granular magnetic materials, transition metal oxides, magnetic properties and electrical transport in nanocrystalline oxides, and magnetic, structural and thermal properties in strongly correlated electron systems during his Ph.D. In late 2013, he joined BCMaterials in a postdoctoral position and currently is a Senior Researcher with work focused on the study of the magnetocaloric effect and their physical features on single crystals of magnetic shape memory alloys and on the coercivity enhancement of nanostructured rare-earth (RE)-free hard magnets by grain boundary engineering. Recently, he started

to study the formulations for the design and development of multifunctional magnetic inks and pastes and powders as feedstock material for 3D-printing processes. Dr. Salazar has co-supervised three doctoral thesis (7 more in progress), 6 master thesis and several TFGs, and has published more than 60 SCI papers and 3 book chapters (H-index Scholar: 18, i10: 30, citations: 910). He has contributed to over 60 international conferences, given 14 invited talks in international conferences, as well as multiple seminars in international institutions (Oak Ridge National Laboratory (USA), Lawrence Livermore National Laboratory (USA), AMES Laboratory (USA), TU Darmstadt (Germany), University of Pittsburgh (USA), University of Cagliari (Italy), Texas A&M University (USA), etc.). He has been a visiting scientist in NCSR-Demokritos (Greece), University of Delaware (USA), and University of Valle (Colombia). He has participated in 14 publicly funded projects and one privately funded project, being co-PI of a European Project (H2020). He has organized 14 international symposiums/workshops and is a board member of five scientific advisory committees. He is the former Chair of The Minerals, Metals & Materials Society (TMS) Magnetic Materials Committee and Board Member of the Scientific Program. He is also member of the Spanish Club of Magnetism, European Magnetism Association, SOCIEMAT, and E-MRS.

Part I
Natural Fibers: Fundamentals and Applications

Recovery of Textile Waste and Its Viability and Resistance for Application in Orthopedic Prostheses

A. F. Campanhão, B. N. S. Cobuci, D. C. R. Velasco, N. T. Simonassi, C. M. F. Vieira, and F. P. D. Lopes

Abstract The textile industry is one of the most polluting sectors in the world, contributing to a complex and abundant post-consumer waste stream. With technological advancement in modern orthopedic medicine, several composite materials have been used in this area. Given the lack of scientific studies, the objective of this research is to recover different types of fabrics and test their viability and resistance for use in orthopedic prostheses. For this purpose, a tensile test was carried out on textile scraps, based on the ABNT NBR ISO 13934-1 standard. In this work, the resistance of different fabrics can be observed, as well as their viability and use in composites.

Keywords Sustainability · Feasibility · Textile waste · Composites

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Introduction

Textile products play an essential role in everyday life, with applications that go beyond clothing to areas such as food, housing, and health. Many are made from vegetable raw materials such as cotton and linen, guaranteeing quality and sustainability [1]. These modern textiles are used in innovative sectors such as medicine, construction, the car industry, and even the space industry, where they offer resistance and lightness. The evolution of textile products highlights their importance and adaptability in various facets of contemporary life [2].

The textile industry is the third most polluting in the world, and its environmental implications have increased in the last decade, driven by consumer disposal habits and the depletion of raw materials by producers. This industry has a significant impact on the three pillars of sustainability: the environment (emissions, energy, and water consumption), the social aspect (poverty, work ethic), and the economy (purchases, manufacturing costs, and resource depletion) [3, 4].

The Brazilian Textile and Apparel Industry Association (ABIT) (2024) highlights the challenges facing the textile sector in Brazil, with an emphasis on sustainability. The sector is one of the pillars of the Brazilian economy, employing more than 1.5 million people and accounting for around 5% of industrial GDP [5].

In this context, in response to the environmental pollution caused by textile waste, several researchers have begun studies into the possibility of using these materials in composites. On the other hand, companies have discussed strategies and some have already been adopted with the aim of recovering residual fibers, avoiding their disposal in landfills [6]. There are currently several options for recycling fibers into composites, with several recent studies reporting on the use of recycled textile waste fibers in different matrices, including thermoplastic polymers, thermosetting resins, natural constituents, and concrete, considering specific applications [7].

Composites are currently outperforming basic materials, as they combine the best characteristics of each of them individually. They have already demonstrated their effectiveness in various applications, especially in the production of prostheses for amputees and people with disabilities [19].

This work stands out for exploring a field that has yet to be investigated in the literature: the use of textile waste as reinforcement in polymeric matrices for the manufacture of prostheses. While there are several studies that demonstrate the potential of textile waste in polymer composites, as indicated in [2, 8], the specific application of these recycled materials in the context of medical devices, such as prostheses, remains little explored. There are examples in the literature of the use of polymeric matrix with other types of reinforcement, such as the research by the authors [9], who used epoxy composite reinforced with jute fibers for sockets. This study seeks to fill this gap by recovering and testing different types of fabric that have not yet been explored, assessing their viability and resistance for this purpose. The novelty therefore lies in combining the reuse of textile waste with an approach aimed at developing prostheses, broadening the scope of applications for these sustainable materials.

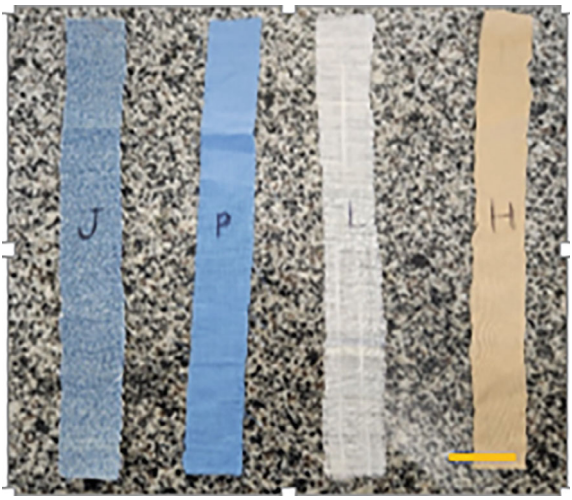

Fig. 1 Jean, poplin, linen, and helanquin fabrics before the tensile test. Scale bar = 1 cm

This article makes an original contribution by investigating the feasibility of using textile waste, such as linen, jeans, poplin, and helanquin, as reinforcements in polymeric matrices aimed at prosthetic applications.

Materials and Methods

Materials

Four types of raw textile waste were used in this work: linen, jeans, poplin, and helanquin, labelled J, P, L, and H respectively. The fabrics were collected from a clothing factory located in the city of Campos dos Goytacazes, state of Rio de Janeiro. The conditions of the fabrics were arranged as shown in Fig. 1.

Methodology—Tensile Test

The tensile test was used to obtain the modulus of elasticity. A total of twenty specimens were used to carry out this test, five of each type of fabric mentioned above. As it was not feasible to use ABNT NBR ISO 13934-1 [10], we opted to use the standard (ASTM) D 3039 [11] which is applied to composite [2]. The test specimens were made in accordance with standard (ASTM) D 3039 [11], with dimensions of approximately 250 × 25 × 3 mm thick, as shown in Fig. 1. The test was carried out using the INSTRON 5582 machine, located in the Advanced Materials Laboratory (LAMAV) at UENF at room temperature with a speed of 20 mm/min. The speed was

adjusted as the fabric deformed. The test began at a speed of 5 mm/min, increasing by 10 mm/min until it reached a speed of 20 mm/min, which best suited these materials. Figure 2 shows the test specimen in the INSTRON, and Fig. 3 shows the test specimens already tested.

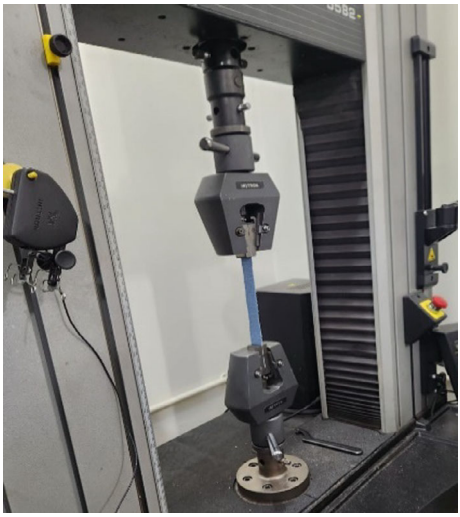

Fig. 2 INSTRON testing machine with jeans fabric

Fig. 3 Jean, poplin, linen, and helanquin fabrics after the tensile test. Scale bar = 1 cm

Results and Discussion

Below are the graphs of deformation and maximum tensile strength absorbed by each type of fabric (Figs. 4 and 5).

In Fig. 4 the jeans fabric, represented by the black line, shows low elasticity and limited tensile strength, as evidenced by its smooth stress–strain curve. This makes it unsuitable for applications requiring flexibility or high mechanical support. In contrast, poplin (red line) stands out for having the highest tensile strength, in excess of 80 MPa, and a significant deformation (±0.16), characteristics that make it ideal for supporting large loads and for use in applications requiring high durability and mechanical strength [12].

Linen fabric (green line) offers moderate tensile strength (±35 MPa) and good deformation capacity (±0.12), making it suitable for areas requiring flexibility and impact absorption. On the other hand, helanquin (blue line), with the lowest strength and deformation values, is less suitable for structural parts subject to high stresses, being more appropriate for intermediate layers or coatings [13, 18].

Figure 5 shows that the jeans and linen fabrics have low maximum tension values, which results in low resistance. As the graph shows, these fabrics have a high degree of variation compared to poplin and helanquin. On the other hand, poplin has this

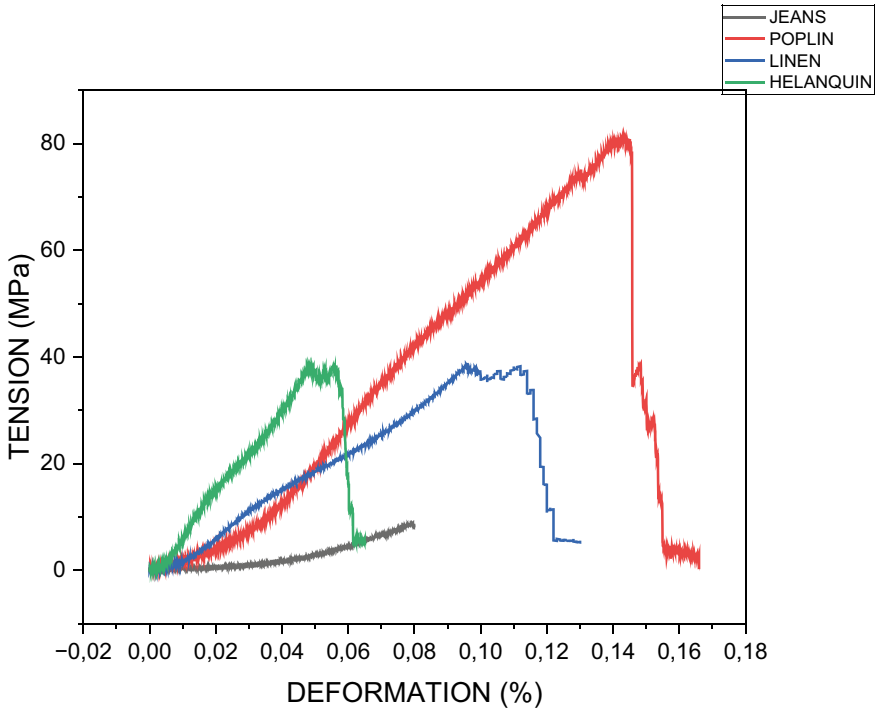

Fig. 4 Tension and strain graph for each type of fabric

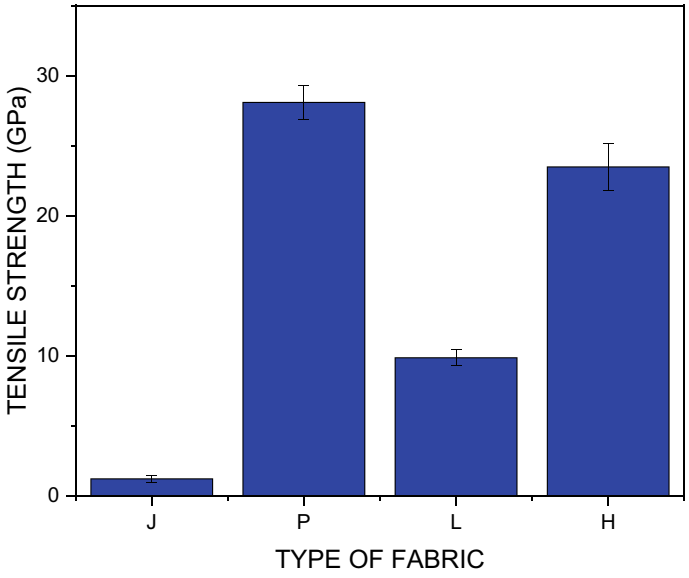

Fig. 5 Maximum tension graph for each type of fabric

characteristic because it was the fabric that withstood the greatest tension without breaking, which shows that it has greater resistance and durability, which corroborates Refs. [14, 15].

The jeans and linen fabrics have low maximum tension values, which results in low resistance compared to poplin and helanquin, which withstand greater tension without breaking. Poplin is noted for its durability and strength, making it a superior choice for applications requiring robustness. In contrast, while jeans is tough, its lower deformation capacity can be a disadvantage for applications requiring flexibility. On the other hand, linen, with its intermediate characteristics, offers a balance between comfort and support [16, 17].

The deformation results of poplin presented in this study are similar to those of polyester shown in Table 1, suggesting that both materials have comparable deformation behaviors. Although poplin is composed of different fibers and is most commonly found in blends of cotton and polyester, or 100% cotton, its flexibility is close to that of polyester, which is known for its moderate elasticity and resistance to deformation. This can be attributed to the mechanical properties of the fibers and the way they react to applied forces [2].

Table 1 Tensile modulus, maximum stress, and deformation of fabrics adapted from Azminor et al. [2]

Type of fabric	Tensile modulus (GPa)	Maximum tension (MPa)	Deformation (%)
Lycra	0,46	±30	7
Polyester	0,26	±25	12
Cotton	0,62	±23	5

Conclusion

Based on the analysis of the fabrics evaluated, it is possible to conclude that flax stands out as the most resistant material, showing the greatest capacity to withstand tensions without failing, which makes it ideal for applications requiring high durability and mechanical resistance. This characteristic makes the fabric a potential candidate for use as reinforcement in polymeric matrices intended for prosthetic applications. However, more studies are needed to fully validate this application. On the other hand, jeans, poplin, and helanquin fabrics, despite having lower tensile strength, offer specific advantages for different applications.

Poplin, with its ability to withstand large loads and its significant deformation, is the most robust option and is suitable for applications that require a balance between strength and flexibility. Jeans, on the other hand, although resistant, has a lower deformation capacity, limiting its use in situations that require greater flexibility. Finally, helanquin, due to its low resistance, is more suitable for applications that do not require high levels of tension, and is suitable for coatings and intermediate layers. Therefore, the appropriate choice of material will depend on the specifics of the application, balancing resistance, elasticity, and durability to maximize performance and meet the needs of the intended use.

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Comparison of Advanced Sample Preparation Techniques for High-Resolution Imaging of Sponge Spicule Cross-Sections

Fariborz Tavangarian, Niloofar Fani, Armaghan Hashemi Monfared, and Sorour Sadeghzade

Abstract This study comprehensively investigates the effectiveness of three advanced sample preparation techniques—ion milling, laser cutting, and Focused Ion Beam (FIB) milling—for high-resolution imaging of Euplectella aspergillum sponge fibers called spicules. Primarily composed of silica layers with nanometer-scale organic interlayers, spicules possess complex hierarchical structures which are critical for their mechanical properties. Accurate characterization of these structures requires advanced sample preparation to prevent artefacts and preserve structural integrity. Ion milling introduced significant surface degradation and uneven material removal in spicules. Despite its precision, laser cutting caused thermal damage and induced micro-cracks, compromising the microstructural integrity. In contrast, FIB milling provided superior results, producing smooth, artefact-free cross-sections with minimal thermal and mechanical stress. The real-time imaging capability of FIB milling further ensured optimal sample preparation, making it the most suitable technique for delicate biological materials like spicules. The findings of this study provide valuable insights into the preparation of biological samples for further research and analysis.

Keywords Spicule characterization · Nanoscale imaging · Laser-based techniques

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Introduction

The spicules of the Venus Flower Basket (Euplectella aspergillum), a deep-sea glass sponge, are known for their remarkable structural design and strength. Made mainly of silica, these spicules have a layered structure, with a central organic core surrounded by concentric rings of silica that vary in thickness [1]. This unique design gives the sponge both strength and flexibility, allowing it to withstand the high pressures of the deep ocean without breaking [2]. The spicules are arranged in a cylindrical lattice, which makes the sponge's body stable and strong, helping it stay anchored to the ocean floor. Understanding the detailed architecture of spicules is essential not only for unravelling the biology and ecology of sponges but also for exploring the potential applications of this natural architecture in the development of advanced structures. The nanoscale features and microstructure of spicules are key factors in their exceptional mechanical properties. This makes them a valuable subject of research for developing new materials that mimic these natural designs [3–5].

The characterization of spicules involves high-resolution imaging techniques capable of revealing their internal structures in great detail. Techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atomic force microscopy (AFM) are commonly used to examine the morphology, composition, and mechanical properties of spicules [6, 7]. However, the success of these imaging techniques largely depends on the quality of the sample preparation process. Proper preparation is essential to reveal the internal structure of spicules without causing any damage, which could affect the accuracy of the analysis. Given the delicate nature of spicules and their susceptibility to thermal and mechanical stresses, sample preparation methods must be carefully chosen and executed to preserve the fine structural details [8].

Laser cutting technique works by using a focused laser beam to cut through materials by heating them along a specific path. This technique is effective because it allows samples to be cut quickly and accurately with minimal physical contact, reducing the risk of mechanical damage [9]. Huang et al. demonstrated the effectiveness of laser cutting in fabrication hallow fibers, showing that it is particularly useful for processing different materials that are difficult to handle with traditional methods [10].

Ion milling is another important technique to cut the samples in materials science, especially for preparing samples for transmission electron microscopy (TEM) [11, 12]. Ion milling relies on sputtering, in which energized ions physically eject other atoms and molecules from the sample surface through momentum transfer. Ion milling is commonly used in materials science and engineering for applications such as cross-sectioning samples, thinning samples for electron transparency, and removing layers for deeper analysis. It creates smooth, polished surfaces, which are perfect for high-resolution imaging [13, 14].

Focused Ion Beam (FIB) milling is another highly precise technique used to prepare samples at the nanometer scale. FIB uses a finely focused beam of ions, usually, to carefully remove material, allowing for extremely fine and accurate cuts.

This method is especially valuable for handling delicate samples, such as biological tissues and microelectronic components. FIB milling also offers the advantage of in situ imaging during the milling process, which helps ensure the quality and precision of the final cross-sections [15].

There are two main methods for sample preparation using ions: focused ion beam (FIB) and ion milling (also known as near-parallel ion technique). The primary difference between these methods is in the ion beam characteristics. FIB uses a highly focused beam of high-energy ions, typically gallium, at energies up to 30 keV, allowing for precise milling in very small areas (around 50 × 50 μm). However, this process can be time-consuming due to its precision. On the other hand, ion milling uses lower energy ions, usually less than 20 keV, and creates a broad, unfocused ion beam from an inert gas like argon. This beam is directed at the sample surface, where the ion's kinetic energy is converted to heat and momentum, causing atoms to be detached and gradually removing material in thin layers. By moving the ion beam across the sample, the surface can be milled to the desired depth with control over the milling rate, resolution, and surface quality. Over time, advancements in ion sources, beam optics, and sample handling have significantly improved the precision and efficiency of these techniques [16].

In this study, we systematically evaluate how well ion milling, laser cutting, and FIB milling work for preparing cross-sections of spicules for high-resolution imaging. By comparing the results of these methods, we aim to find the best technique for preserving the structure of spicules during sample preparation. This research not only advances our understanding of sponge biology by improving how we study spicules but also has wider applications for preparing other delicate biological specimens in materials science and biomimetics.

Materials and Methods

Three groups of spicule bundles were carefully extracted from the sponge skeleton using a Dremel. The first set of bundles was securely affixed to the laser cutting platform using a tape to prevent any movement during the cutting process. The Universal M-360 Laser Cutter was used for this procedure. The laser power was adjusted to 5% of the machine's maximum capacity, while the cutting speed was maintained at 2% of the machine's maximum speed. The process was controlled using EngraveLab v10 software. The next collection of spicule bundle extracted was attached to the stage of the Ion Beam Milling System (Leica EM TIC 3X) using carbon tape. The ion energy was set to 6 keV, and a beam current of 2.2 mA was chosen based on the manufacturer's guidelines. To provide mechanical support during the FIB milling process, the third group of bundles of spicules were affixed to a stub using hot glue. The sample was placed in a Scanning Electron Microscope (SEM, Thermo Scientific™ Scios), and the FIB system was operated at a low beam current 65 nA and voltage (30 kV) to achieve precise material removal while minimizing surface damage.

Results and Discussion

In laser cutting, the laser's energy caused rapid, localized heating, which could lead to melting, vaporizing, or even destroying parts of the spicules, as shown in Fig. 1. This heat could damage the spicules, causing them to crack, warp, or melt. Moreover, the quick heating and cooling during laser cutting could create mechanical stress in the spicules, leading to tiny cracks. These cracks made it difficult to get a clear, detailed view of the spicules, which was important for studying their structure.

Figure 2 shows the SEM images of the cross-sections of spicule bundles after cutting by the ion milling method. The highlighted areas show noticeable surface damage, including rough textures. The spicules were deformed and partially melted due to the localized rise in temperature during the process, especially in the circular cross-sections where ion milling caused significant distortion. These issues made it difficult to see the fine details of the spicules, affecting the quality of the analysis. The main problem with ion milling for spicules was that the high-energy ions can damage the surface. This damage could result in rough surfaces and defects that change the spicule's natural structure. Although this method was not suitable for cutting spicule cross-sections, other studies have demonstrated its effectiveness with different materials.

Wei et al. [17] used ion milling to create a smooth surface for easier observation and to minimize the impact of artificial fractures. The three-beam ion polishing

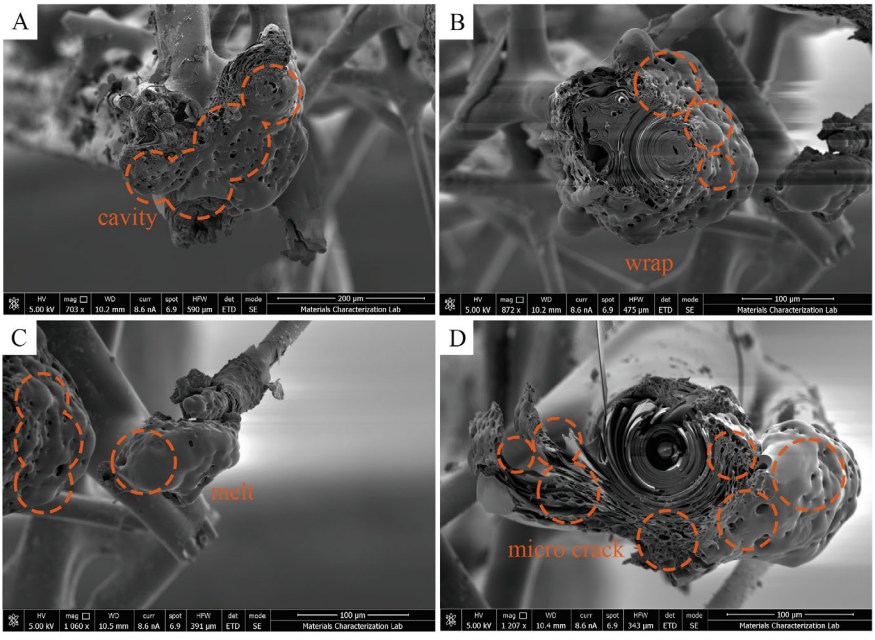

Fig. 1 SEM images **a–d** of spicule bundles after cutting the samples by laser cutting technique

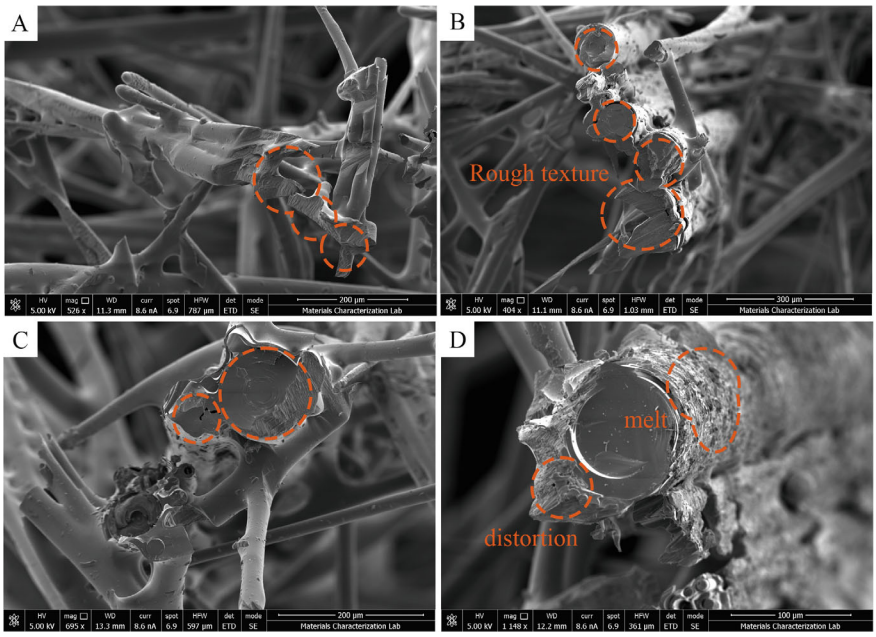

Fig. 2 SEM images **a–d** of spicule bundles damaged after being exposed to ion milling

method removed approximately 8 µm of surface material, resulting in a flat, rectangular organic-rich shale surface ideal for scanning electron microscope (SEM) observation. Coutinho et al. [18] utilized ion milling for the preparation of tooth-biomaterial interfaces for transmission electron microscopy (TEM) analysis. While the TEM successfully revealed the structural details of the interface, the high-energy ion milling process introduced molten areas and fogging on the cross-section of the sample. Kleiner et al. [14] demonstrate that argon broad ion beam (BIB) sectioning is an effective method for preparing high-quality surfaces of hydrated alite for scanning electron microscopy (SEM) imaging. It produced flat, smooth surfaces with minimal artifacts, allowing for high-resolution imaging of nano-sized pores. However, in our study, the ion milling technique resulted in significant damage to the samples and proved to be an unsuitable method for sectioning the spicules.

In addition to the above-mentioned techniques, FIB milling was used to prepare spicule cross-sections. Figure 3 shows the SEM images of the cross-section of spicules milled using FIB. The surfaces created by FIB milling were smooth and didn't have the damage seen with other techniques. This showed that FIB method is a better solution to cut the delicate biological samples such as spicules. One of the main advantages of the FIB milling technique is the lower temperature generated during the milling process compared to the other techniques. The gentle sputtering action of the ion beam also reduced mechanical stress, preventing the formation of tiny cracks or other structural damage. This makes FIB milling an excellent choice for preparing spicule cross-sections as it preserves the fine details of the structure

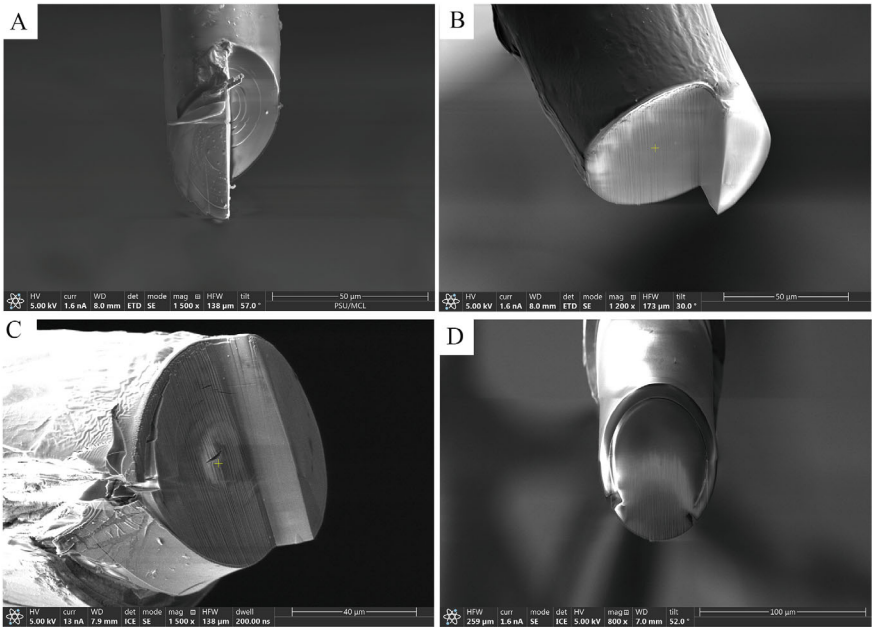

Fig. 3 SEM images **a–d** of spicule cross-sections cut with FIB milling. The surfaces are smooth and free of damage or heat effects, showing FIB's precision in maintaining spicule integrity

and avoids introducing any unwanted changes. Additionally, FIB systems allow for real-time imaging during the milling process. This means researchers can adjust the process immediately, ensuring that the cross-sections meet the required standards and are free from defects.

Our study using focused ion beam (FIB) techniques aligns with the findings of Giannuzzi et al. [19]. They successfully employed FIB to prepare multiple scanning electron microscopy (SEM) images from sequential cross-sections. This approach allowed for both two-dimensional and three-dimensional analyses of bone/dental implant interfaces. In contrast, our results differ from those of Meerbeek et al. [20]. Their application of FIB to the resin-dentin interface suggested the formation of artifacts, likely due to heat and recrystallization effects, which obscured the actual ultrastructure.

Table 1 compares the key characteristics of three cutting techniques: Ion Milling, Laser Cutting, and Focused Ion Beam (FIB) milling.

Table 1 Comparison of key features for Ion Milling, Laser Cutting, and Focused Ion Beam (FIB) techniques for preparing Euplectella aspergillum Spicule cross-section

Feature	Ion milling	Laser cutting	Focused Ion Beam (FIB)
Precision	Moderate precision, generally less than FIB and laser cutting	High precision (micrometer scale), but less than FIB	Extremely high precision (nanometer scale)
Material removal	Sputtering, which can be uneven depending on material properties	Vaporization of material, less controlled	Layer-by-layer, highly controlled
Thermal damage	Minimal direct thermal damage, but ion bombardment can induce some heating	Significant thermal damage due to localized heating	Minimal
Surface finish	Can be rough, depending on the material and milling duration	Rough, with possible heat-affected zones	Smooth, ideal for high-resolution imaging
Speed	Moderate, faster than FIB but slower than laser cutting	Fast, depending on material thickness	Relatively slow, due to the high precision required
Cost and complexity	High cost, requires specialized equipment and expertise	Moderate cost, more accessible technology	High cost, requires specialized equipment and expertise

Conclusion

This study provided a comprehensive comparison of three advanced techniques—Ion Milling, Laser Cutting, and Focused Ion Beam (FIB) Milling—for the preparation of sponge spicule cross-sections aimed at high-resolution imaging. While ion milling and laser cutting were effective in various material science applications, their use in preparing delicate biological structures like spicules introduced significant challenges. Ion milling caused surface damage and uneven material removal. Laser cutting, on the other hand, introduced thermal damage and micro-cracks. Both of these issues compromised the structural integrity of the spicules and obscured fine details essential for accurate analysis. In contrast, FIB milling emerged as the most effective technique, providing smooth, artifact-free cross-sections with minimal thermal and mechanical stress. The precision and control offered by FIB milling, along with its real-time imaging capabilities, made it the preferred method for preparing fragile biological specimens similar to Euplectella aspergillum spicules. The findings of this study have important implications not only for the sample preparation of marine sponges but also for the broader application of these techniques in biomimetics and materials science, where preserving the integrity of complex structures is essential.

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Part II
Sustainable Composites and Biocomposites

Towards Sustainable Construction: Characterization of Vegetable Polyurethane Composite Reinforced with Fique Fiber for Warm and Humid Environments

L. R. Arrubla Agudelo, N. T. Simonassi, L. F. Fortunato de Freitas, C. M. Fontes Vieira, H. A. Colorado Lopera, and F. Perissé Duarte Lopes

Abstract This study investigates the mechanical properties of a new composite material consisting of castor oil-based polyurethane resin reinforced with linearly arranged fique fibers. The main objective is to develop a material with high flexural strength and toughness, making it suitable for construction applications. These properties are especially relevant for construction applications in humid environments and at high temperatures. To evaluate the durability and resistance of the composite to adverse conditions, an accelerated aging test was performed in a controlled chamber. Hardness measurements were taken before and after the aging process to evaluate the behavior of the material under simulated environmental stress. The results of this study provide information on the potential of this composite material for construction applications with an interesting possibility of being used as thermal insulation in roofs, highlighting its mechanical strength and its resistance to adverse environments.

Keywords Composite material · Castor oil-based polyurethane · Fique fiber · Mechanical properties · Flexural strength · Toughness

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Introduction

The growing global population has driven an increasing demand for innovative and sustainable materials. Composite materials, combining the properties of diverse components, offer a promising solution. Among them, natural fibers, derived from biological sources, have gained significant attention due to their environmental benefits and potential for enhancing material properties. The incorporation of these fibers not only reduces reliance on synthetic materials but also minimizes the environmental impact associated with traditional composite manufacturing processes [1–3].

Fique Fibers are extracted from the fique plant (*Furcraea andina*), native to the Andean region of South America, which has characteristics very similar to Sisal and Henequen [4]. Fique fiber, a resilient natural material, has emerged as an attractive reinforcement for composite structures due to its exceptional physical, chemical, and mechanical properties have fueled a surge in research and development. This coupled with a bulk density value of 723 kg/m^3, one of the lowest in the common studied fibers, makes it ideal for use in composite materials due to its low weight [5]. Unlike synthetic alternatives, fique fiber offers a sustainable and biodegradable option, contributing to a more environmentally friendly manufacturing process. In addition to the above, castor oil-based polyurethane (PU) is used, which is an alternative resin to the commonly used synthetic resins, giving the material studied the property of being a bio-composite material where the reinforcement fiber and matrix come from renewable sources, which is a very advantageous property thinking about the fulfillment of the Sustainable Development Goals (SDGs) proposed by the United Nations Organization (UNO). The Sustainable Development Goals related to this research are shown in Fig. 1.

This study focuses on the development of a novel composite material utilizing fique fiber as reinforcement in a castor oil-based polyurethane matrix. The combination of these natural components aims to create a material with enhanced mechanical properties, durability, and a reduced environmental impact. By leveraging the unique characteristics of both the fiber and the resin, we seek to produce a composite suitable for a wide range of applications, including construction, automotive, and consumer goods.

Fig. 1 Sustainable Development Goals (SDGs) involved in the present research

Fique fiber, derived from the leaves of the *Furcraea Andina* plant, has garnered significant attention as a reinforcement material in composite structures due to its remarkable physical, chemical, and mechanical properties. These properties include high tensile strength, stiffness, and low density, which make fique fiber an attractive alternative to synthetic fibers in various applications, particularly in the field of composite materials. Research has demonstrated that fique fibers can enhance the mechanical performance of composites, as evidenced by studies showing improved tensile and flexural strengths when fique fibers are incorporated into polymer matrices [5–7]. To evaluate the durability and resistance of the composite material to adverse conditions, an accelerated aging test was conducted in a controlled UV chamber. This type of test simulates the environmental conditions to which the material could be exposed during its service life, such as exposure to humidity, temperature changes, and UV radiation. By subjecting the material to these extreme conditions, its degradation can be evaluated, and its expected service life can be determined. Accelerated aging is essential to ensure that the composite material is suitable for outdoor or demanding applications. By understanding how the material degrades over time, areas for improvement can be identified and strategies can be developed to increase its durability. Additionally, this information is crucial to assess the material's potential as a thermal insulator, as exposure to high temperatures can affect its thermal properties. For instance, studies have shown that accelerated aging can lead to notable degradation in the mechanical properties of composites, including reductions in tensile strength and flexural strength, as well as changes in thermal properties due to the breakdown of the polymer matrix [8–10].

By evaluating the mechanical and thermal properties of the composite material before and after aging, its ability to maintain its performance under real-world conditions can be determined. If the composite material retains its mechanical and thermal properties after being exposed to adverse environmental conditions, it can be considered a promising candidate for applications in construction, where weather resistance and thermal insulation are critical factors. In summary, the exceptional properties of fique fiber, coupled with ongoing research and development efforts, position it as a promising reinforcement for composite materials, paving the way for innovative applications in sustainable engineering and materials science [11, 12]. The sum of the mechanical properties of the developed material, with the thermal properties and the chemical and UV radiation resistance, gives this material very important properties in insulation and mechanical reinforcement structures for roofs that can be used in areas where high temperatures are reached, helping to maintain the temperatures inside the houses in reasonable values for the realization of the basic activities of the inhabitants.

In the following Fig. 2, we can see an alternative for the use of the material as a structural part of roofs providing thermal and acoustic insulation, characteristics that have been studied by authors such as [11, 13], and that in this case is complemented with the mechanical study of a composite material between a thermosetting resin and the fique fiber. The polyurethane can certainly increase the working temperature of the insulating material, being that at temperatures higher than 250 °C the fique fiber

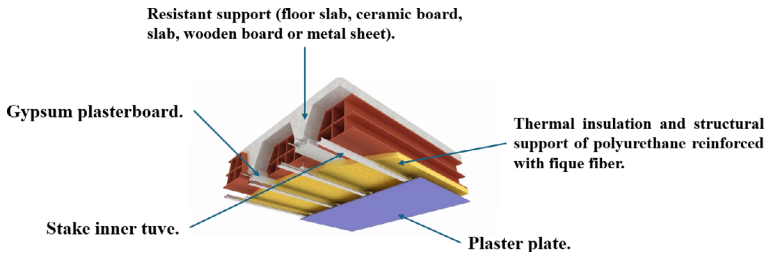

Fig. 2 Alternative for the use of the designed bio-composite material [25]

by itself begins a thermal degradation losing its properties due to the degradation of the cellulose chains [14].

Flexural strength and shore hardness measurements were performed on the biocomposite material using various volumetric proportions of fiber within the polyurethane matrix, 10, 20, 30, and 40 and compared with the resin without any reinforcement, i.e. 0%, following ASTM D790. The Shore hardness of the material before and after accelerated aging is also measured, the hardness being directly related to the preservation of most of the material properties.

Materials and Methods

Materials

Fique Fiber

The fique fiber used in this study was provided by one of the authors (Henry A. Colorado) in the form of a fiber fabric. The required fibers for specimen manufacturing were obtained. The fibers were treated using water and a metal comb to get clean fique fibers in a linear arrangement for subsequent added to the polyurethane matrix. In the following Fig. 3 is shown the stages in the obtention of the align and cutting fique fibers used in the manufacturing of the biocomposite Fique-PU.

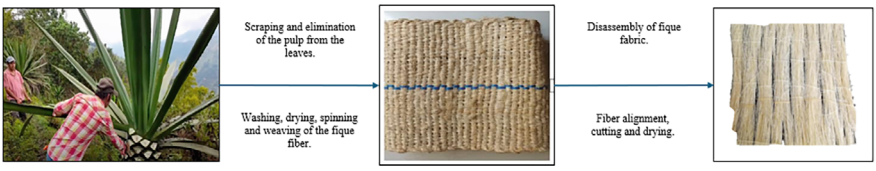

Fig. 3 Stages in the production of cut, aligned, and dried fique fibers

The fibers are cut to a length of approximately 12.7 cm, which is the lateral measurement of the mold in which the material is then prepared. Finally, the fibers are dried in an oven for 24 h at 60 °C, thus avoiding contamination of the specimen with moisture present in the fiber.

Polyurethane Oil-Based Polyurethane Resin

The used polyurethane, derived from castor oil, is the product of a cure reaction between a polyol that is synthesized from castor oil for the company Imperveg S.A. with identification code AGT 1315, with a density of 0.96 g/cm^3 and a specific mass around 1.0–1.2 g cm^{-3} [15]. The polyol, with a ricinoleic acid approximate amount of 89% [16], reacts with a coupling agent or prepolymer derived from diphenylmethane diisocyanate forming covalent cross-links bonds to generate the thermosetting polymer.

The prepolymer was synthesized from diphenylmethane diisocyanate (MDI) [15]. The isocyanate groups react with the hydroxyl groups present in the ricinoleic acid, generating cross-linked chains and providing greater rigidity to the resulting resin. It is beneficial to have two isocyanate groups per molecule as it increases the efficiency of the cure reaction. The prepolymer density is 1.22 g/cm^3, and the dynamic viscosity is between 170 and 250 mPa-s measured to 25 °C. The cure reaction occurs during 24 h at an average temperature of 27 °C in a closed compression mold.

Methods

Manual placement of the fibers is used to manufacture the samples. The fibers are carefully placed on the mold and impregnated with the polyurethane resin until the volume of fibers required for each formulation is completed. A hydraulic press is then used to obtain a pressure of 5 tons. It is demolded after 24 h obtaining a plate of material ready to be cut. A steel mold with dimensions of 15 cm long and 12.8 cm wide and a thickness of approximately 12 mm is used. Following ASTM D790, specimens were cut to measure approximately 60 mm wide, 12.5 mm high, and 10 mm thick. The test was conducted using an Instron model universal testing machine using a deformation rate of 1 mm/min and was established a distance between supports of 50 mm approximately.

Flexion Test

As illustrated in Fig. 5, the typical flexural force–deflection curves for fique reinforced polyurethane composite specimens exhibit a limited plastic deformation phase following the initial linear elastic region. Subsequently, a sharp decrease in load marks the onset of failure. From curves of force–deflection for each specimen,

the flexural strength σ_f (maximum bend stress) was calculated for each test specimen using the following Eq. (1):

$$\sigma_f = \frac{3F_m L}{2hd^2} \tag{1}$$

where F_m is the applied flexural force, L the distance between support points, h is specimen width (25 mm), and d is the specimen thickness (10 mm) [17]. A graph of the variation of the average flexural strength of the specimens corresponding to each percentage of fique fiber, 0, 10, 20, 30, and 40%, is made and compared by means of the standard deviation of the data. The same procedure is performed for the specimens of the studied material after being subjected to the action of an accelerated ultraviolet light chamber for 10 days, to verify the effect that aging has on the flexural strength of the Fique-PU material.

Shore Hardness

The Shore hardness of the specimens is measured using a Satra durometer. The hardness for the test bodies is measured before and after they pass through the UV accelerated aging chamber. For the hardness measurement the specimens are divided into three planes, and the hardness will be measured in each of these planes.

Shore hardness is a measure of a material's resistance to penetration by the needle of a durometer under a defined force. It is determined by numbers from 0 to 100 on scales A or D. The higher the number, the greater the hardness. The letter A is used for flexible types, and the letter D for rigid types. For the material studied, Fique-PU, it was found that Shore hardness tends to increase as the percentage of fique fiber added to the matrix increases.

To evaluate the possible anisotropy in the hardness of the samples, Shore hardness tests were performed on different planes of the Fique-PU material samples. Following ASTM D2240 standards, at least six measurements per sample were used to calculate the standard deviation. The choice of the Shore durometer was based on the expected hardness range of the composite material. As in Zhao et al. [18], the durometer was pressed on the sample surface with a specific force and speed to measure the depth of indentation. This methodology allowed to compare the hardness in different orientations of the fique fibers by varying the plane and to evaluate the influence of the fiber direction on the mechanical properties of the material. The orientation of the chosen planes is shown in Fig. 4.

A comparative graph is made between the hardness of the specimens with different fiber concentrations before and after aging, thus relating the changes in the hardness with the aging time, seeking to have an approximate characterization of the useful life of the material, functioning as a quality control of the designed material [19] and also verifying the probably anisotropic character of this material when using the compression molding method in its manufacture.

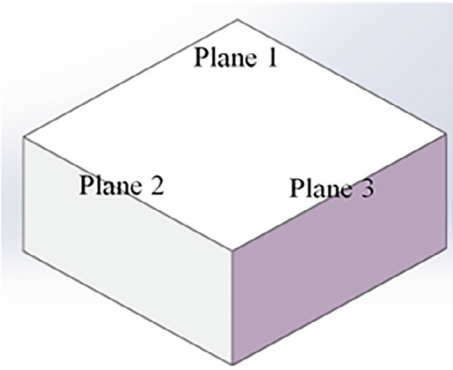

Fig. 4 Shore hardness tested planes. Zhao et al. [18]

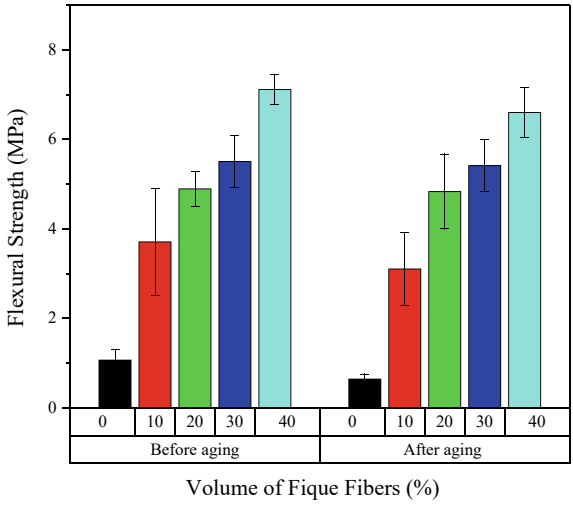

Fig. 5 Flexural strength before and after aging

Accelerated Aging

To evaluate the long-term durability of the Fique-PU samples, accelerated aging tests were carried out in a chamber exposed to UV radiation and condensation for 10 days, which is equivalent to approximately 1 year of aging under natural conditions. During the UV irradiation phase, the samples were subjected to a temperature of 60 °C and an irradiance of 0.5 W/m^2, using short-arc fluorescent lamps. Subsequently, condensation was induced at a temperature of 40 °C and a relative humidity close to 100%. This cycle was repeated continuously during the 10 days of testing, following the ASTM-G53/154 standard. The main objective of these tests was to evaluate the effect of accelerated aging on the mechanical properties of bending and Shore hardness of the material, as well as to evaluate the aesthetic changes of the fique—PU biocomposite material.

SEM Micrographs

SEM micrographs of the failure sites in the tested flexural specimens are performed. Micrographs are performed for the test specimens to evaluate the change that occurs in the failure mode of the specimens. A Tescan MIRA scanning electron microscopy was used, with an energy of 5 kev, thus avoiding a significant increase in temperature that could change the local characteristics of the material at the time of imaging at 500× magnification.

Results and Discussion

Flexion Test

The results of the flexural test, presented in Fig. 5, show a decrease in the flexural strength of the composite material after accelerated aging. This decrease is more pronounced in the samples with higher content of fique fiber (30 and 40%), suggesting that the fique fiber, despite improving the mechanical properties of the material in virgin state, may be more susceptible to UV-induced degradation. The decrease in flexural strength may be attributed to polyurethane degradation, weakening of the fiber-matrix interface, and possible degradation of the fique fiber.

The fiber-matrix interface in composites is crucial for effective load transfer. Degradation at this interface can severely compromise the mechanical performance of the composite. The interface is susceptible to environmental factors such as moisture and UV radiation, which can lead to debonding and reduced adhesion between the fiber and matrix. This degradation can significantly impact the overall hardness and flexural strength of the composite material, as the load transfer efficiency diminishes with the deterioration of the interface [20].

Shore Hardness

Polyurethane degradation is a significant concern in the field of materials science, particularly when exposed to environmental factors such as UV radiation and humidity. Indeed, it was reported that UV radiation initiates localized melting of the polyurethane films, which results in a decline in molecular chain length and then subsequent reduction in hardness [21]. The following Fig. 6 shows the Shore hardness measurements for the designed Fique-PU composite material, for various volume fiber fractions.

Several studies have shown that UV exposure alters the chemical and physical properties of polyurethane, including discoloration and mechanical degradation,

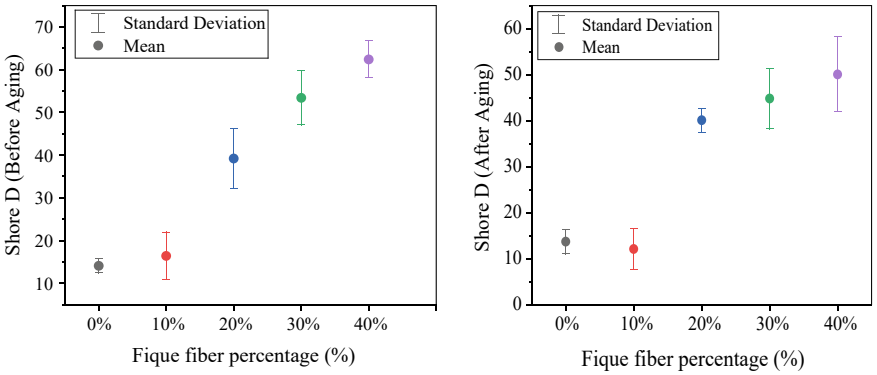

Fig. 6 Shore hardness before and after aging

which is primarily attributed to the breakdown of urethane linkages and the formation of free radicals that further propagate degradation processes [22, 23]. With the polyurethane being the majority material in the composite material studied, it is evident that exposure to the conditions simulated in the chamber generate some degradation although the hardness continues to be functional for the purpose of the material.

SEM Micrographs

SEM micrographic analysis shown in Fig. 7 reveals a predominantly brittle failure mode in the composite material, characterized by evident fractures in the relatively large diameter, rough-surfaced fique fibers. Bond separation at the fiber-matrix interface, evidenced by areas of detachment, suggests that this has been a critical point in fracture propagation. These results indicate that the combination of weak interfacial adhesion and fiber brittleness contributed significantly to the failure of the material during the flexural test.

It can be considered that the predominant failure mode in this composite material has been a combination of brittle fracture of the fibers and debonding at the fiber-matrix interface.

In the results obtained, a slight decrease in hardness and flexural strength was observed after subjecting the material to the accelerated chamber. However, the environmental conditions to which the material will be exposed are significantly friendlier than the extreme conditions simulated in the chamber since it will have structural coverings that protect the material providing greater durability without significant loss of mechanical, chemical, and thermal characteristics.

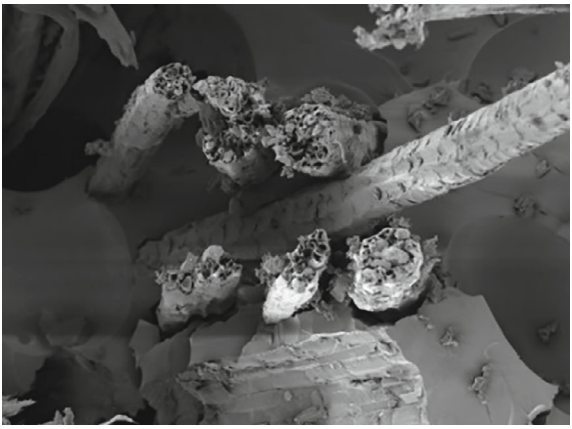

Fig. 7 SEM micrograph before using accelerated aging

Conclusions

- The oxidation of fique fibers, as a natural material, poses a risk to the mechanical properties of the composite. When exposed to environmental conditions, the fibers can undergo oxidative degradation, which affects their structural integrity and mechanical performance [24]. This oxidation process can lead to a reduction in the tensile strength and stiffness of the fibers, consequently impacting the hardness and the flexural strength of the composite.
- The interaction between fiber oxidation and the degradation of the polyurethane matrix further complicates the durability of the composite, as both components are interdependent in maintaining the overall mechanical properties. It is for this reason that it is concluded that in the purpose of using the material as thermal insulation and structural support in hot climates, there will not be great advantages to increase the volumetric percentage of fiber much more than 40%, since the material will be less resistant to the states of heat and humidity for which it is designed.

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Exceptional Strength of Mycelium-Bound Composite: A Sustainable Brick Alternative for Construction

Deepak Sharma and Hortense Le Ferrand

Abstract Mycelium-bound composites (MBCs) are grown by fungi onto waste lignocellulosic substrate and hence hold significant potential as sustainable materials. However, their wide range of adoption is limited by their typically low strength. Low strength in MBCs is due to the root-like networks of hyphae. The mycelium filaments loosely bind the organic structure, resulting in a porous and lightweight material that lacks the rigidity required for structural applications. This study develops a new method to fabricate MBCs. We use additive manufacturing to fabricate porous triply periodic minimal surface (TPMS) scaffolds from wood-Poly Lactic Acid (PLA) material. The porous TPMS structure provides a higher surface area and a continuous supply of nutrition and oxygen for mycelium development. Mycelium from *Ganoderma lucidum* is grown on porous structures for 21 days. This study considers two types of TPMS structures: gyroid (G) and inverted wrapped package (IWP). The resultant MBCs showed exceptional strength of 14 MPa, comparable to clay bricks. A comparison between porous structure with and without mycelium showed a 1.27 times improvement in peak strength for the G structure and 1.30 times for the IWP structure at 50% relative density. Mycelium growth depends on the relative density of the organic porous structure, with a maximum mycelium density on 10% and a minimum on 50% porous structure, respectively. Furthermore, results showed mycelium growth is dependent on the design of the porous structure, which opens an avenue for advanced and engineered MBCs.

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Keywords Mycelium-bound composites · 3D printing · Sustainable building materials · Bio-composites

Introduction

As global population expands, the demand for construction materials is increasing, causing significant challenges in terms of sustainability. Traditional resources like steel and concrete put pressure on natural ecosystems, with intensified deforestation and the use of toxic adhesives in application [1]. While alternatives such as mass-engineered timber and engineered bamboo composites hold potential, their safety and sustainability still require thorough investigation [2–4]. Therefore, it is crucial to explore fully recyclable, environmentally friendly building materials that can sustainably support the needs of future cities without compromising ecological integrity.

One such solution involves the creation of engineered hybrid-living materials that are made by growing living organisms on either biological or synthetic scaffolds. This approach allows the material-producing and regenerative capabilities of these organisms while also adjusting aspects like shape and the type of substrate [5–7]. Such innovation has led to the development of technologies such as bacterial cellulose composites, self-healing bacterial concrete, and mycelium-bound composites (MBCs), with potential applications in broad areas like medical devices, packaging, furniture, and building materials [8–10].

MBCs have been recently studied for their high thermal insulation, fire resistance, sound absorption, and biodegradability [11–14]. Even though MBCs have good functional properties, they still lack mechanical strength which restricts their application to non-structural applications [15]. For instance, a hemp-based mycelium foam produced by Ecovative Design LLC has a modulus of 1.14 MPa, similar to that of polystyrene foam [16]. MBCs have been mostly fabricated using moulding and 3D printing methods. Moulding mostly leads to simple shapes and open voids as moulds must be designed to open and close in a way that ensures the moulded material can be removed easily. 3D printing instead offers some design freedom but generally results in MBCs with even lower stiffness. The mechanical and pneumatic extrusion-based methods also suffered from low printing resolution, hence limiting the shaping ability to only crude shapes [17, 18].

In this work, we propose a new method of the fabrication of high strength MBCs. The method uses Fused Deposition Modeling (FDM) to 3D print porous wood-PLA organic scaffolds in the shape of triply periodic minimal surfaces (TPMSs). Due to their high mechanical strength and high surface area, this study considers two TMPS-based structures, gyroid (G) and inverted wrapped package (IWP) [19, 20]. This study compares the mycelium growth of *Ganoderma lucidum* on these structures at different relative densities and measures their mechanical strength. The results show exceptional mechanical strength and tuneable response of the MBCs.

This opens an avenue for advanced high-strength MBCs to replace traditional bricks with more sustainable and functional buildings.

Materials and Methods

Materials

The Wood-PLA filament of diameter 1.7 mm was procured from Filamentive Ltd England. The filament is made from 40% recycled wood fibers and 60% PLA. Peptone, malt extract, and agar powder were purchased from Sigma-Aldrich. *Ganoderma Lucidum* (*G. Lucidum*) spawn was obtained from commercial farm Malaysian Feedmills Farms Ltd.

Liquid Mycelium Culture

Mycelium spawn was used to prepare the inoculum. 2.5% w/v agar solution in distilled water was stirred for 5 min, followed by sterilization in an autoclave (Hirayama, HG-80) at 120 °C for 1 h and 30 min of warming at 50 °C. Sterilized agar solution was poured into 90 mm petri dishes. A small agar plug was placed at the centre of the dishes. The agar plug is a small cutout from a previous mycelium petri dish. The complete process is carried out in a bio-safety cabinet (Gelman, BH Class II) under ultraviolet light to avoid contamination. Agar plates were stored in a dark enclosed cabinet for 21 days. The inoculums from the agar plates were transferred into the culture media prepared from 1.7% w/v malt and 0.3% w/v peptone in distilled water and sterilized similarly. The liquid mycelium culture was stored for 14 days before it could be used to inoculate the porous scaffolds.

Nutrition Rich Solution

Mycelium needs a continuous supply of nutrients to grow efficiently. Because we intend to grow mycelium onto Wood-PLA, the amount of nutrients that the mycelium can naturally absorb is limited. Therefore, peptone (P), malt (M), and agar (A) were combined to create a nutrient-rich solution referred to as the PMA solution. Indeed, amino acids are found in peptone, carbohydrates are found in malt, and moisture and adequate rheology are maintained and adjusted using agar. Repetitive trial tests were used to determine the solution's composition. For the mycelium to grow best, a solution containing 0.1% w/v peptone, 5% w/v malt, and 2.4% w/v agar in de-ionized water was finalized. To prevent contamination during the incubation phase, the PMA

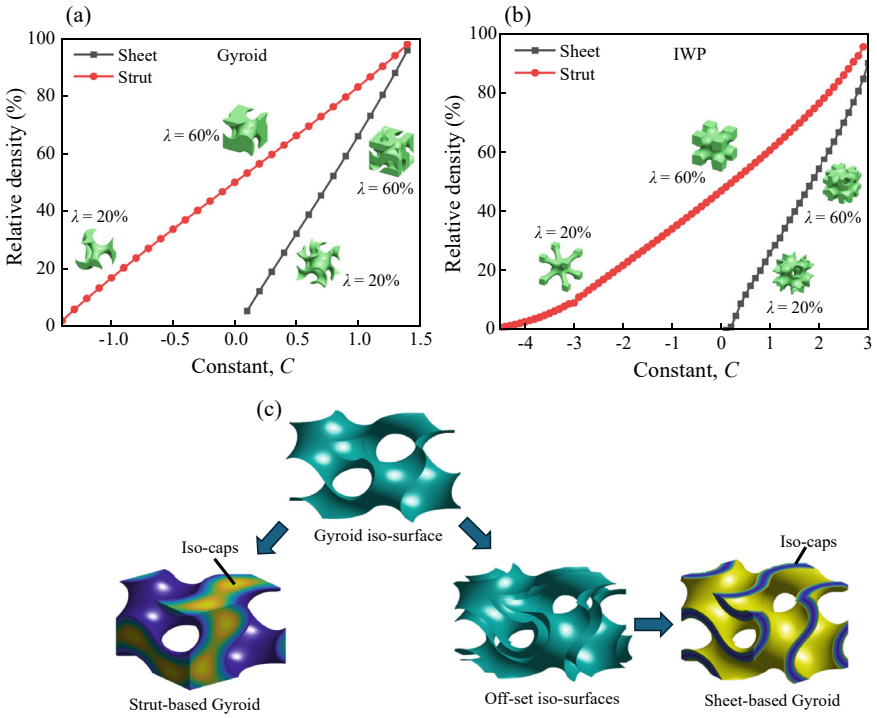

Fig. 1 Comparison of relative density variation with level-set constants for sheet-based and strut-based TPMS structures: **a** Gyroid, **b** I-WP, and **c** approaches to model TMPS unit cells

solution was autoclaved for one hour at 120 °C, followed by warming at 50 °C for 30 min.

Design

Mycelium growth depends on the surface area of the porous scaffold. Due to their high surface area, G and IWP were selected for the current work. The level-set equations for G and IWP iso-surfaces are given in Eqs. 1 and 2. Porous structures with different porosities were modelled in MATLAB R2024b. The relative density (λ) of the structures depends on the level-set constant *C*. The relations between λ and *C* for both G and IWP are shown in Fig. 1a, b:

$$\sin x.\cos y + \sin y.\cos z + \sin z.\cos x = C \tag{1}$$

$$2(\cos x.\cos y + \cos y.\cos z + \cos z.\cos x) - (\cos 2x + \cos 2y + \cos 2z) = C \tag{2}$$

$$\lambda = 1 - \Phi \tag{3}$$

where $x = \frac{2\pi X}{L_x}$, $y = \frac{2\pi Y}{L_y}$, $z = \frac{2\pi Z}{L_z}$, and L_x, L_y, and L_z decide the unit cell size in principle directions (X, Y, Z), respectively. C controls the relative density of the structure, which is also known as the level-set constant and Φ is the porosity of the structure.

The iso-surfaces generated from Eqs. 1 and 2 can be converted into solid structures in two ways. Both ways are explained in Fig. 1c. In the first approach, one of the domains in the minimal surfaces is solidified by iso-caps, and this group is referred to as solid/network/strut-based TPMS structures. In the other approach, the ends of the off-set minimal surfaces are closed with iso-caps and extended off-set sets the λ of the structure, and this group is referred to as the sheet-based TMPS structures. The sheet-based TPMSs have better-defined curvature as compared to strut-based TPMSs.

Another problem with a strut-type structure is the lower limit on λ (Pinch-off problem); below this λ, the structures lose their connectivity at the nodes [20]. For instance, G TPMS has a lower limit of 10%, and below this value, the nodes lose their connectivity. Nodes also have the least cross-sectional area and are the point of stress concentration. Furthermore, the total surface area of the sheet TPMS is much higher than the solid TPMS. For a 1 mm length, the surface area of sheet G and IWP are 7.45 mm^2 and 8.03 mm^2 for strut G and IWP are 4.85 mm^2 and 4.62 mm^2. Therefore, the current study is restricted to only sheet G and IWP TPMSs due to their higher surface area supporting mycelium growth.

In this study, three relative densities considered were 10%, 30%, and 50% with 20 mm unit cell size and structure as a cube with a 40 mm side length.

3D Printing

The current work uses an industrial-grade FDM printer Flashforge Creator 3 Pro, with wood-PLA composite filament. This filament was selected with the understanding that mycelium thrives on lignocellulosic biomass. PLA, which is also biodegradable, is used to improve the material's printability. Wood-PLA filament was printed with the typical commercial printing parameters for PLA.

Porous MBCs Preparation

Prior to applying a thin layer of PMA solution onto the porous structures, every 3D-printed scaffold underwent a 30 min UV sterilization. The PMA solution tends to harden at room temperature because of the agar. As a result, each sample was submerged for 10 s right after making a fresh solution. The PMA-coated samples

were once more stored under UV light to eliminate any chance of contamination. Using a micropipette, 1 ml of mycelium liquid culture was added on the top surface of each sample. Although other various mycelium strains could be used, *G. lucidum* was selected due to its natural tendency to thrive on hardwood and woody substrates [21]. The entire procedure was conducted in a biosafety cabinet. Figure 2a displays the entire process flowchart. Subsequently, every sample was housed in an individual container, sealed against dust using parafilm while permitting ambient air to permeate inside. For 21 days, the containers were kept in a dark cabinet at 23 °C and 80% relative humidity. Figure 2b displays images of the IWP samples taken on various days during incubation demonstrating the efficiency of the process.

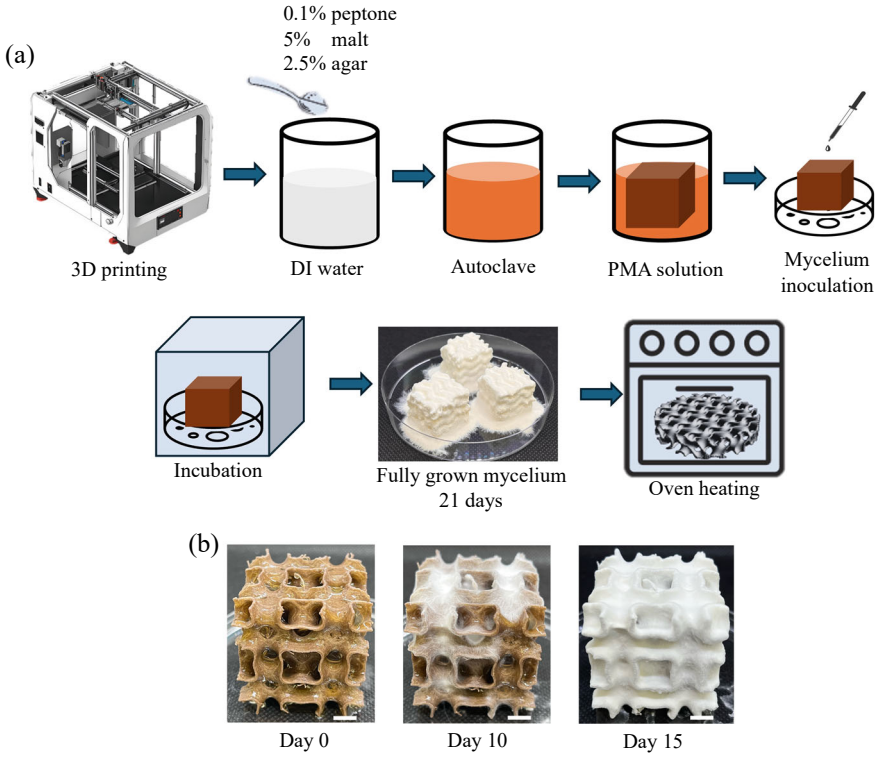

Fig. 2 Fabrication process and growth dynamics of mycelium-based composites: **a** Stepwise fabrication process of MBCs, and **b** Mycelium growth progression on IWP TPMS over different days

Characterization

The grown MBC samples were dried overnight in an oven (IKA, Malaysia) at 48 °C before further characterization. A field emission scanning electron microscope (JOEL JSM-6360) was used to capture electron micrographs. Sputter coating with 5 nm of gold using a mini sputter coater (Quorum SC7620) was carried out to improve the electrical conductivity of the samples. The micrographs were analysed using Image J to measure the mycelium layer density (used threshold to isolate mycelium from pores) and hyphae diameters. Compression tests were carried out using an MK-2100S universal testing machine according to ASTM D1621 standard. A 5 mm/min loading rate was selected for a steady and controlled compression.

Results and Discussion

Mycelium Growth

The growth of the mycelium is dependent on the medium. Figure 3 compares the growth of mycelium in different growth mediums showing efficient growth of the mycelium in the conditions determined in this study. SEM electron graphs show dense mycelium growth in liquid mycelium culture as compared to petri dish and porous structure. However, there is no significant difference between the average hyphae diameter (d_a) and mycelium layer density (r_m).

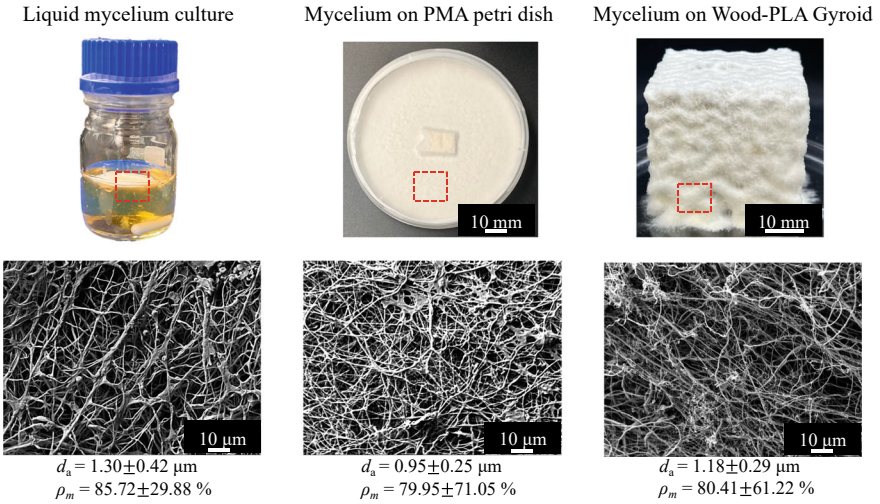

Fig. 3 Mycelial growth parameters across different media: Liquid culture, Agar plate, and Wood-PLA show microscopic fibrous network, average hyphae diameter, and density of mycelium layer

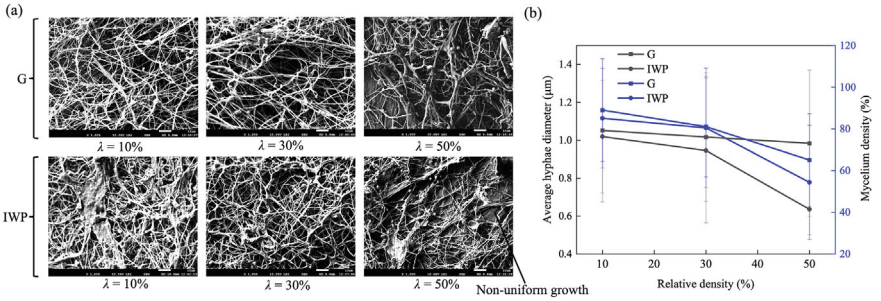

Fig. 4 **a** Electron micrographs of the mycelium skin grown in different porous structure design and relative densities and **b** change in average hyphae diameter (μm) and mycelium density with relative density of the porous scaffolds

The improvement in mechanical properties directly depends on the growth of mycelium on the porous structures. The growth of the mycelium was characterized using the electron micrographs. Figure 4a shows electron micrographs of mycelium grown on G and IWP porous structures of different relative densities. The results show that the average diameter of the hyphae (mycelium filaments) is almost similar for all the G TPMS structures. However, the density of the mycelium decreases with an increase in the relative density of the porous scaffold. Furthermore, no significant difference was observed in the mycelium grown on G and IWP structures at $\lambda = 10\%$ and 30%; however, the IWP structure with $\lambda = 50\%$ shows non-uniform growth, suggesting the less supply of air for proper growth of the mycelium.

Mechanical Properties

Compression test results show improvement in mechanical properties after mycelium growth for both G and IWP structures. The increase in peak strength and energy absorption is presumably caused by the mycelium acting as a foam between the unit cell walls. The deformation mechanism for G30 and G30_my structures at different strains is shown in Fig. 5a. G30 refers to a Gyroid structure with 30% relative density and without mycelium, whereas G30_my represents the same structure with mycelium. The presence of the mycelium helps the structure to deform uniformly, whereas the G30 structure without mycelium tends to globally buckle, which decreases the strength and energy absorption of the structure. Values show an increment in both peak strength and energy absorption after mycelium growth. In addition, G structures show higher mechanical performance than the IWP structures.

All the stress–strain curves shown in Fig. 5b follow the same pattern. In the elastic region, the stress value increases sharply as the load is below the elastic limit of the structures. As the strain increases, the unit cell walls start to buckle resulting in fluctuation in the stress value. When the strain value increases above

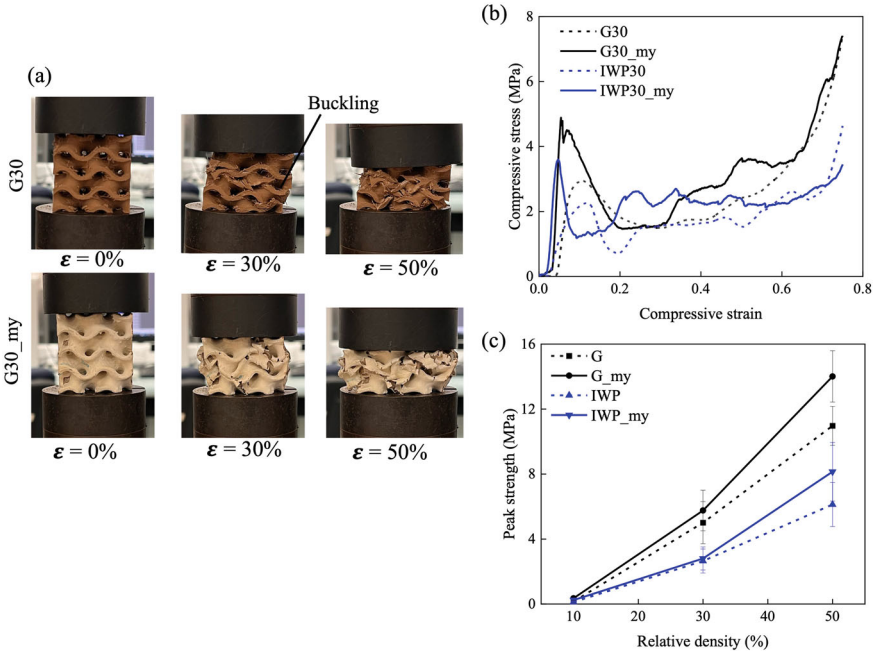

Fig. 5 **a** Comparative deformation of the mycelium and without mycelium structures, G TPMS structure at different strains (*e*). **b** Compressive stress–strain curves of the G and IWP mycelium and without mycelium structures at $\lambda = 30\%$. **c** Peak strength at different relative densities

the porosity of the structure, the unit cell walls enter in contact with each other, resulting in an abrupt increase in stress value. Mycelium acts as a cushion between the cell walls, hence improving the overall performance, especially in the plateau and densification region. The numerical values calculated from mechanical tests are presented in Table 1. Figure 5c shows the change in peak stress value with relative density for G and IWP structures. For both structures, peak stress increases with relative density, and for G structure peak stress is higher than IWP structures.

Conclusions

The current study has reported using 3D-printed wood-PLA porous structures as a matrix to grow mycelium. The results showed similar mycelium growth across different mediums. Further, the study is focused on design, with MBC's performance dependent on the relative density of porous structure and type of unit cell. The current method of MBC fabrication has achieved strength as high as 14 MPa, which has never been reported for MBCs, which opens a new way of fabricating high-strength MBCs.

Table 1 Mechanical properties calculated from compression tests. (*Note* Peak strength is measured at 70% strain)

Relative density of the structure (%)	Peak strength (MPa)	Peak strength (MPa)_with mycelium	Energy absorption (kJ/kg)	Energy absorption_ with mycelium (kJ/kg)
Gyroid samples				
10	0.16 ± 0.05	0.35 ± 0.13	0.05 ± 0.03	0.11 ± 0.05
30	5.01 ± 1.3	5.75 ± 1.25	1.85 ± 0.66	2.25 ± 0.9
50	10.97 ± 1.2	14.01 ± 1.58	5.95 ± 0.95	8.33 ± 1.3
IWP samples				
10	0.13 ± 0.04	0.24 ± 0.07	0.03 ± 0.008	0.08 ± 0.04
30	2.64 ± 0.74	2.80 ± 0.71	1.33 ± 0.51	1.64 ± 0.51
50	6.12 ± 1.35	8.14 ± 1.8	3.95 ± 0.51	6.24 ± 1.1

In the future, studies on the reliability, sustainability, and multifunctional properties of the porous mycelium structure could be focused. Porous MBCs showed strength comparable to clay bricks, suggesting the potential replacement of the pollution-causing clay bricks with sustainable MBCs. Porous materials offer low thermal conductivity, sound absorption, and a high strength-to-weight ratio, enhancing the multifunctional properties of building walls. The cushioning effect of mycelium porous structures significantly enhances wall vibration absorption, suggesting applications in earthquake-resistant buildings.

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Processing of Composites Incorporated Within 3D Printing as a Potential Way of Producing High-Performance Glasses

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Abstract UV-curable 3D printing has shown extensive applications; sports and odontology materials are examples of this application. Therefore, this study aims to develop and apply high-performance glasses in polymer composites with glass particulate waste. The incorporation of glass residue into the photosensitive resin for 3D printing does not alter the printing capacity, leaving the items with high precision. Adding the glass residue to the print resulted in a relative improvement in toughness. The reuse of glass waste, makes it possible to reduce the waste of resources that have potential to be used in the production of high-performance glasses.

Keywords 3D printing · Polymer composites · Glass waste · Izod impact · Spectacle frames

Introduction

The growing application of 3D printing in sport has revolutionized the development of high-performance equipment. This technology allows for the creation of highly personalized products that meet the specific needs of athletes, from shoes to helmets. The possibility of producing parts with complex geometries and advanced materials puts 3D printing at the forefront of sports innovation, providing improvements in performance and safety [1, 2].

When it comes to sports glasses, 3D printing is starting to transform the way these accessories are produced [3]. Some brands, such as Oakley, have experimented with the use of this technology to create spectacle frames that perfectly fit the shape of the wearer's face, something that is fundamental for athletes who depend on clear visibility and protection during sports practice.

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The traditional eyewear manufacturing process involves injection moulding, which, although efficient for mass production, limits customization and can result in products that are less adaptable to the wearer's individual needs [4]. 3D printing makes it possible to manufacture lightweight, durable frames with aerodynamic designs that can be adjusted according to the specifics of each sport [5].

As such, 3D printing technology is advancing rapidly, especially in the sports sector, where customization is crucial [6]. High-performance athletes are looking for equipment that not only fits their bodies perfectly, but also improves their performance in competition [2]. 3D printing offers flexibility, allowing for the creation of bespoke items such as customized insoles, helmets with optimized ventilation, and even shoes with complex internal structures that would be impossible to manufacture with traditional methods [7]. This technology also makes rapid prototyping possible, shortening the development cycle for new products and allowing for precise adjustments before large-scale production [8].

In this sense, the use of waste glass in the manufacture of composites for 3D printing represents a significant innovation, both from an environmental and technical point of view [9]. Since glass is a widely recyclable material, it can be reprocessed and incorporated into new products, reducing the amount of solid waste and the demand for virgin raw materials [10].

In the research in question, waste glass is used as a reinforcement in composites, helping to improve the mechanical and thermal properties of printed materials. This innovative composite not only maintains the durability required in high-performance eyewear, but also offers a sustainable solution, in line with growing environmental concerns in the development of new products [11].

As such, photopolymerization is a key process in various 3D printing applications, especially in the production of parts that require high precision and durability, such as high-performance glasses. In this process, photosensitive materials, such as liquid resins, are solidified using UV light or another specific light source, which initiates polymerization and transforms the liquid resin into a rigid solid.

This technique is widely used due to its ability to produce extremely fine details and high-quality surface finishes, essential characteristics for accessories such as glasses, where precision and comfort are crucial [12, 13].

Therefore, 3D printing technology based on photopolymerization, such as stereolithography (SLA) and Digital Light Processing (DLP), has been particularly little found in the literature to produce spectacle frames. These methods allow the creation of complex and customized designs with high resolution, something that would be difficult to achieve with traditional methods [14, 15].

Photopolymerization also offers the possibility of using a wide range of materials with varying properties, such as flexibility, rigidity and impact resistance, all of which are important when manufacturing glasses that meet the demands of different sports disciplines [16].

Fig. 1 Culminated and sieved material

Therefore, the aim of this study is to manufacture composites using a 3D printer by photopolymerization, incorporating glass particulate waste. Mechanical characterization will then be carried out using the izod impact test to evaluate the properties of the composites, with a view to possible applications in the manufacture of high-performance spectacle frames.

Materials and Methods

Glass Waste

The material was prepared using glass waste, which was sourced from bottles and packaging. The glass was milled in a ball mill for about 24 h. Following the grinding process, the material was sieved through a 100 Mesh sieve (0.150 mm) to reduce the aspect ratio of the final product. Below is a Fig. 1 shows the waste after it has been sieved.

Light-Curing Resin

The 3D cure brand resin has flexible properties and is designed to be compatible with 3D printers based on LCD and DLP technologies that use UV light sources

Table 1 Properties of the resin used [17]

General characteristics	
Rigidity	Low
Flexibility	High
Impact resistance	High
Shore D hardness	35–40
Temperature resistance	Up to 200 °C
Tear resistance	Low
Easy printing	Low
Density	1,1 g/cm^3

with a wavelength of 405 nm. Due to their chemical composition, the resins in the Flex range are not biocompatible and should only be used in external applications.

These resins have a low viscosity, which contributes to reducing material waste, minimizing the formation of excess residue on the surface of printed parts and facilitating the process of cleaning the resin tank. In addition, Cure Flex offers an excellent surface finish and cures quickly, optimizing production time. Table 1 shows the properties of the resin used in the research.

3D Printer

The Anycubic Photon Mono 2 4 K resin 3D printer, illustrated in Fig. 3, is an advanced device that uses SLA (Stereolithography) technology for additive manufacturing. Equipped with a 405 nm wavelength UV light source, this equipment is ideal for producing parts with high precision and detail.

Key features include

- **Ultra-high resolution**: The printer has a 6.6-inch 4 K monochrome LCD screen, with a resolution of 4096 × 2560 pixels, allowing fine details to be reproduced with micrometric-scale precision, essential for applications requiring high definition.
- **Print volume**: It offers a print volume of 143 × 89 × 165 mm, making it suitable for a wide range of 3D printing projects, from small components to more complex parts.
- **Speed and precision**: The printer operates at a speed of 50 mm/h and allows the layer thickness to be varied between 10 and 150 μm, offering an optimized combination of production speed and finishing precision.
- **Software compatibility**: The equipment is compatible with slicing software such as Photon Workshop, Chitubox, and Lychee, as well as being operable on Windows, Linux, and MacOS systems, providing versatility and ease of integration into different working environments.

This set of features makes the Anycubic Photon Mono 2 4 K an effective tool for producing detailed parts in a variety of areas, including prototyping, industrial design, and scientific research.

Mechanical Tests

The izod impact samples were prepared in accordance with ASTM D256 [18]. The notch resistance and impact resistance properties were extracted using a Pantec XC-50 impact machine with a 22 J hammer, available at LAMAV (UENF).

Composite Processing

The particulates were incorporated into the resin using manual stirring for 5 min to maximize the homogeneity of the mixture. After homogenization, the resulting mixture was transferred to the printer reservoir for the additive manufacturing process. After printing, the composites were washed for 10 min, followed by curing under UV light for 10 min, ensuring complete solidification and stabilization of the material.

Scanning Electron Microscopy (SEM)

In the first image, the surface morphology reveals a highly irregular and rough texture, which is characteristic of brittle fracture. The glass particulates appear to be dispersed within the resin matrix, though there are noticeable voids and pores, suggesting areas where debonding or poor adhesion may have occurred during the impact or mechanical testing. These voids could be stress concentrators, potentially affecting the overall mechanical properties of the composite. The angular and sharp edges of the fractured surfaces of the glass particulates indicate the brittle nature of the glass residue. Additionally, the resin surrounding the glass residue seems to have fractured, exposing the glass particles and contributing to the jagged surface (see Fig. 2).

In the second image, a closer view shows a more detailed interaction between the resin matrix and the glass particulates. The glass particulates are embedded in the resin, with visible cracks propagating through the matrix and along the particle–matrix interface. This suggests that the composite may have undergone interfacial debonding or matrix cracking due to stresses applied during testing. The fractured surfaces indicate weak bonding between the glass particles and the resin, which is a key factor in impact resistance and energy absorption. The presence of small voids and cracks around the particulates may also be a sign of resin shrinkage during curing or inadequate wetting of the glass particles during the manufacturing process.

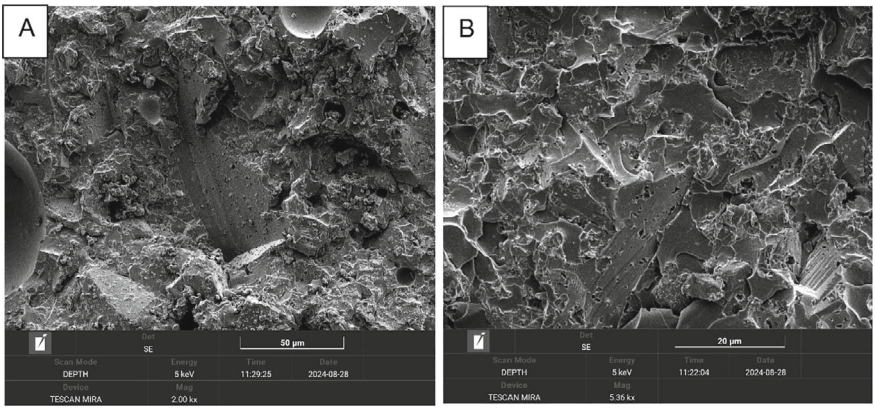

Fig. 2 Scanning electron microscopy of the composite

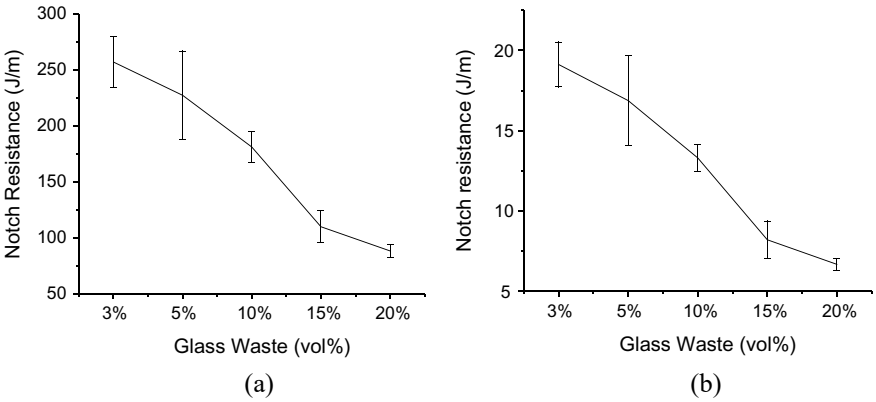

Fig. 3 **a** Notch resistance and **b** impact resistance

Overall, the SEM analysis of this composite highlights the challenges in ensuring good adhesion between the glass residue and the photocurable resin. The brittle nature of the glass, combined with the visible interfacial debonding and the presence of voids, could negatively impact the composite's mechanical properties, particularly in applications requiring high impact resistance. To improve the performance of the composite, optimizing the interface adhesion and reducing porosity would be essential steps in further development.

Results

The results showed that the notch resistance of the composites decreased significantly as the concentration of glass residue increased. In the corresponding figure, as the amount of glass incorporated increases, the material's ability to resist crack propagation decreases, indicating that the composite becomes more fragile. This behavior suggests that the addition of glass particles, which are stiffer than the resin, may have led to the formation of stress concentration regions, facilitating the propagation of flaws under load [19] (see Fig. 3).

Similar to the notch resistance, the impact resistance also decreased as the glass residue content increased. The data indicates that the composite lost some of its ability to absorb energy during impacts. The stiffness of the glass particulate, being greater than that of the resin, may have caused a less efficient energy distribution during the impact tests, resulting in a lower energy dissipation capacity and therefore a greater propensity to fracture.

The addition of glass residue to the composites resulted in an increase in the fragility of the materials, as evidenced by the reduction in notch and impact strengths. The intrinsic rigidity of the glass particles, in contrast to the polymer matrix, may have led to a poor distribution of stresses, compromising the structural integrity of the composites.

These results suggest that, although the incorporation of glass waste can be beneficial in terms of sustainability, it is crucial to carefully control the concentration of these particulates to avoid degradation of the essential mechanical properties of the composites, especially when aiming to produce high-performance glasses by 3D printing [20].

Conclusions

However, as the concentration of glass increased, the mechanical properties of the composites began to deteriorate, especially at concentrations above 10%, which suggests that excess glass residue can compromise the structural integrity of the materials. The behaviour observed at 15% concentration, where there is a partial recovery of the mechanical properties, indicates that there is an ideal concentration range in which the glass particles are well distributed, optimizing the characteristics of the material.

Therefore, the research demonstrates that by properly controlling the amount of glass waste incorporated, it is possible to develop composites that combine sustainability with high performance, making it possible to produce high-performance glasses through 3D printing. This approach not only contributes to innovation in the design and manufacture of glasses, but also promotes the sustainable use of recycled materials, in line with environmentally responsible development trends.

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Enhancing the Properties of Polyester Composites Using Unidirectional Acetylated Fibers from *Luffa Cylindrica*

Eduarda C. R. de Melo, Heliane R. Amaral, Roseméri B. S. dos Silva, Verônica S. Cândido, Felipe P. D. Lopes, Sergio N. Monteiro, and Michel P. Oliveira

Abstract Polymeric composites reinforced by lignocellulosic fibers are an eco-friendly option for various applications that utilize the benefits of natural fibers and polymers to create materials with improved mechanical properties and minimal environmental impact. In this study, we acetylated unidirectional fibers from *Luffa cylindrica* and incorporated them into a polyester matrix. X-ray diffraction and Fourier-transform infrared spectroscopy analyses confirmed the success of the acetylation process, replacing hydroxyl groups with acetyl groups. Moreover, thermal analysis showed that the acetylated fibers had no mass loss up to 200 °C, indicating their hydrophobic nature. Scanning electron microscopy displayed an excellent interface between the acetylated fibers and the polymeric matrix. The Charpy impact resistance for the acetylated fibers showed a remarkable 1865% increase compared to pure polyester. Additionally, the composite with acetylated fibers absorbed only 2.54% of water. These results demonstrate that lignocellulosic fibers' in situ acetylation process offers a more sustainable alternative to synthetic fibers.

Keywords Biomaterials · Composites · Polymers · Lignocellulosic fibers

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Introduction

The increased focus on sustainable materials has led to the widespread use of lignocellulosic fibers (LF) in various applications. This helps to reduce waste, optimize resource utilization, and produce more eco-friendly materials that align with principles promoting longer life cycles and reduced environmental impact. Due to their lightweight, high specific modulus, and ease of handling and processing, these fibers are commonly used in the automotive industry [1–3]. Natural fibers, which are also known as LF, can be derived from various sources such as plants, animals, and minerals. However, fibers of plant origin are most commonly used in polymeric composites [4]. These materials are mainly composed of cellulose, lignin, and hemicellulose. The hydrophilic nature of LF is due to the presence of hydroxyl groups (OH) in the cellulose and hemicellulose structures [5].

Natural fibers are gaining popularity in the field of composite reinforcement due to their affordability, biodegradability, and safe properties [3, 6]. Researchers have explored several types of natural fibers such as sisal [7], jute [8], bamboo [9], coconut [10], and Kenaf [11] for composite reinforcement purposes. *Luffa cylindrica* (LC) is becoming increasingly popular among those interested in biocomposite materials, commonly known as the vegetable sponge, belonging to the *Cucurbitaceae* family and cultivated in tropical countries in Asia and Africa [12]. In Brazil, the vegetable sponge is widely used for personal hygiene and household chores. Like other LF, the chemical composition of LC depends on external factors such as soil, climatic conditions, and plant origin, among others. It is estimated that LC fibers contain approximately 57–74% cellulose, 14–30% hemicelluloses, 1–22% lignin, and 0–12.8% other components [13], including ash and extractives.

Lignocellulosic fibers have a lot of inherent advantages. However, their mechanical properties are limited due to variations in their structure and composition. Their natural hydrophilicity also makes it difficult to use them with polymeric matrices, which makes it challenging to produce high-performance composites [14]. To overcome these limitations, researchers have explored chemical modification techniques that can reduce their hydrophilicity and make them compatible with polymeric matrices [12]. Various methodologies are described, such as mercerization, acid treatment, acetylation, silane application, isocyanates, permanganate, and peroxide [15]. Among these methods, the mercerization technique, also known as alkalization, is the most commonly employed pre-treatment technique before other stages in reinforcing the fiber. This process helps to remove non-cellulosic substances and increase the contact area with the matrix, and contributes to superior mechanical properties [5, 16].

Similar to alkaline treatment, the acetylation technique increases the fiber's surface area, but this process involves the replacement of hydroxyl groups with acetyl groups (CH_3CO). As a result, this process significantly reduces the fiber's hydrophilicity, which can decrease by up to 55% after 24 h of water immersion [17]. Acetylation creates a rough surface, thereby enhancing the potential for applications in composites [16]. D'Almeida et al. (2005) conducted a study to investigate the effect of

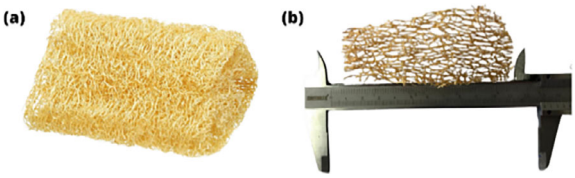

Fig. 1 *Luffa cylindrica* fibers **a** in nature and **b** after cleaning for application in polyester composites

chemical treatment on the structure and surface morphology of vegetable sponge fibers [18]. The researchers demonstrated a decrease in hydrophilicity following acetylation, which also led to the removal of the superficial layer of the vegetable sponge fibers. Consequently, the internal fibrous structure was exposed, resulting in an increased available surface area for interaction with the matrix.

The aim of this study is to investigate how acetylation affects the structural, morphological, thermal, and impact resistance characteristics of unidirectional fibers. The hypothesis is that by reducing the hydrophilicity of LC unidirectional fibers, their interaction with the polymeric matrix will increase, making them more suitable for use in polymeric composites. This, in turn, will contribute to the development of sustainable materials derived from lignocellulosic fibers, with a particular emphasis on promoting a circular economy.

Materials and Methods

Materials

The vegetable sponges were purchased at a local market in Vitória, Espírito Santo, Brazil. First, non-aligned fibers were cut off to obtain only unidirectional fibers, as shown in Fig. 1. Afterwards, they were dried in an air-circulating oven at 100 °C for 24 h. The materials used for the acetylation process included anhydrous acetic acid (P.A 100%, Labsynth), acetic acid (P.A 65%, Labsynth), sulfuric acid (P.A. 98%, Qhemis), and low-viscosity polyester resin (Redelease).

Acetylation Process of the Fibers from Luffa Cylindrica

The process of acetylation was carried out following the procedure described in D'Almeida et al. [18]. First, the fiber was washed with deionized water. Then, it was immersed in a solution containing acetic anhydride and acetic acid (in a ratio of 1.5:1.0 v/v) along with 20 drops of sulfuric acid for every 500 mL of solution. The mixture was kept in an ultrasonic bath for 3 h, and the fibers were left in the solution for 24 h. After that, the product was washed with deionized water at pH 7 and then dried in an air-circulating oven at 100 °C for 24 h.

Production of Polymeric Composites Using In-Nature and Acetylated Fibers from Luffa Cylindrica

To produce the composite material, 30% v/v of both in-nature and acetylated fibers were placed in a silicone mold, based on previous study findings [19]. The resin, along with 2% weight of the catalyst, was added to the mixture. The mold was then put into a compressed air reactor at 90 bar. After 24 h of curing, the polymerization process was complete. The samples were labeled as CFIN and CFAC, respectively. The polymeric composites with unidirectional fibers were produced in accordance with ASTM D6110 specifications [19] for impact mechanical testing. Five specimens were created for each type of composite.

Characterizations

Samples from FIN and FAC were subjected to X-ray diffraction (DRX) analysis using a Rigaku MiniFlex 600 diffractometer equipped with Cu-Kα radiation. The scanning angle (2θ) was adjusted to range from 3 to 70° with a 0.05° step, and the scan speed was set to 2° min^{-1}.

Fourier transform infrared (FTIR) spectroscopy—Experiments using the attenuated total reflectance (ATR) technique on a Bruker Tensor 27 spectrometer, analysing the region from 4000 to 600 cm^{-1} with 4 cm^{-1} resolution.

Thermogravimetric Analysis (TGA) was conducted on a LabSys Evo apparatus. Samples were heating from 25 to 700 °C at a rate of 10 °C/min under nitrogen.

Scanning Electron Microscopy (SEM), Shimadzu SSX-550, Tokyo Japan, was used to analyze the morphology of the materials. Samples were fixed, metalized with gold, and observed at 10 kV. Scanning Electron Microscopy (SEM) was utilized to observe the samples after they had been fixed and metalized with gold at 10 kV.

Charpy impact tests were conducted using a XJ equipment from Time Group (Beijing, China), coupled with a QK-20 ARMS notching machine. A pendulum of 11 J was used, following ASTM D6110 [20].

The water absorption test was carried out according to the ASTM D570 [21]. Five composite samples of treated and untreated fibers were tested. First, the samples were weighed after drying in an analytical balance, then they were immersed in distilled water at room temperature for 24 h. After that, the samples were dried and weighed again. This process was repeated for 14 consecutive days. To calculate the percentage of water absorbed, Eq. (1) was used, where *wu* represents the weight of the wet sample and *ws* is the weight of the dry sample:

$$\text{Water Absorption} = \frac{w_u - w_s}{w_s} \times 100. \tag{1}$$

Results and Discussion

Characterization of Acetylated Luffa Cylindrica *(FAC) and In-Nature (FIN) Fibers*

The diffractograms for the FAC and FIN samples are displayed in Fig. 2. The FIN sample displayed peaks at 15.6°, 22.3°, and 34.8°, which were previously observed by de Melo et al. [19]. On the other hand, the FAC sample showed peaks at $2\theta = 9.06°$, 17.08°, and 21.18°, indicating a change in the fiber structure after chemical treatment. In a related study, Santos et al. [22] extracted cellulose from a vegetable sponge and then performed acetylation treatment to obtain cellulose acetate. The authors observed peaks at $2\theta = 8.5$, 17.7, and 21°, which were similar to the peaks observed in our study. Upon visual comparison of the diffractograms, we observed that the peaks for the FAC sample were broader, which is characteristic of amorphous materials. This suggests that after the acetylation process, a larger portion of amorphous material was formed, resulting in reduced crystallinity. This change could be attributed to the substitution of hydroxyl groups by acetyl groups, which occupy a larger volume, leading to increased interfibrillar distance and reduced material crystallinity [23].

The literature highlights the 8° peak as a critical feature of acetylated cellulose [24]. Our study reveals that this peak is slightly shifted to $2\theta = 9.06°$, suggesting that the fiber has been acetylated and exhibits characteristics of cellulose acetate.

The FTIR spectra for the FIN and FAC samples are shown in Fig. 3. The FTIR spectra exhibit absorption bands at 1033 cm^{-1}, which are due to C–O stretching in holocellulose and lignin, and at 2890 and 3474 cm^{-1}, which are assigned to the C–H stretching vibration of aliphatic methylene groups and the cellulose O–H vibration, respectively.

The acetylation treatment causes a structural change in the fiber, which is confirmed by the appearance of three new peaks that are characteristic of the acetyl

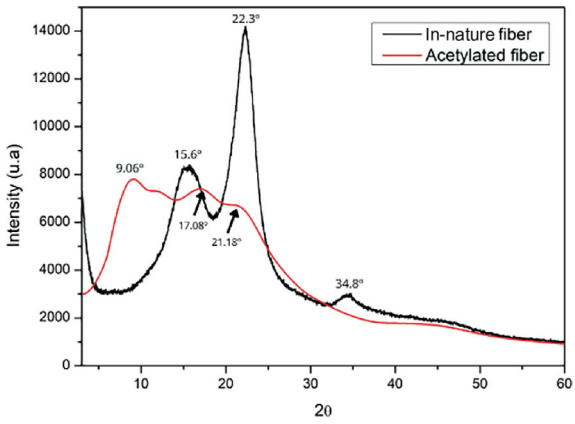

Fig. 2 X-ray diffractograms recorded for the FIN and FAC samples

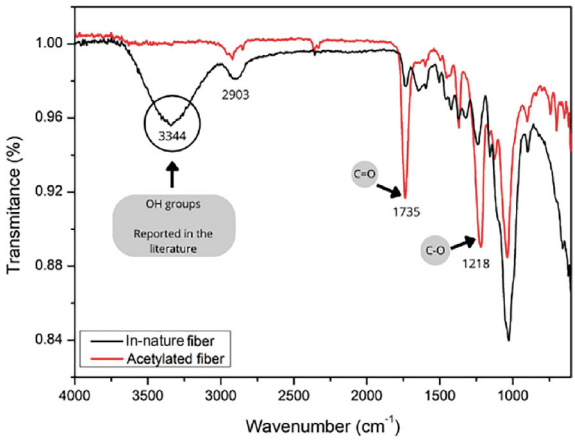

Fig. 3 FTIR spectra of acetylated for the in-nature fiber (FIN) and acetylated fiber (FAC) samples

group. The increased intensity in the bands observed at 1735, 1369, and 1218 cm^{-1} provides strong evidence of the effectiveness of the acetylation treatment. The band at 1735 cm^{-1} corresponds to the stretching of the carbonyl (C=O) bond in the ester group, while the band at 1369 cm^{-1} can be attributed to the stretching of the methyl group in $-OCOCH_3$. The peak at 1218 cm^{-1} is related to the C–O bond of the acetyl group [18, 23, 25, 26]. These peaks are absent in in-nature fibers. Additionally, the absence of peaks between 1840 and 1760 cm^{-1} suggests that there was no unreacted acetic anhydride after acetylation [27–29].

The band around 3400 cm^{-1}, which is characteristic of the hydroxyl groups present in in-nature lignocellulosic fibers, is absent in the FAC sample. The reduction in the intensity of this band was attributed to a partial esterification of the fibers [18, 29]. By analogy, we can assume that the absence of this band indicates complete esterification of the fiber. This may result in a reduction in the hydrophilic nature of the fiber, as indicated by Kabir et al. [30].

The thermal behavior of the *Luffa cylindrica* fiber was studied by TG with consecutive weight losses attributed to the release of water, and to the decomposition of the lignocellulosic matrix. The last stage involves the degradation of lignin, which decomposes at a slower rate, initiating during the second stage and producing ash as residue [31–34].

The weight loss of the FAC sample up to 200 °C (Fig. 4) was found to be only 0.36%, indicating that this sample did not absorb any water from the environment. This suggests that the fiber acetylation process altered its hydrophilicity as confirmed by FTIR (Fig. 3), which showed the absence of the band at 3400 cm^{-1}. Between 200 and 430 °C, the weight loss of the FAC and FIN materials was 68% and 78%, respectively. After complete degradation at 800 °C, the residual weight observed was 26% for FAC and 17% for FIN. These results indicate that the incorporation of acetyl groups in the fiber increased its thermal stability [35].

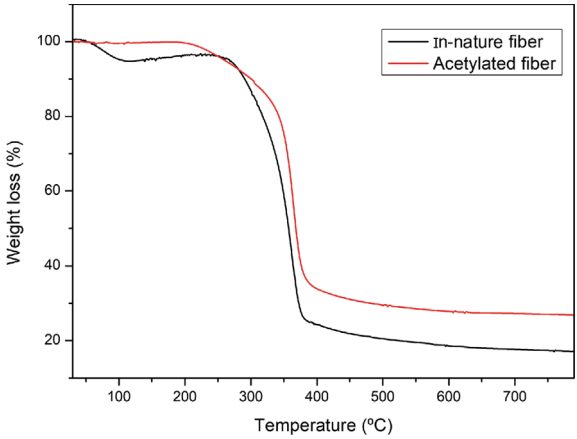

Fig. 4 TG curves for the FIN and acetylated FAC samples

Characterization of Polyester and the Composite Reinforced Within In-Nature Fibers (CFIN) and with Acetylated Fibers (CFAC) of Luffa Cylindrica

Figure 5 presents the spectra of the polyester, the CFIN and CFAC. The spectral bands of the three samples are very similar to each other. This is due to the ATR-FTIR analysis method, which examines the sample surface only, with a depth of penetration ranging from 0.2 to 5 μm. As a result, only the characteristic bands of the polyester could be observed, but with varying intensities. It is suggested that the fiber is positioned deeper within the composites.

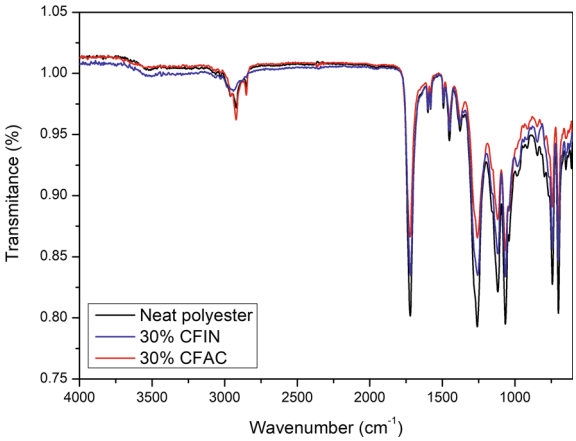

Fig. 5 FTIR spectra of the polyester and composite produced CFIN and CFAC

The interface between the in-nature fiber and the polyester matrix is presented in Fig. 6a. The arrows indicate areas where the fiber and matrix did not bond, revealing weak interfacial adhesion in the composite [37]. Additionally, these defects contribute to the low mechanical strength of the composite, as described in previous studies [6, 38, 39].

After the introduction of acetylated fibers (as seen in Fig. 7b), there are no flaws between the matrix and the fiber, as indicated by the arrows. This absence of imperfections suggests a better interface between the fiber and matrix. This improvement can be attributed to the potential replacement of hydroxyl groups on the cell wall with less polar acetate groups [40].

Figure 7 displays the Charpy impact test results for the pure polyester and the composites reinforced with in-nature and acetylated fibers. The impact strength increased by 1256% to CFIN, while the CFAC composite improved by 1865% compared to pure polyester. These results suggest that when adding a reinforcing

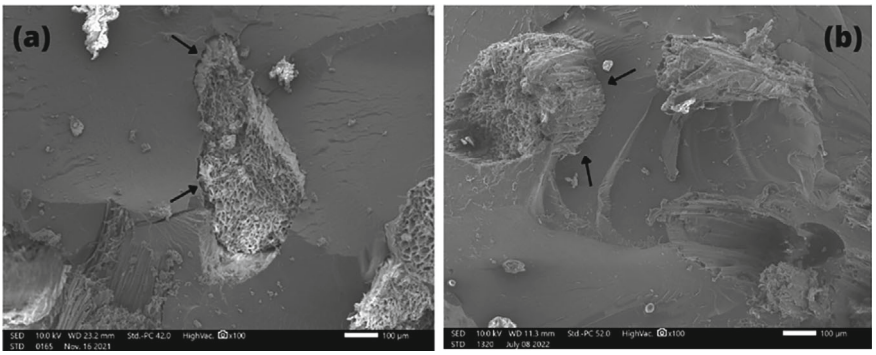

Fig. 6 SEM images for the composites with 30% fiber **a** in-nature and **b** acetylated fiber

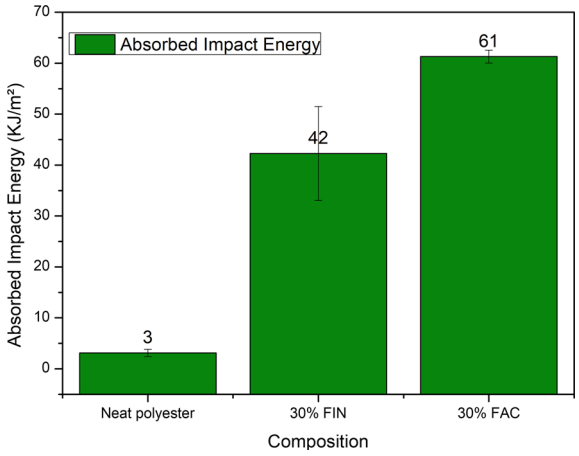

Fig. 7 Charpy impact strength of pure polyester and CFIN and CFAC composites reinforced with 30% fibers

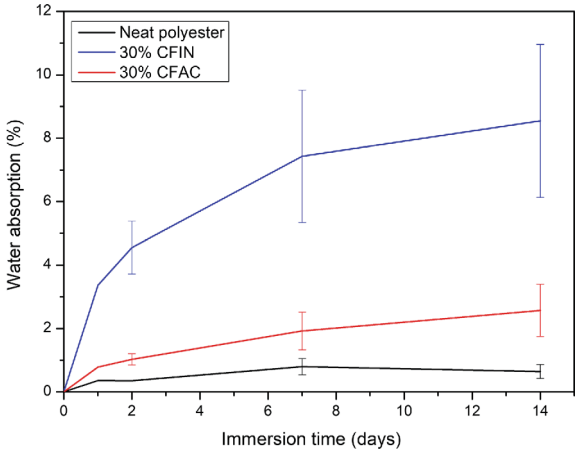

Fig. 8 Water absorption curve as a function of time for pure polyester and CFIN and CFAC composites reinforced with 30% fibers

material more energy is required to break the interaction between the fibers and the matrix once the fibers act as an impact absorption source. The Tukey test results, with a 95% confidence level, revealed that the CFAC composite is statistically different from CFIN. Therefore, it is clear that the acetylation process significantly improves the impact resistance of polyester by promoting the interfacial bond between the fiber and the matrix, as demonstrated by the SEM images in Fig. 6b. Consequently, the CFAC composites are capable of absorbing more energy, preventing crack propagation, and thus increasing impact resistance.

In Fig. 8, we can see the water absorption data over time. The polyester matrix remains stable with only 0.2% water absorption after 14 days, indicating its hydrophobic nature. However, when natural fibers are added to the polyester matrix, the CFIN composite absorbs 8.55% water, suggesting that the lignocellulosic fibers in it are hydrophilic. On the contrary, the CFAC sample shows a remarkable reduction in water absorption, with only 2.54% absorption. This decrease in water absorption is 69% when compared to CFIN, which indicates that the acetylation treatment reduces the hydrophilicity of the LC fiber, making it more hydrophobic.

Conclusions

The incorporation of *Luffa cylindrica* fibers into the polyester matrix has had a pronounced impact on various properties. The acetylation process had remarkable effects on the fibers, notably reducing water absorption and establishing a stronger bond with the matrix. This improved the interfacial adhesion and modified the fiber's hydrophilic nature, resulting in a more hydrophobic behavior. As a result, the performance of the composites significantly improved, particularly in terms of impact resistance and reduced water absorption. The acetylated fibers played a crucial role in enhancing the composite's ability to absorb energy, hindering crack propagation, and

ultimately improving its impact resistance. Therefore, acetylated fiber presents itself as a promising alternative for developing lighter, stronger, and more environmentally responsible materials.

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Development of Anticorrosive Coatings Reinforced with Eucalyptus Residue

D. L. R. Oliveira, D. C. R. Velasco, L. F. F. Freitas, R. R. R. Júnior, C. M. F. Vieria, and F. P. D. Lopes

Abstract Corrosion is a critical problem in industrial environments, resulting in structural and financial damage, especially in sectors such as oil platforms and industrial pipelines. To mitigate this damage, the application of coatings, such as pipe coatings and structural paints, is a common solution. Epoxy resin is often used due to its suitable properties, such as resistance to corrosive media, low cost, and satisfactory strength. However, epoxy resin has the disadvantage of not being recyclable. The aim of this work is to use particulates from eucalyptus waste as a dispersed phase in epoxy resin, with the aim of developing a cheaper and more environmentally friendly material.

Keywords Agro-industrial waste · Epoxy · Composites · Anticorrosive coatings

Introduction

The research and development of sustainable materials has become increasingly evident and widespread in both academia and industry [1]. This is due to the growing need for policies, products, and materials that are more sustainable, motivated by the environmental problems that are becoming increasingly evident [2].

In parallel, some research focuses on developing materials for industrial applications, a practice that has been gaining momentum over the years [3]. One of the largest global industries is oil and gas, which generates billions of dollars annually [4]. However, one of the main challenges faced by this industry is sustainability, which

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creates an urgent need to align this sector with environmental demands [5]. In addition to environmental problems, corrosion represents a major obstacle, generating high costs for companies in this field [6].

Corrosion not only results in significant monetary expenditure, but also in structural and reliability damage [7]. These impacts directly affect production capacity and quality, and can even cause downtime, which leads to considerable financial losses [8]. To mitigate this damage, anticorrosion paints and coatings are widely used in the oil and gas industry [9]. These coatings and paints are usually based on epoxy, a two-component thermosetting polymer which, after curing, forms an ordered structure that is highly resistant to corrosive media [10].

The chemical resistance of epoxy is mainly due to the curing process, which creates long cross chains, making the material durable and resistant [11]. In addition, the low cost of these coatings adds value, further encouraging their use [12].

Eucalyptus, a plant from the Myrtaceae family [13], is the main raw material for obtaining cellulose in Brazil [14]. This tree is widely used in the production of furniture, doors, and windows due to its physical and mechanical properties, which are particularly suitable for these applications [15].

Due to the high production of eucalyptus, it is logical that there is also a large generation of waste. Although some of this waste can be used as compost, the large quantity often results in disposal, often in nature [16]. Even though it is a natural waste, depositing it in large quantities can cause problems for the soil, such as altering the pH, making it difficult for certain types of vegetables and plants to thrive.

Considering these factors, this work aims to develop a pipe coating that is not only ecologically friendly, but also costs less than the products currently available on the market.

Materials and Methods

The materials used in this work were epoxy resin, specifically Diglycidyl Ether Bisphenol A (DGEBA), and the hardener Diethylenetetramine (DETA). These products are marketed under the names SQ2050 and SQ3131, respectively, and were supplied by the company Avipol. Below is an image of the materials mentioned (Fig. 1).

Eucalyptus sawdust obtained from lumber mills was used, which underwent a comminution process to reduce the aspect ratio of the particles. The material was first ground in a knife mill and then milled in a ball mill. After comminution, the material was sieved through a 100 Mesh with an aperture of 0.150 mm. Below is an image of the particulate before and after sieving (Fig. 2).

After this process, the material was placed in an oven at 60 °C for around 24 h to remove the moisture, making it suitable for manufacturing the test specimens. The oven used was the model 3 of the Brasdonto brand. Four different formulations were made, varying according to volume. Below is a table with the respective formulations (Table 1).

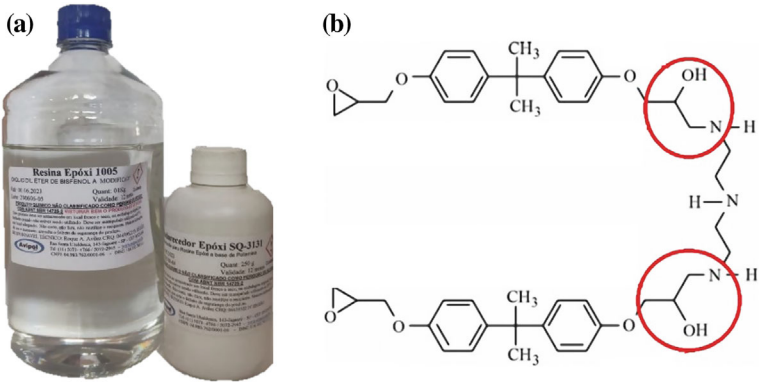

Fig. 1 **a** Epoxy system utilized in this work; **b** how this system is crosslinked

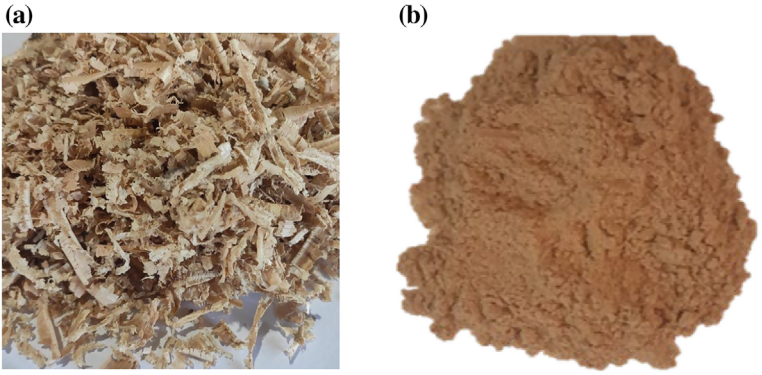

Fig. 2 Eucalyptus sawdust **a** before and **b** after sieving

Table 1 Formulations (volume) produced for this work

ID	Epoxy (%)	Eucalyptus (%)
EP	100	0
5EU	95	5
10EU	90	10
15EU	85	15
20EU	80	20

The specimens were developed in accordance with ASTM D 790-17 [17] and ASTM D 695-15 [18]. For each test and each formulation, around 10 specimens were manufactured and tested. The tests were carried out on an Instron model 5582 universal testing machine at room temperature. For the bending test, the speed rate adopted was 1 mm/min, while the compression test was conducted at

Fig. 3 Universal testing machine INSTRON model 5582. Flexural configuration

a speed of 1.3 mm/min. Below is an image of the universal testing machine with the configuration used for the bending test (Fig. 3).

Results and Discussion

The results obtained from the bending and compression tests will be presented below, as well as some microscopy images.

Compression Resistance

Figure 4 shows the modulus of elasticity and maximum compression strength obtained in the compression tests.

As can be seen in Fig. 4a, the modulus of elasticity of the specimens showed a slight variation when the particulate was applied in lower percentages. However, as the percentage of particulates increased, there was a little tendency to decrease this modulus, considering only the averages. However, when the standard deviations were considered, this change was not significant.

Figure 4b shows that the compressive strength increased slightly with the addition of 5% eucalyptus, but decreased as the percentage of particulates increased. Analyzing the graphs in Fig. 4, it can be seen that as the amount of particulate increased in relation to the matrix, both the strength and the modulus of elasticity

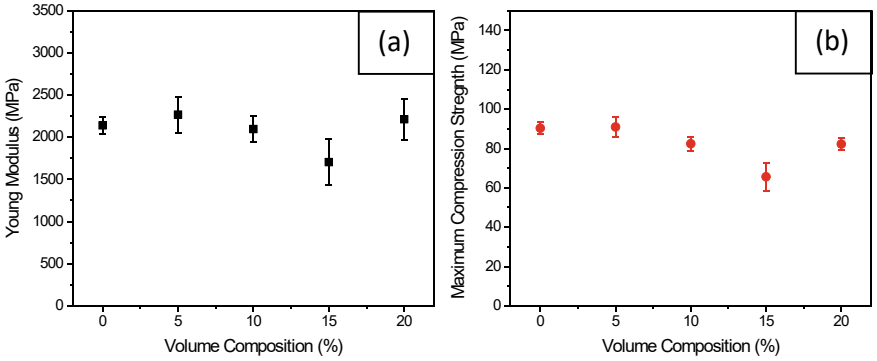

Fig. 4 **a** Young modulus and **b** maximum strength graphs for the compression testing

decreased until reaching a level of 65 MPa, remaining constant up to the maximum volume investigated, which was 20% eucalyptus particulate.

Table 2 provides a comparison of the compressive strength of materials incorporating lignocellulosic powders, sawdust, and fibers.

The table shows a noticeable difference in the compressive strength between the specimens containing eucalyptus powder and those with eucalyptus fiber. This disparity can be attributed to the superior stress distribution provided by the long fibers in the matrix. However, when comparing the other materials analyzed, the variation in compressive strength was not as significant.

Figure 5 shows the fracture surface after compression test, at 100x and 700x magnification. It shows the presence of bubbles, which may have formed during making the specimens or due to the vaporization of trapped epoxy resin volatile components, or even due to possible moisture remaining in the particulates, considering that the curing temperature for epoxy systems can reach up to 120 °C.

The drop in performance can be attributed to the particle's difficulty in adhering properly to the matrix. As shown in Fig. 5, the eucalyptus particles are apparently loose on the fracture surface. In addition, the significant presence of bubbles, which

Table 2 Comparison of results 1 [19–21]

Material	Compression strength (Mpa)	Parameters (mm/min)
Eucalyptus powder	Minimum—90.93	1.3
	Maximum—65.61	
Eucalyptus fiber	Minimum—182.05	1.3
	Maximum—191.42	
Eucalyptus sawdust	Minimum—28.53	1.3
	Maximum—61.71	
Tamarind seed powder	Minimum—40.00	1.3
	Maximum—76.00	

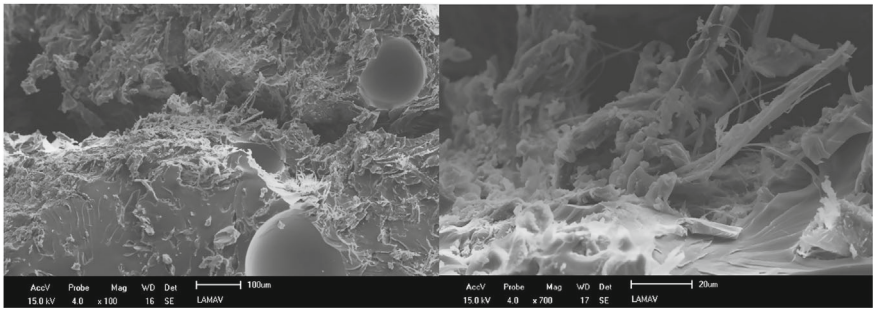

Fig. 5 Scanning electron microscopy of 15% specimens

were more easily incorporated into the specimens with larger amounts of particulate, also contributes to this reduction in performance [22].

Flexural Resistance

Figure 6a shows the Young modulus and Fig. 6b the maximum flexural testing.

The addition of particles resulted in a significant increase in the standard deviation of the modulus of elasticity. This increase can be attributed to differences in the size and morphology of the particles, which affect the displacement of the polymer chains and, consequently, the hardness of the material. The irregular morphology of the particles also hinders workability and can create areas that are not properly wetted, which contributes to the variations observed in the modulus of elasticity. In addition, the particles can influence the curing process of the material, creating areas where curing does not occur uniformly.

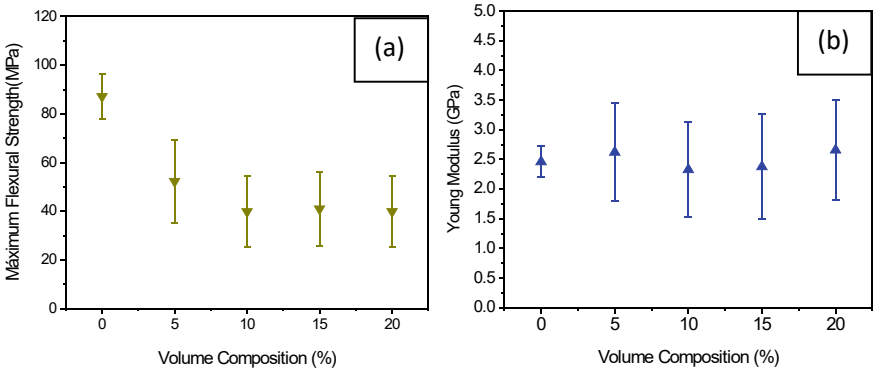

Fig. 6 **a** Young modulus and **b** maximum strength graph for flexural testing

Table 3 Comparison of results 2 [25–27]

Material	Flexural strength (Mpa)	Parameters (mm/min)
Eucalyptus powder	Minimum—40.09	1
	Maximum—53.18	
Coconut shell powder	Minimum—27.17	1
	Maximum—35.48	
Sundi wood dust powder	Minimum—2.61	1
	Maximum—18.58	
Olive pits powder	Minimum—49.97	1
	Maximum—61.12	

The maximum strength graph shows a significant reduction in the strength of the composite with the addition of particles. It can be seen that the formulation with 5% particulates showed a smaller reduction compared to the formulations containing larger quantities of particles. This effect can be attributed to the increase in the resin's viscosity during the curing process, which occurs as the particulate load increases [23]. The higher viscosity made it difficult to eliminate bubbles during the curing of the resin, resulting in the formation of voids in the specimens. These voids can generate significant stress concentrations, especially if they are located in critical regions, such as the center of the specimen. In addition, the morphology of the particles contributes as a stress concentrator [24].

Table 3 presents a comparison of the flexural strength results observed in the Eucalyptus sawdust composite with those of other materials.

It can be seen in the table above that the Eucalyptus particulate obtained a relatively high resistance compared to the other particulates. The comparison materials were chosen with a view to better adapting the test parameter and the matrix used, in order to generate a better comparison effect.

Conclusions

Through this work, it can be seen that the application of the percentages presented does not reinforce, but on the contrary, reduces the strength of the composite. Only the formulation with 5% particulate showed a slight tendency to increase the compressive strength, but the same volumes, that decreased the strength, reduced it to a plateau of around 65 MPa in the compression test and in the bending test to a value of 40 MPa, which still shows an acceptable mechanical strength for various applications, and makes the final composite more eco-friendly, as the amount of final epoxy resin is reduced.

The fractographs show the bubbles created during processing and the low strength of the interfaces between particles and resins, which may be the reason for the

reduction in the compressive and flexural strength of the composites with the increase in the volume of eucalyptus waste particulates incorporated. However, the strengths remain at a pleasant level for possible application as industrial coatings.

Acknowledgements We would like to thank CNPq for the financial support (Process No. 301634/2018-1) and FAPERJ for the support through the project (Ref. Proc. No. E-26/203.874/2022).

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Compressive Strength of Epoxydic Composite Reinforced with Coffee Ground Waste

Bruna Nogueira Simões Cobuci, Mayara Tito Campos, Noan Tonini Simonassi, Sérgio Neves Monteiro, Carlos Mauricio Fontes Vieira, and Felipe Perissé Duarte Lopes

Abstract Coffee, in its liquid form, is consumed globally. Brazil ranks as the second highest consumer, which leads to significant waste. This study aims to evaluate the compressive strength of a composite material made from epoxy resin and coffee grounds for use in High Performance Coating (HPC). The compressive strength of such composites is crucial, as it measures a material's ability to endure compressive forces. Thus, specimens were prepared with coffee grounds at volume fractions of 0, 5, 10, 15, and 20%, and cured at room temperature for 48 h. The testing was conducted according to ASTM D695 standard. The results indicated that coffee waste can effectively be used as reinforcement in polymer composites for HPC applications.

Keywords Compression test · High-performance coating · Coffee grounds

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Introduction

Given the rising concern for sustainability, there is an increasing emphasis on eco-friendly practices, especially in recycling and environmental conservation, which has driven research into polymer composites reinforced with natural materials. As plastic and food waste remain major issues, current investigations focus not only on creating compostable plastics but also on enhancing the value of these waste streams by using discarded agricultural materials, such as lignocellulosic compounds, as biofillers in polymers [1].

The improper disposal of coffee grounds is a common practice that can have significant environmental consequences. When discarded in regular household waste, coffee grounds end up in landfills, where their decomposition releases greenhouse gases such as methane, contributing to global warming [2]. Moreover, when coffee grounds are disposed of through drains and sewers, they can cause blockages and strain wastewater treatment systems, increasing the risk of pollution in rivers and oceans. The pollution resulting from improper coffee ground disposal is concerning, as this practice exacerbates soil and water contamination, harming local flora and fauna [3]. The organic components present in coffee grounds can, when accumulated in water bodies, lead to eutrophication, a process that results in an excessive increase in nutrients, causing algal blooms and subsequently disrupting the aquatic ecosystem [4].

However, coffee grounds hold great potential for beneficial and sustainable reuse. A typical method of reuse involves composting, where the grounds act as a nitrogen-rich component. This helps break down organic matter and results in high-quality natural fertilizer [5].

Research indicates that coffee grounds can be reused to support different industrial sectors, thus reducing pollution and promoting sustainability. Moreover, they are valuable in producing cosmetics, cleaning items, and construction materials due to their abrasive and texture-enhancing qualities and can also be used in manufacturing natural-fiber-strengthened composites [6, 7].

These composites are frequently used in high-performance coatings (HPC), characterized by the presence of epoxy resins combined with different mineral aggregates. This action aims to enhance their physicochemical properties as well as increase their resistance to loads, abrasion, impacts, and other forms of wear, which is essential for applications in civil construction. Previous studies have demonstrated the effectiveness of piassava and coconut natural fibers, in improving the properties of composites. In this context, the present work explores the use of coffee grounds as an aggregate for HPC production. The combination of epoxy resin with natural materials, such as coffee grounds and sugarcane bagasse, not only promotes more efficient waste reuse but also contributes to the development of more sustainable and innovative construction products [8–10].

Therefore, this study aims to evaluate the possibility for reusing coffee ground, a widely daily waste, in the production of innovative epoxy-based composite for high-performance coatings. This combination has the potential to meet rigorous

mechanical demands, such as high compressive strength, essential for applications requiring durability and robustness, such as industrial or infrastructure coatings. Furthermore, using coffee grounds not only helps reduce the environmental impact caused by their improper disposal but also promotes sustainability by assigning economic value to this waste material. Therefore, this study seeks to illustrate a balance between mechanical efficiency and environmental sustainability, aligning with current trends in sustainable construction and circular economy practices.

Materials and Methodology

The composite specimens were produced by combining coffee ground waste, as aggregates, with commercial epoxy resin, the matrix. The coffee grounds (density = 1.45 g/cm^3) were obtained at no cost from a local coffee shop in Campos dos Goytacazes, RJ, Brazil. The epoxy resin DGEBA/DETA (SQ 3131/1005) was purchased from AVIPOL, a distributor of Silaex products in São Paulo, Brazil. The ratio between the components that used specimen predation was corresponded to phr 16.

Prior to preparing samples for mechanical testing, X-ray diffraction (XRD) analysis was performed on the coffee grounds. This examination aimed to explore their crystalline structure and evaluate their adhesion potential to the epoxy matrix. This step is crucial to understand the physical and chemical properties of the coffee grounds, ensuring they can effectively interact and adhere to the polymer, thereby enhancing the composite's efficiency and integrity.

The XDR was carried out at the Advanced Materials Laboratory, Universidade Estadual do Norte Fluminense Darcy Ribeiro (LAMAV/UENF), using a SEIFERT model URD 65 X-ray diffractometer. The scanning method was stepwise with an interval of 0.02° every 2 s with an angular variation of 5º–100º over a range of 2θ.

The preparation of the test specimens involved drying the coffee grounds in a laboratory oven for 48 h. The dried grounds were milled for 12 h with a ball mill (Servitech model C7-240 VA, located at LAMAV/UENF) to achieve finer particle size. Subsequently, they were sieved through a 100 mesh sieve, which has an aperture of 0.149 mm. The test specimens were then cured at room temperature (25 °C) for 5 days.

The compression test specimens were prepared with coffee grounds volume fractions ranging from 0 to 30%, increasing in 5% increments. Six specimens were produced for each proportion (Fig. 1). An open silicone mold with dimensions of approximately 13 × 13 × 6 mm was used. Before testing, the specimens were sanded to remove any burrs and to ensure they conformed to the dimensions specified by the standard. Finally, the samples were subjected to compression testing using the universal testing machine Instron, model 5582 (LAMAV/UENF), in accordance with ASTM D695/2015 [11]. The test was conducted at a speed of 1.3 mm/min, at room temperature (25 °C).

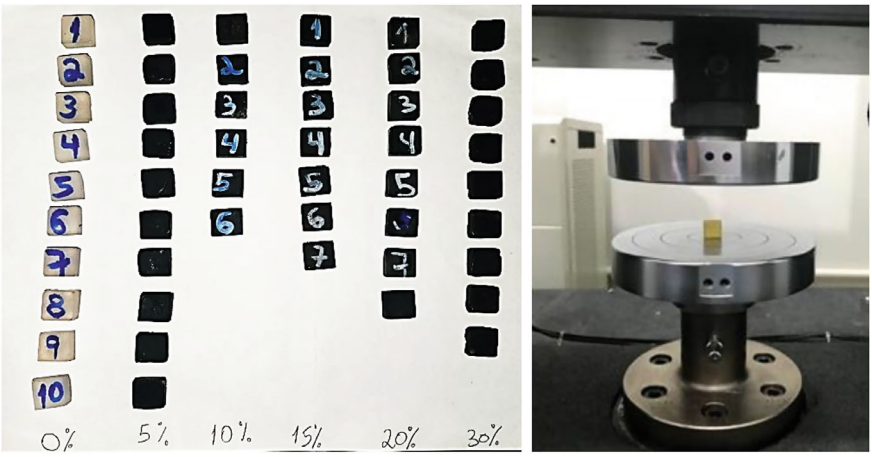

Fig. 1 **a** Specimens; **b** compression test setup

Results and Discussion

The X-ray diffraction (XRD) of the coffee grounds is presented in Fig. 2.

To compare the crystallinity of the coffee grounds in this study, we used a diffractogram from prior research conducted in Portugal as a reference [12]. Figure 2 reveals peaks corresponding to the crystalline planes of lignocellulosic materials. The diffraction at peak A ($2\theta = 16.5°$) indicates the presence of crystalline cellulose, representing a well-organized portion of the material. On the other hand, peak B (located at 2θ

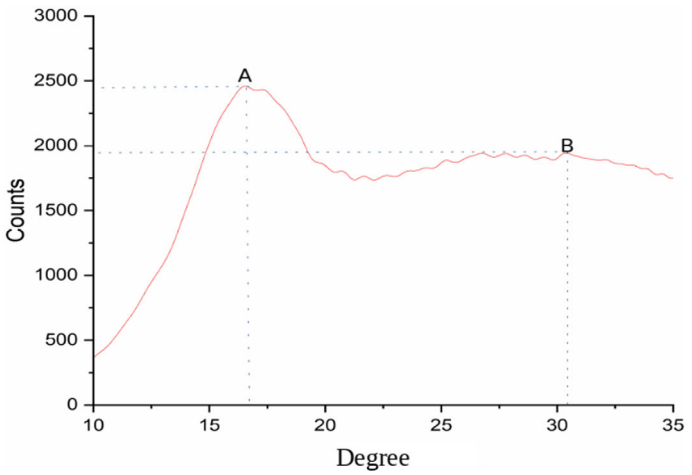

Fig. 2 X-ray diffratogram of the coffee grounds waste

Table 1 Compressive strength of composite reinforced with coffee grounds

Coffee ground volume (%)	Compressive strength (MPa)
0	55.30 ± 2.46
5	37.69 ± 2.82
10	38.83 ± 1.75
15	40.11 ± 5.03
20	47.35 ± 2.28
30	22.44 ± 1.86

= 30.5°) indicates the presence of amorphous regions within the material, which are composed of various other substances including hemicellulose, lignin, pectin, and amorphous cellulose. These findings offer an understanding of the structure of coffee grounds, which can affect properties such as their adhesion capacity to epoxy resin [12].

Table 1 and Fig. 3 illustrate the compressive strength in MPa as a function of the percentage of coffee grounds added to the epoxy resin.

Table 1 presents the compressive strength results for each composite, along with their respective standard deviations.

Figure 3 shows the compressive strength results as coffee grounds were added up to 30% in the epoxy resin matrix. The sample containing 0% of coffee grounds was used as a reference. This analysis reveals the relevance of incorporating waste materials to achieve greater strength capacity.

It is observed that the addition of coffee grounds resulted in a decrease in compressive strength; however, as the amount of coffee grounds increased, there was also a tendency for strength to rise up to 20%. Adding 30% coffee grounds was ineffective. According to [13, 14], incorporating 30% coffee grounds can greatly decrease

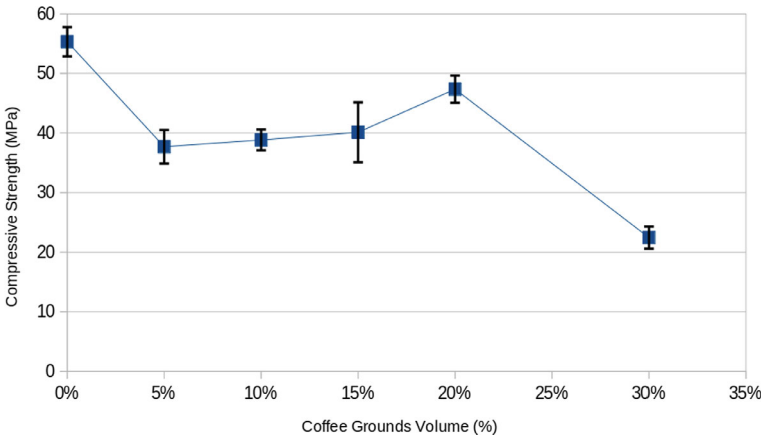

Fig. 3 Compressive strength of coffee ground composite at different proportions

wettability with the epoxy matrix, negatively impacting the composite's mechanical properties. The samples at each proportion exhibit a small standard deviation, indicating strong interaction between the matrix phase and the dispersed phase.

The reduced mechanical strength of coffee-ground-based composites, compared to the reference sample, can be explained by the addition of particulates into thermoset polymer matrices. This process introduces defects in the final material, including bubbles, interfaces, and microcracks. If the strength is maintained at an acceptable level for the proposed composite, in this instance, HPC, it is highly advantageous because it decreases the use of synthetic resins sourced from non-renewable materials.

When examining the maximum deformation, it is clear that there is a significant increase in the capacity to absorb impact loads, which is a property required by the standard NBR 14,050, governing HPCs.

NBR 14,050 states that the minimum compressive strength for HPCs is 45 MPa [8]. Thus, the optimal formulation for polymeric matrix HPC reinforced with coffee grounds is 20% coffee grounds. Nonetheless, the outcomes are still in an initial stage due to the extensive potential for additional investigation.

Conclusion

Epoxy resin mixed with coffee grounds is an excellent choice for High-Performance Coatings (HPC) due to its crystalline structure. The findings showed that the mechanical strength was satisfactory and met the ASTM D 695 and NBR 14,050 standards. Testing revealed that adding coffee grounds into the epoxy resin matrix enhanced compressive strength and durability in certain instances, which are crucial for high-performance coatings. These characteristics are essential for guaranteeing the durability and performance of coatings in harsh conditions.

Additionally, using coffee grounds as filler in composites offers technical advantages and promotes environmental sustainability. Using organic waste in construction materials supports a circular economy and cuts down on landfill waste, aligning with sustainable development goals.

The positive results demonstrated by this composite also indicate a considerable potential for its use in sectors beyond high-performance coatings, such as construction, where the demand for sustainable building materials is growing. This versatility makes the epoxy resin composite with coffee grounds a relevant and sustainable alternative to conventional materials that have greater environmental impact.

This research demonstrates the composite's effectiveness for use in High-Performance Coatings and paves the way for new innovations in sustainable materials, emphasizing its important role in the industry and its support for more environmentally friendly practices.

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Part III
Poster Session

3D Printing in Additive Manufacturing in Stereolithography: Coconut Shell Powder Additive

Victor Paes Dias Gonçalves, David Coverdale Rangel Velasco, Henry A. Colorado, Carlos Maurício Fontes Vieira, and Felipe Lopes Perisse

Abstract This study investigates the use of coconut shell powder as an additive in photopolymerizable acrylic resins used in 3D printing via stereolithography (SLA). The primary objective is to evaluate the influence of this additive on the mechanical properties of the resin, focusing on impact resistance, compression, flexural strength, tensile strength, and hardness. Resin samples containing 1% processed and unprocessed coconut shell powder were compared to a control sample without additives. The results indicate that the addition of processed coconut powder enhances the mechanical properties of the resin compared to both unprocessed coconut powder and the control, particularly in terms of impact resistance and hardness. Microstructural analysis using scanning electron microscopy (SEM) revealed that the processed fibers act as physical barriers, delaying crack propagation and thereby increasing the material's durability. This study demonstrates the potential of coconut shell powder as a sustainable and effective additive in resins for 3D printing, promoting sustainability in additive manufacturing processes.

Keywords Additive manufacturing · SLA · Coconut shell powder · Mechanical property

Introduction

Liquid-based additive manufacturing (AM) methods with vat-based printing, such as Stereolithography (SLA) and Digital Light Processing (DLP), were introduced in the 1980s with the aim of creating 3D objects by curing a photopolymerizable

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polymer under ultraviolet light [1, 2]. SLA is an AM process in which an object is created from a three-dimensional (3D) model, layer by layer. Both SLA and other AM processes rely on three different stages: data acquisition, data processing, and object fabrication [3, 4].

The SLA printer, an early adopted vat-based AM technique, operates in the 3D printing process using photopolymerization. Photopolymerization refers to a technique that uses light rays to propagate a chain polymerization process resulting in the photo-crosslinking of pre-existing macromolecules [5, 6].

SLA resin exhibits excellent layer-to-layer bonding, resulting in high printing quality, strength, dimensional stability, and low viscosity, which facilitates equipment cleaning. These resins incorporate hybrid technology, combining acrylates and epoxies. They possess suitable properties such as hardness, abrasion resistance, solubility, water absorption, and dimensional stability. Various types of resins are commercially available today, with different reinforcement materials [3, 7, 8].

One of the reinforcing possibilities highlighted in the literature is the use of natural fibers. The use of natural fibers as an alternative to synthetic fibers for reinforcing various types of composites, especially polymeric composites, has been showing increasing rates [9, 10]. This is due to the unique characteristics of natural fibers, such as abundance, biodegradability, low density, non-toxicity, lower abrasiveness, useful mechanical properties, and low cost [11].

Fibers can be classified based on their origin, including Seed fibers (e.g., cotton), Stem fibers (e.g., banana, jute, flax, hemp), Leaf fibers (e.g., sisal, piassava), Fruit fibers (e.g., coconut), and Root fibers (e.g., zacatão).

The use of plant fibers, such as cotton, jute, sisal, and coconut, is frequently employed as reinforcement in composite materials due to their various beneficial properties, including low density, reduced abrasion during processing, high filler levels that enhance material stiffness, and improved durability. Additionally, their biodegradability and renewable sourcing contribute to their low cost [12, 13].

The utilization of raw materials from renewable sources such as curaua, coconut, sisal, ramie, sugarcane bagasse, jute, and pineapple as reinforcements in polymeric materials has garnered significant interest in scientific research and studies due to their potential to replace synthetic products. The prospects for natural fiber applications are substantial in areas such as the automotive industry, textile industry, internal lining of trucks, buses, and cars, and construction [14, 15].

Fibers can impart unique properties and play a role in tackling environmental issues, such as decreasing the decomposition time of plastics [16]. Furthermore, the improper disposal of fibers in the environment can lead to public health problems, including proliferation of vectors, uncontrolled reproduction of harmful animals to human health, occupation of large areas in landfills, gas production, and soil contamination [17–19].

Thus, the article aimed to evaluate the influence of coconut fiber on UV-photopolymerizable resin—SLA.

Table 1 Characterization of materials, methods, and standards (e.g., American Society for Testing and Materials, ASTM)

Test	Machine	Standard	Parameters
Compression	Universal Tensile Tester, Instron 5582	ASTM D645 [20]	Test speed: 4 mm/min
Impact (Izod)	Impact Energy: 22 J Impact Velocity: 3.5 m/s	ASTM D256-10 [21]	Pendulum Impact tester, Pantec XC-50
Tensile Test	Universal Tensile Tester, Instron 5582	ASTM D638 [22]	Test speed: 500 mm/min
Shore D Hardness	Shore D Hardness tester	ASTM D2240 [23]	Five indentations in an "X" format were performed, with a minimum distance of 12 mm from the edges and 6 mm between the test points
Characterization of the Microstructure	JEOL JSM 6700R	–	Scanning electron microscopy (SEM)

Materials and Methodology

Material Selection and Sample Preparation

For this study, the materials selected for the application in intraoral devices were Dental Resin (3DCURE, São Paulo-SP, Brazil) processed via SLA.

Samples for each test were designed using Autocad Fusion software following the standards presented in Table 1. The designs were saved in STL file format. The designs were then imported into Chitubox Software, where supports were also designed and saved in a format compatible with the printer.

Using an Anycubic 4 k Photon Mono printer (Anycubic, Shenzhen, China), the samples were printed—the first 10 layers of printing (constituting the final bottom layer) had the layer height adjusted to 0.05 mm, exposure time set to 4.5 s, base layer exposure time to 70 s, and retraction distance to 6 mm.

After printing, the samples were released and placed in a 10-min bath of 99% isopropyl alcohol using the Wash&Cure 2.0 (Anycubic, Shenzhen, China) and subjected to a 10-min UV curing process with a light source of 405 nm wavelength using the same device.

Mechanical Characterization

To gain a deeper understanding of the materials, studies were conducted on mechanical, durability, physical, and thermal characteristics. The Compression, Izod Impact,

Tensile, and Shore Hardness tests followed the ASTM standards referenced in Table 1.

Reinforcement Detailing

The unprocessed coconut fiber was simply sieved to remove larger particles, while the processed fibers were refined using a sieve with a 0.15 mm opening and a 100 mesh. The processed material, before sieving, was ground for 24 h in a ball mill, obtained from Servitech, model CT-240/A. This implies that the two materials used in this work, despite being passed through the same sieve, present a significant difference in average size and morphology.

Results

The compressive strength and deformation characteristics of the control group samples, along with those reinforced with processed and unprocessed coconut fibers, were analyzed (Fig. 1a, b). The control samples demonstrated a compressive strength of 106.6 MPa (±5.4) with a maximum deformation of 41.70% (±1.20%).

In comparison, the sample reinforced with processed coconut fiber exhibited a compressive strength of 98.32 MPa (±4.5) and a maximum deformation of 39.50% (±1.44%). The sample reinforced with unprocessed coconut fiber showed results of 109.5 MPa (±5.8) and 43.10% (±1.12%), respectively.

These findings suggest that, although the inclusion of unprocessed coconut reinforcement did not result in a significant difference in the material's compressive strength, it did lead to a noticeable variation in maximum deformation capacity.

The Shore D hardness of the samples was evaluated (Fig. 2), with a focus on the control group and the groups reinforced with coconut fibers. The control group

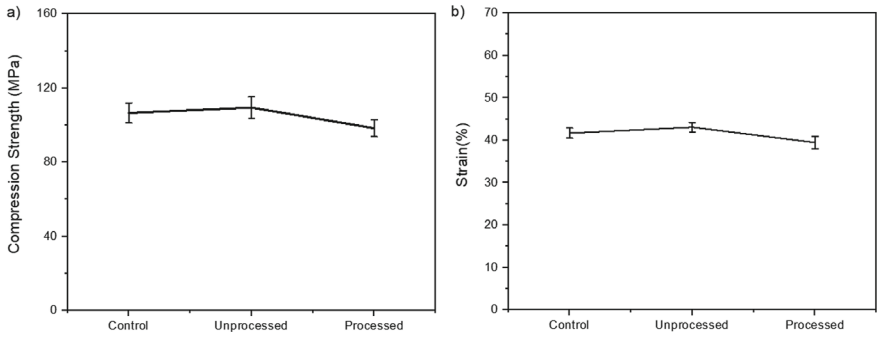

Fig. 1 Compression properties: **a** strength and **b** strain

exhibited a hardness of 83.2 (±0.4). For the samples reinforced with coconut fibers, the results varied depending on the particle size used.

The sample reinforced with unprocessed coconut showed a Shore D hardness of 80.3 (±1.33), slightly lower than that of the control group. In contrast, the sample reinforced with processed coconut exhibited a Shore D hardness of 84.0 (±1.1), slightly higher than that of the control group.

The control group exhibited a notch resistance (Fig. 3a) of 20.1 J/m (±2.6). The reinforcement with coconut fibers showed variations depending on the particle size. The sample reinforced with unprocessed particles displayed a notch resistance of 17.3 J/m (±2.2), which is lower than the control group.

On the other hand, the sample with processed particles demonstrated a notch resistance of 24.2 J/m (±2.4), which is higher than the control. For Izod impact resistance (Fig. 3b), the control group recorded a value of 1.7 kJ/m^2 (±0.6). The

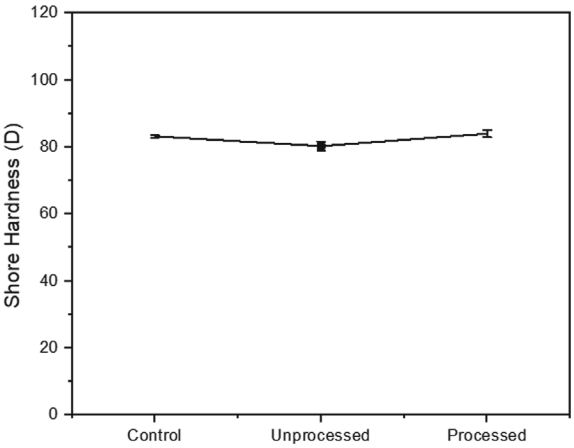

Fig. 2 Results Shore D samples

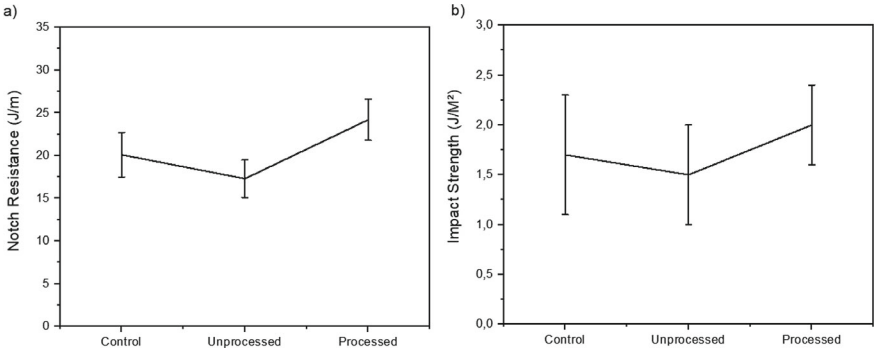

Fig. 3 Izod impact resistance properties: **a** notch and **b** impact strength

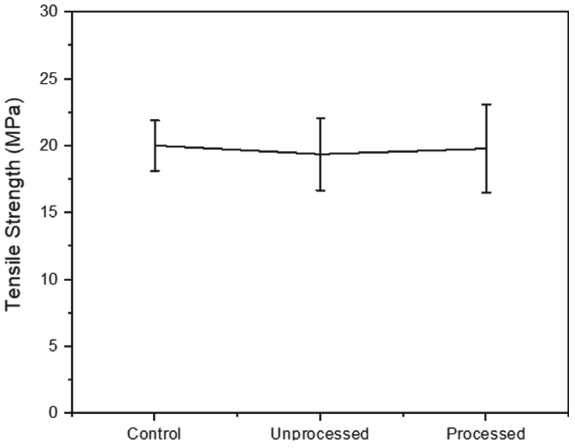

Fig. 4 Tensile properties: tensile strength

sample with unprocessed particles showed an impact resistance of 1.5 kJ/m^2 ($\pm$0.5), which is lower than the control. In contrast, the sample with processed particles exhibited an impact resistance of 2.0 kJ/m^2 ($\pm$0.4) significantly higher than the control value.

Regarding the Tensile Strength test (Fig. 4), the control group exhibited a strength of 20.03 MPa ($\pm$1.9). The unprocessed sample showed a result of 19.38 MPa ($\pm$2.7), while the processed sample showed a result of 19.8 MPa ($\pm$3.3). These results suggest that both processing and the absence of processing did not lead to significant changes in tensile strength compared to the control group.

Upon analyzing the SEM of the control resin (Fig. 5a) after being subjected to a flexural test, it exhibits a brittle fracture with beach marks. The micrograph was captured at a high magnification of 250x, allowing for a detailed view of the microstructures on the fracture surface, with the presence of microplastic deformations in the polymer matrix.

The image obtained with a 300 $\times$ magnification reveals details on the surface of the resin, particularly around the inclusion of unprocessed coconut fiber (Fig. 5b). The micrograph shows the incorporation of the fiber into the resin matrix, highlighting it as an irregularity on the surface that causes deformations in the surrounding material. A visible crack starts at the edge of the coconut fiber, suggesting that its presence influences the mechanical behavior of the resin, especially concerning fracture resistance.

The resin surface around the fiber is relatively smooth but shows fine lines of cracking and small deformations, indicating that the coconut fibers might play a significant role in fracture propagation. According to the micrographs, the fibers are well embedded in the matrix in various areas, suggesting that they act as physical barriers, deflecting or slowing down crack propagation, which improves the impact resistance and durability of the material.

The analysis of SEM micrographs of samples with processed coconut fibers (Fig. 5c) shows their uniform surfaces and anchoring within the resin matrix, playing

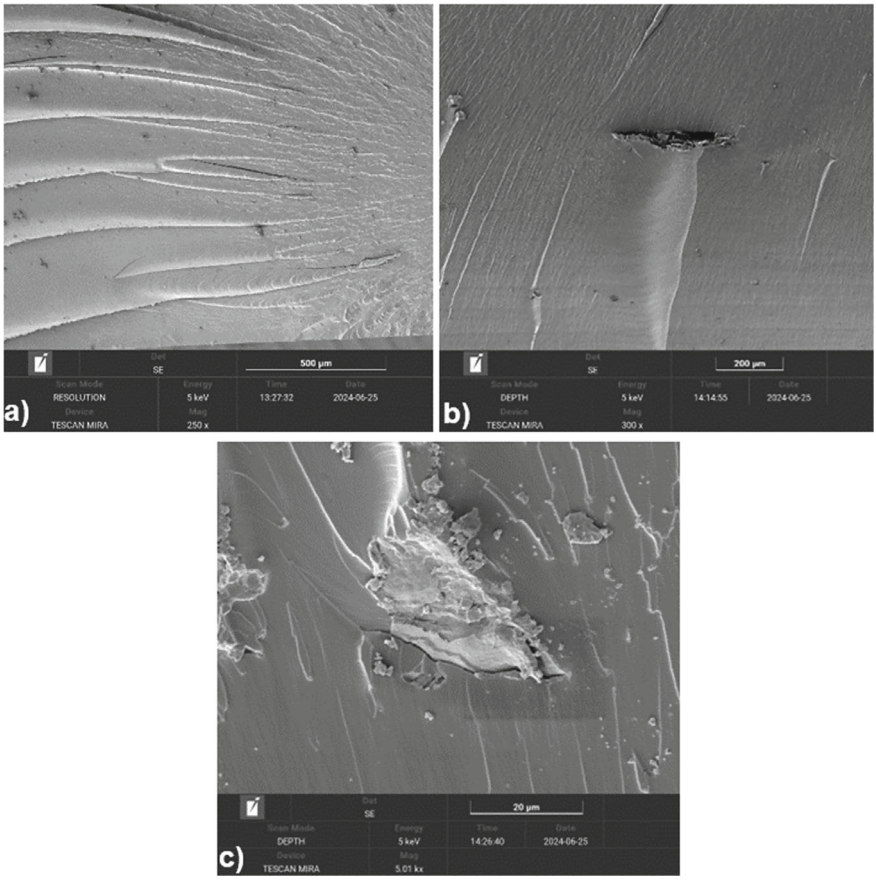

Fig. 5 SEM micrographs of fractured surface for samples at magnifications of **a** Sample Resin without reinforcement; **b** Sample Resin with reinforcement unprocessed; **c** Sample Resin with reinforcement unprocessed

a crucial role in dissipating fracture energy. The micrographs reveal that cracks, when present, tend to be interrupted or diverted upon encountering processed coconut fiber. This behavior suggests that processed fibers act as effective barriers against crack propagation, a phenomenon essential for improving the material's impact resistance.

The fractures occurring in the resin matrix are predominantly brittle, characterized by rapid crack propagation. However, the inclusion of processed coconut fibers alters this dynamic, promoting a more controlled fracture and delaying crack propagation through the structure.

The impact of processed coconut fibers on crack dissipation is significant. Due to their better adhesion to the matrix, the fibers prevent cracks from propagating linearly, forcing them to deviate or stop. This deviation in the crack's path results in more efficient energy dissipation, enhancing the toughness of the composite material.

Additionally, the improved interface between the fiber and the matrix reduces the formation of micro-cracks, which are points of crack initiation, further contributing to the material's durability and structural integrity. These results indicate that processing the fibers is an effective strategy to optimize the mechanical properties of the material, making it more suitable for applications where impact resistance and durability are critical.

As future works, coconut husk of diverse granulometries and loading contents can be fabricated [24] aiming energy dissipation applications, or even using other additive manufacturing technologies to produce coconut husk cement/ceramics matrix composites [25]. The results from this research enable the possibility of using these natural fiber printed composites in armor [26] and even in dental applications [27].

Conclusions

The study concludes that coconut shell powder, especially when processed, is a promising additive for photopolymerizable resins used in SLA 3D printing.

The inclusion of this material not only improves the mechanical properties of the resin, but also contributes to environmental sustainability by promoting the use of agricultural waste in the production of advanced materials.

These findings suggest that processed coconut shell powder can be a viable alternative to synthetic additives, expanding the use of sustainable materials in the additive manufacturing industry.

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Characterization of Novel Ecofriendly Polymeric Composites Based in Montmorillonite Clay

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Abstract The demand for new eco-friendly materials is generating a race to discover good alternatives to replace the synthetic materials used commercially at this time. Raw materials from plant resources are becoming a good option in this regard, especially natural fibers that are usually discarded in landfills. The purpose of this work is to develop a new environmentally friendly polymer composite based on montmorillonite clay powder. Clays (mainly montmorillonite-rich, in situ weathered volcanic rocks—bentonites) are considered the most suitable material for the external multibarrier system. The main objective of this work is to thermally and mechanically characterize the composites made with a common epoxy resin (DGEBA/DETA) and the ground montmorillonite clay, whose incorporation was in volume from 5 to 30%, made in an open silicone mold. The results show good thermal stability and satisfactory mechanical properties for engineering applications, e.g. rigid casing structure and piping in industry with very low cost.

Keywords Montmorillonite clay · Epoxy Resin · Characterization · Eco-friendly materials

Introduction

In Brazil, we found a giant variabilite of ecosystems spread across its territory, resulting in an extremely abundant supply of natural products. The presence of different types of minerals, such as clay, has also gained visibility in studies beyond

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their common use, including in the field of aesthetics. In this context, we have montmorillonite clay, a mineral abundantly found in nature, which has shown promising results in polymer composites due to its properties [1–3].

Aligned with the idea of greener development, there has been growing interest in less "synthetic" modernization, with reduced environmental impact and utilizing natural resources for research in engineering. In this scenario, many researchers in the field of composites are using natural fiber particulates as reinforcement for biodegradable alternatives [4, 5].

Plant-based natural fibers (lignocellulosic fibers) have the advantage of offering a vast variety of species for research, being produced directly in nature or indirectly as waste mainly from the agroindustry. This use reduces CO_2 emissions into the atmosphere and enhances economic potential, benefiting both the environment and engineering [1].

Brazil stands out globally in the production of composites reinforced with natural fibers, especially in the automotive sector, and is expanding with the support of national companies seeking sustainability, particularly in the WPC sector. With greater dissemination of the benefits of natural fibers, research and scientific publications on them will increase, enabling new products from biomass and contributing to the environment [6].

Future work with lignocellulosic fibers should explore the advantages of these materials, such as the crystalline arrangement of cellulose microfibrils. Their isolated application generates countless possibilities due to the chemical differences and structure of the main components of this type of fiber, as well as its wide variety [7].

Similar to natural fibers, we also find "particulate," which appears in various forms in nature. Unlike fibers, particulates are better utilized after being ground [8].

With sustainability in mind, this study will focus on using montmorillonite clay powder, aiming to generate a composite resistant to mechanical tests, such as compression, which could potentially become a material for anticorrosive reinforcement or in civil construction [9].

Materials and Methodology

Materials

The materials used in this research were the polymeric resin DGEBA/TETA—Diglycidyl Ether of Bisphenol A/Triethylenetetramine and the powder of montmorillonite clay, as its shown in Fig. 1. The mentioned epoxy system, marketed by the company Avipol, is commercially known as SQ2050/SQ3131.

Fig. 1 Montmorillonite clay

Methods

The clay is commonly sold in powder form, and its granulometry is already accurate in the laboratory (0.149 mm, corresponding to a 100 mesh sieve). Before being incorporated into the epoxy matrix, its density is obtained using a pycnometer to adjust the correct proportions for the preparation of test specimens, based on ISSO 1133 and following this equation (Fig. 2):

$$\frac{B-A}{D-A} - (C-B)$$

A: Mass of the empty pycnometer.
B: Mass of the pycnometer with the substance.
C: Mass of the pycnometer with the substance and water.
D: Mass of the pycnometer filled with water.

Prior to mixing with the epoxy system, the clay powder is placed in an oven at 60 °C for approximately 12 h to ensure maximum moisture absorption. A total of 48 samples were prepared, 8 for each composition, with a mixing process of

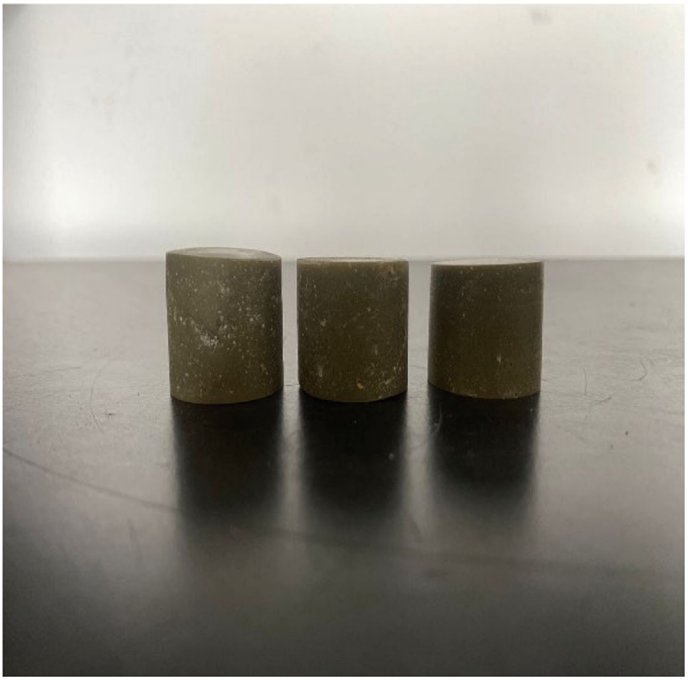

Fig. 2 Formula of density by the use of pycnometer and water

approximately 1 h before being poured into the open mold, allowing the particles to better adhere to the resin.

The compression test was conducted using an INSTRON universal testing machine, model 5582, available at the Advanced Materials Laboratory (LAMAV) at UENF, at a speed of 1.3 mm/min and at room temperature, according to ASTM D6641-16 [10].

Results

Approximately 6 samples were made for each composition. After seven days of curing the resin, the specimens were properly sanded and sent for testing. The Fig. 3 represent a sample ready to be tested.

The density found for this clay was 2.35 g/cm^3, and it is directly associated with the composition with the natural reinforcement/polymeric resin/hardener before it went to the open molds, as shown in Fig. 2.

The results obtained (Fig. 4) in the compressive test of montmorillonite powder were satisfactory with 5 vol% of its composition starting to fall with 10 vol% until reaching the minimum on 25 vol%, and then increasing again on 30 vol%, but all

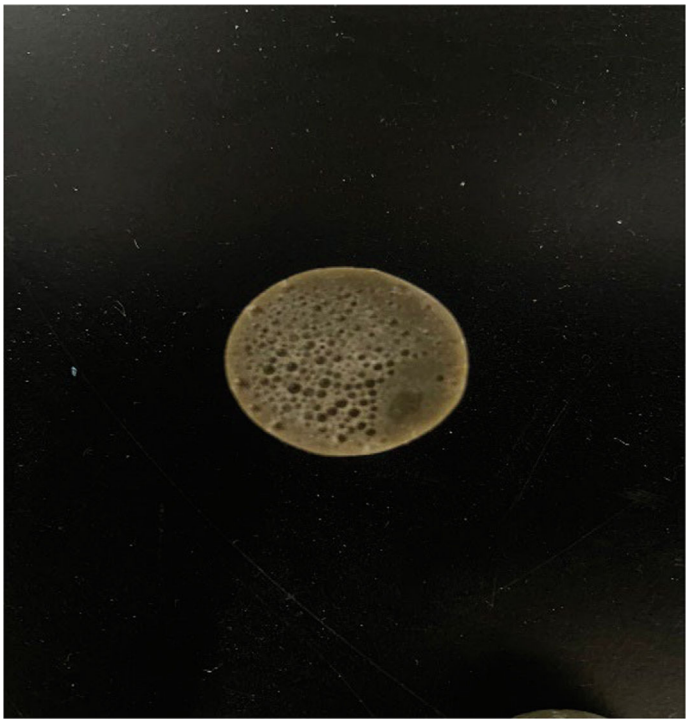

Fig. 3 Samples of montmorillonite clay's compressive test

percentages proposed do not fall enough to discard completely, and still have strength to be use in other applications. After all the samples tested, its obtained this result (Figs. 3 and 5).

In applications where we do not need much strength, it is possible to use all the formulations proposed, for example in esthetic coating in facades, reducing the amount of synthetic resins commonly used for this.

Fig. 4 Montmorillonite clay composition

ID	RESIN	CASUARINA
EP	100%	0%
5MontC	95%	5%
10MontC	90%	10%
15MontC	85%	15%
20MontC	80%	20%
25MontC	75%	25%
30MontC	70%	30%

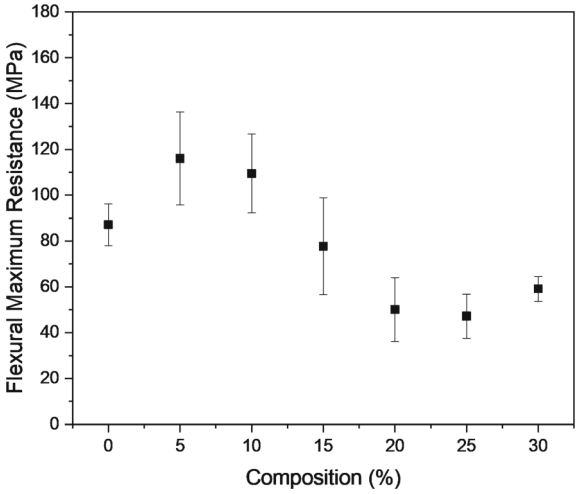

Fig. 5 Compressive strength for the composites proposed

Conclusions

The results obtained in the compressive test of casuarina powder were satisfactory with 5% of its composition, starting to fall with 10%. This can be due to numerous factors, such as increased appearance of bubbles and an unsatisfactory mixture of the particulate with the polymeric matrix (epoxy resin), since the increase in composition generates greater difficulty in mixing. Casuarina powder has good results in tension, compression, and flexural tests when compared to other woods, which makes it a promising product in the area of natural composites.

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Characterization of Composite Polymers Reinforced with Coconut Sheath Fibers

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Abstract In recent years, scientists have been studying materials that aim to replace synthetic fibers. Their main bets are on natural fibers, as they have several benefits, such as having low density and being biodegradable. This study is based on coconut sheath fibers, used as polymer reinforcement. This research was carried out using fiber, extracted from the coconut tree, and 5 test bodies were made, with 4, 5, and 6 layers. Their results indicate that the specimen made with 6 layers of coconut sheath fiber has better flexural resistance.

Keywords Sustainability · Polymer composites · Coconut fabric fiber · Mechanical test · Flexural testing

Introduction

Natural fibers offer a great diversity of fibers, and with the growth of their uses, the need to use them in engineering has arisen. [1]. Scientists are studying sustainable materials to replace synthetic fibers. The use of natural fibers (NF) has been studied with applications in polymer composites, and considered using it as reinforcement. They are found in nature as seeds, fruits, leaves, and stems. Some examples are cotton, coconut, pineapple, flax, bamboo, etc. [2]. They are considered renewable fibers since they are biodegradable [3].

Due to the great environmental degradation of the last century, caused by unbridled industrialization, alternatives are being studied to deal with the consequences. Currently, with environmental laws, researchers are looking for sustainable materials for their applications. Plant fibers as a substitute for synthetic materials are a possibility [4]. In recent years, the use of natural fibers has shown technological advances, seeking to be more widely used, thus being rapidly studied [5].

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Because they are fibers found "ready-made" in nature, some have shapes and dimensions that cannot be influenced [2]. To determine the mechanical characteristics of natural fibers, their length and orientation are important. Still, each fiber has structural properties that must be considered [4]. The cost–benefit of using natural fibers is noteworthy, as they are low-density fibers, abundant in nature and inexpensive.

The NF used in this work is as a polymeric reinforcement, and its matrix material is epoxy resin. *Cocos nuciferas*, known as the coconut palm, is abundant in Brazil. The fiber used in this work is the natural tissue found in the coconut palm sheath, between the petiole and the trunk of the tree [4] (Figs. 1 and 2) to show its mechanical flexural strength characteristics. This fiber is composed of a core with a larger diameter and intertwined with an outer fiber [6].

Previous work on the characteristics of other parts of the coconut, such as leaves or the husk, is common. The use of the coconut fabric is a recent area of research, and there are few auxiliary articles. The aim is to show properties based on mechanical tests. The studies found are more common fibers that have a random matrix. The coconut sheath fiber has a natural formation that partially determines the results of the test. The positioning of the fiber characterizes the result in the future. Thus, there are sides with greater and lesser mechanical resistance. The specimens were made with the fiber seeking the highest strength, i.e. using all the fabrics used for the best result [7].

Fig. 1 Location of the sheath on the coconut palm and the coconut sheath tissue

Fig. 2 Matrix under pressure during the making of the plates

Materials and Methods

Materials

Basically, the material used was the tissue found between the petiole and the trunk of the coconut tree. The coconut plantation is located in the 12th district of Campos dos Goytacazes, Morro do Coco, where the producers donated the coconut sheath fiber. They were extracted by hand and sent without any processing.

The epoxy resin Diglycidyl Ether of Bisphenol A (DGEBA) was used to make the specimens—in a mixture with diethylene tetramine hardener (DETA), both from Avipol and manufactured by Silaex.

Methods

To start making the specimens, the fabric was cut using the same measurements as the matrix in which it would be used in the future. After cutting, they were washed in running water (to remove any impurities found in the fabric) and then taken to an oven to dry at 60 degrees for 24 h.

To make the specimens, a closed mold was used; each part of the mold to be used was checked for cleanliness, followed using a release agent in each part of the mold. This was followed by a layer of plastic film on the bottom and top of the mold, and then another use of the same, for better removal of the specimen. A carbon steel plate was used and placed in the closed die to reduce the thickness of the plate and determine the thickness of the specimen. This plate was coated as before, with a release agent-plastic film-release agent. Only the release agent was used in the middle part of the matrix. For production, a mixture of 124.4 ml of resin and 19.9 ml of hardener was used. It was mixed for between 3 and 5 min, or until the temperature changed, and heated. Each layer was assembled by manual lamination, increasing the number of layers from 4 to 6 for the bending test. They were then sent to the press at 3 Tons for 24 h, as shown in Fig. 2.

Thus, with the use of objects to remove the mold, a manufactured plate is obtained, or the still "raw" specimen (Fig. 3). In order to cut the slab to make the specimens, it was taken to the band saw with a mark 15 mm wide and 130 mm long. Once cut, this yielded 8 specimens, but 5 were used for each layer, then their thickness, width, and length were measured and then sanded to remove any resin left on the board. The specimen is then ready.

Once the specimens had been produced, they were taken for a three-point bending test, 90 mm away from the mold.

Results and Discussion

Table 1 shows the results of the bending test, and the average and standard deviation for each layer were taken from the maximum stress, in order to be able to assemble the maximum resistance graph for the specimens.

According to the values found in Table 1, they were assigned to the graph below, Fig. 4, in order to show the maximum resistance for each layer made.

The graph shows that, as a natural fiber, it has a strength ranging from 60.64 (MPa) to 77.27 (MPa). By using 6 layers of fiber, it can be seen that the specimens had a large increase in toughness. When compared with other studies, it can be seen that their strength was lower. Other authors found the flexural strength of the composites, using polyester resin, was increased from 80.77 to 85.39 MPa against an increase in weight fraction from 6.96 to 13.67% [8]. Therefore, the results obtained were not

Fig. 3 Plate made

Table 1 Flexural strength of epoxy resin composites reinforced with coconut sheath fiber (CSF)

Layers (Unit)	Flexural strength (MPa)	Standard deviation (MPa)
4	66,62	20,99
5	60,64	13,09
6	77,27	20,26

as expected, despite being a high-strength fiber. Another study shows that the fiber used as reinforcement, with alkaline treatment, obtained flexural strength results of 85.39 MPa [6]. In the future, the aim is to produce hybridized composites and utilize other types of chemical treatment in order to improve their flexural resistance.

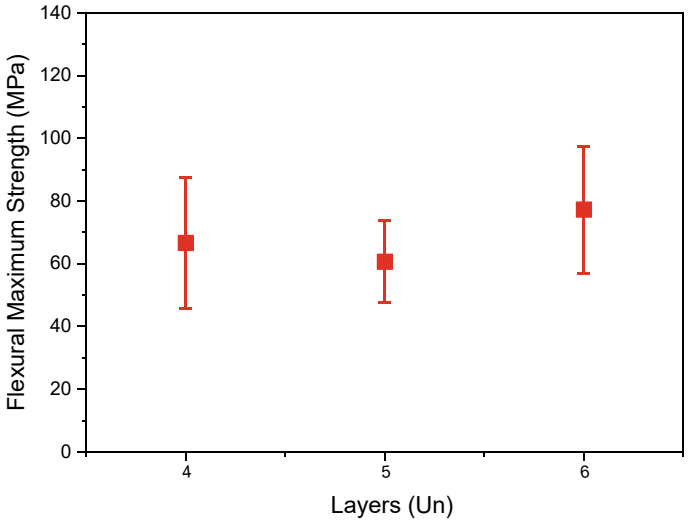

Fig. 4 Maximum strength (MPa) as a function of the amount of layer used

Conclusion

The epoxy composites reinforced with coconut sheath fiber proved to be aligned, as each layer obtained similar results. The incorporation of 6 layers in the specimen showed better flexural strength. This is probably because the fibers are closer together, thus increasing their strength.

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Compressive Strength of Composites Reinforced with Casuarina Particulate for Protective Coatings

Nicole Thomaz Aquino Drumond Coutinho, David Coverdale Rangel Velasco, Afonso Rangel Garcez de Azevedo, Carlos Maurício Fontes Vieira, Felipe Perissé Duarte Lopes, and Geovana Carla Girondi Delaqua

Abstract The need for a greener technology to help the environment and boost studies for engineering started to make those eco-conscious raw materials keeps being explored and studied as alternative solutions to synthetic materials that have being used today. The aim of this work is to develop and compare a novel ecofriendly polymeric composite based in *Casuarina equisetifolia* powder, and its results in compressive strength tests. This plant and its seeds have an intrusive growth, and it's located along the Brazilian coast, from probably Oceania. The composites will be made with a common epoxy resin (DGEBA/DETA) and the casuarina seed milled; the incorporation was in volume from 5% up to 30%, made in a silicone open mold. The results show satisfactory mechanical properties for engineering applications, for example, structure and piping hard coating in industry with very low cost.

Keywords Casuarina powder · Epoxy resin · Comparative · Ecofriendly composites · Diametrical compressive strength

Introduction

In recent years, there has been a growing interest in a "greener" modernization with less environmental impact. In this context, various research and studies in the field of composites are using natural fibers as reinforcement for a biodegradable alternative [1].

Plant-based natural fibers (lignocellulosic fibers) have the advantage of offering a vast variety of species for research, being produced either directly in nature or

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indirectly as residues mainly generated by the agroindustry. This utilization helps reduce CO2 emissions in the atmosphere and increases economic potential, benefiting both the environment and engineering [2].

Brazil stands out globally in the production of composites reinforced with natural fibers, particularly in the automotive sector, and is expanding with the support of national companies seeking sustainability, especially in the WPC sector. With greater promotion of the benefits of natural fibers, research and scientific publications on them will increase, making new products from biomass feasible and contributing to the environment [3].

Future work with lignocellulosic fibers should explore the advantages of these materials, such as the crystalline arrangement of cellulose microfibrils. Their isolated application generates countless possibilities due to the chemical differences and structure of the main components of this type of fiber [4].

Similar to natural fibers, there is also the so-called "particulate," which appears in various forms in nature and, unlike fibers, is better utilized after being ground [5].

Among other particulates, this study will focus on casuarina, a multi-use, short-rotation tree that adapts to all ecosystems. Casuarina wood is predominantly sought after for use in the fuel, construction, and paper industries as an alternative to cellulose and firewood [6]. The goal is to analyze its strength to potentially use it as a composite for anticorrosive reinforcement or in civil construction [7].

Materials and Methodology

Materials

For this test was used the casuarina seed particulate as reinforcement and an epoxy matrix composed of SQ 2050 resin and SQ 3131 hardener, both supplied by Avipol and manufactured by Silaex.

Methods

The casuarina seed, after being collected, is washed and placed in an oven until it has dried. Figure 1 shows its seed after this process.

All the seeds passed through a knife mill, and then subsequently in a ball mill.

Next, all the material is sifted through a 100-mesh sieve, and only those that pass through are used. The material that is retained goes back to the mill until it achieves the correct granulometry, as Fig. 2 shows.

After passing through the sieve, the casuarina powder is placed in an oven for 24 h at 60 °C to ensure that all moisture evaporates, and only after it is dry does the study begin.

Fig. 1 Casuarina seeds

Fig. 2 **a** The internal casuarina macrostructure horizontal and longitudinal; **b** microstructure of the center

First, its density is measured using a pycnometer.

Then we start forming the test specimens of this composite, using the particulate + resin + hardener. The polymer matrix was based on the epoxy resin Diglycidyl Ether of Bisphenol A (DGEBA) with the hardener Diethylenetriamine (DETA).

The particulate is mixed with the resin and hardener in the correct proportions and placed in a silicone mold. Once completely dry, the process of sanding begins to eliminate imperfections and achieve a uniform surface.

Results and Discussion

The aspects of internal structures of casuarina seeds are shown in Fig. 2. We can see the macro structures in the form of stars, with the center with high dense and thinning to the end. And if we look closely at the structure, there are very porous.

And after the process of milling and sieving, as shown below in Fig. 3, by SEM pictures we have a range of fibrous particle sizes, some of them rounded and others in fiber shape. Since this particle is too small, they are below the critical length, so, we consider all as particles and not as fibers anymore (Fig. 4).

The density found was 1.420 g/cm^3, and it was used to make the compositions particulate/resin/hardener. With the material duly analyzed and the 5 to 30% samples ready, they were submitted to the compressive test, at a speed of 1.3 mm/min, as shown in Table 1. The results obtained in the compressive test of casuarina powder were

Fig. 3 Casuarina powder being sieved

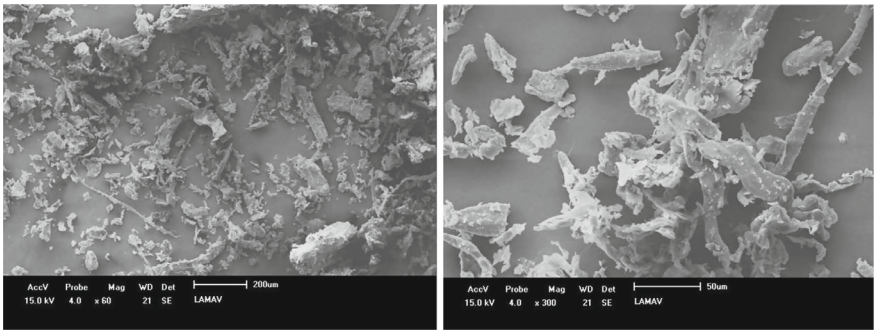

Fig. 4 Particles of casuarina seeds after being milled and sieved

satisfactory with 5 vol% of its composition, starting to fall with 10 vol% until reach the minimum on 25 vol%., and then increase again on 30 vol%, but all percentage proposed does not fall enough to discard completely, still have strength to be use in other applications (Fig. 5).

In applications where we do not need much strength, it is possible to use all the formulations proposed, for example in esthetic coating in facades, reducing the amount of synthetic resins commonly used for this.

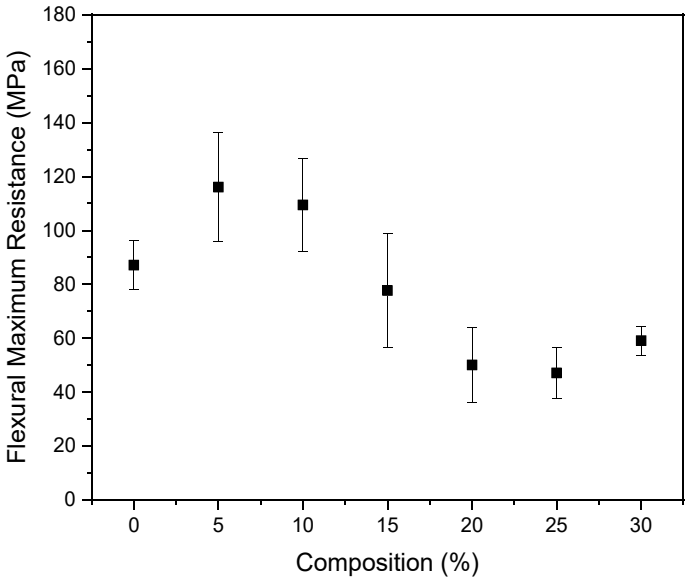

Fig. 5 Compressive strength for the composites proposed

Table 1 Sample's composition based on the addition of casuarina

ID	RESIN (%)	CASUARINA (%)
EP	100	0
5Ca	95	5
10Ca	90	10
15Ca	85	15
20Ca	80	20
25Ca	75	25
30Ca	70	30

Conclusions

This can be due to numerous factors, such as increased appearance of bubbles and an unsatisfactory mixture of the particulate with the polymeric matrix (epoxy resin), since the increase in composition generates greater difficulty in mixing. Casuarina powder has good results in tension, compression, and flexural tests when compared to other woods, which makes it a promising product in the area of natural composites [8].

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Effects of Hardener Content on Properties of Epoxy-Granite Composite Artificial Stones

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Abstract Artificial stones are composed of a matrix and a dispersed phase of hard particles. Polymers are usually used in their matrix, along with waste from the ornamental stone industry as particulate matter. The properties of polymeric materials are directly influenced by their structure. When used as a matrix for the development of composite materials, it is possible to influence the final properties by controlling the characteristics of the matrix. Thus, the present work aims to study the impact of changing the hardener content on the properties of artificial stones. The slabs were produced using a vibration and compaction system with vacuum. Fifteen percent of the epoxy resin was used based on the mass of granite waste. Formulations with 10%, 15%, and 20% hardener were evaluated. The flexural strength improved with the use of 20%, while the compression strength showed better results with the use of 15%.

Keywords Artificial atone · Composite · Polymer matrix

Introduction

Artificial stones represent a technological advance, being produced with polymer resins and a high proportion of natural aggregates, which can be partially replaced by waste. These stones tend to exhibit superior mechanical properties compared to natural stones, as the inclusion of resin reduces porosity and water absorption while

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improving mechanical resistance, making them suitable for demanding applications such as floor and wall coverings [1–3].

Artificial stones are typically developed using ornamental stone waste, although waste from different types and sources can also be utilized. Artificial stones can be produced from SiO_2 or $CaCO_3$ base materials. Both artificial and natural stones, when made from silica, tend to have better physical and mechanical properties [4].

The use of a low-density polymeric matrix in artificial stones results in less weight compared to natural stones, leading to a reduction in the necessary structural loads and logistical costs associated with architectural and structural projects. Therefore, artificial stones are highly recommended for construction applications [4, 5].

Materials based on epoxy oligomers find applications in diverse sectors, including aerospace, automotive, and radio engineering, among others. Epoxy resins based on Bisphenol A (DGEBA) are among the most widely used. These resins, when formulated into adhesives, provide high adhesive strength, mechanical resistance, and chemical resistance, in addition to exhibiting low shrinkage and reduced costs. To optimize the performance of adhesives and ensure the desired strength and durability in adhesive joints, it is imperative to investigate the gelling and curing processes [6, 7].

Epoxy resins are among the most significant and widely used polymers as a matrix, due to their excellent adhesion, tenacity, thermal properties, and adequate electrical conductivity. The mechanical behavior and chemical resistance of EVO-based thermosets are strongly influenced by the nature of the cross-linking reactions, which depend on the chemical properties of the hardeners and the physical parameters of the process [8, 9].

In the pre-polymerized state, epoxy resins are in liquid form and require a cross-linking process to be converted into a thermosetting polymer. Type A epoxy resins have terminal epoxy groups (with two functionalities), which consist of an oxygen atom bonded to two carbon atoms, forming a three-membered ring. The molecules that react with these epoxy groups are called hardeners and curing or cross-linking agents. These hardeners facilitate the formation of a three-dimensional network by promoting bonds between resin molecules [10–12].

This mechanism is highly dependent on the composition and proportion of reactants, as well as temperature conditions and other operational factors [7]. Therefore, the objective of this study is to evaluate the effect of varying the proportion of hardener used in the production of artificial stones.

Materials and Methods

The waste used in this research comes from the cutting stage of Sienna white granite, supplied by the company Brumagran in slab format. To convert it into particulates, the material was initially broken with a sledgehammer, followed by crushing in a jaw crusher and comminution in a ball mill. The material selected for the study

corresponds to the fraction retained between 10 and 40 mesh sieves, with particle size between 2000 and 0.425 microns.

The resin selected was MC 130, supplied by EpoxyFiber. Based on previous work [13], a 15% resin content was adopted in relation to the mass of the residue. Three different rocks were produced by varying the hardener content used, RA10, RA15, and RA20, adding the hardener respectively in proportions of 10%, 15%, and 20% in relation to the resin mass, FD 129 hardener was used, provided by EpoxyFiber.

Initially, the residue was dried in an oven for a minimum period of 24 h at 100 ± 5 ºC. The particulate, after drying, was added to a cylindrical mixer, where the resin was subsequently added. The mass was stirred until it exhibited a homogeneous character and, finally, the hardener was added to the mass, waiting for complete homogenization.

The mixer was coupled to the metal mold and subjected to a vacuum of 600 mm Hg for 2 min. The dough was then poured into the mold, which was moved to the sieve shaker. The mold was vibrated for 2 min at 60 Hz and then pressed using a temperature-controlled hydraulic press. A compaction pressure of 10 MPa was maintained for 20 min, with the press temperature adjusted to 90 ºC.

Plates with dimensions of 100 × 100 × 10 mm were produced, which were later cut into specimens with specific dimensions for each test. The influence of hardener content on physical, mechanical, and thermal properties was analyzed.

Physical indices are a set of three properties that provide important information about the application of rocks: apparent density, apparent porosity, and water absorption. Ten specimens were produced with dimensions of 50 × 50 × 10 mm, in accordance with NBR 15,845–2 [14]. The specimens were washed and submerged in water. After periods of 2 and 48 h, the saturated and submerged specimens were weighed. Then, the specimens were dried in an oven for 24 h and weighed again.

According to NBR15845-6 [15], 5 specimens with dimensions of 25 × 100 × 10 mm were prepared. The 3-point bending test was carried out on a machine with a speed of 0.25 mm/min and a distance between the lower supports of 0.80 mm.

The uniaxial compression test was carried out based on NBR15845-5 [16]. 5 specimens were cut from the specimens used in the bending test, with dimensions of 25 × 25 × 10 mm. The test was conducted at a speed of 0.50 MPa/s.

Results and Discussion

Table 1 presents the results obtained after testing physical indices.

According to Chiod Filho and Rodriguez [17], natural granites must have a density above 2.55 g/cm^3, with an average density of 2.63 g/cm^3. The artificial stones RA10, RA15, and RA20 had densities ranging from 2.00 to 2.05 g/cm^3. The first advantage observed in artificial stones is their lower density, achieved by using polymeric resins as matrices. The difference in density among materials impacts design and logistics, including transportation and storage, resulting in reduced freight costs or a decrease in the structural support required for the plates.

Table 1 Results of physical indices

Physical properties	RA10	RA15	RA20
Density (g/cm^3)	2.05 ± 0.02	2.00 ± 0.06	2.04 ± 0.08
Water absorption (%)	0.68 ± 0.21	0.50 ± 0.07	0.69 ± 0.23
Apparent porosity (%)	1.66 ± 0.39	1.19 ± 0.14	1.64 ± 0.53

The stone application guide [17] states that stones with water absorption between 0.4% and 1.6% are classified as medium water absorption stones. The stones produced in this research fall within this medium absorption range.

Figure 1 presents the results of water absorption and apparent porosity. Water absorption is related to the open porosity of the material and the interconnectivity of the open pores; thus, an increase in porosity results in an increase in water absorption [13], as shown in the graph. It is evident from the graph that the stones RA10 and RA20 exhibit greater water absorption than RA15, which has a lower porosity.

According to Ozgul and Ozkul [13], variations in the percentage of hardener used lead to changes in viscosity. The authors emphasize that hardeners can reduce the viscosity of resins, which affects flow behavior and alters the workability of the mixture.

On the other hand, Borsellino et al. [19] identified distinct behaviors for stones manufactured with epoxy and polyester resin. The higher viscosity of the epoxy resin contributed to a more uniform distribution of particles within the matrix. However, the authors observed that the effects of sedimentation during curing lead to the formation

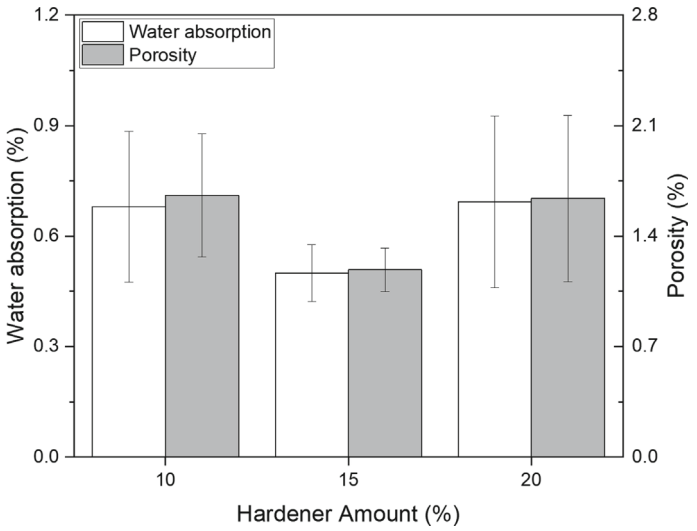

Fig. 1 Results of water absorption and porosity

of regions with varying concentrations of marble particles, resulting in areas with differing densities.

Thus, the variations in porosity and absorption, as well as the increase in standard deviations, can be attributed to changes in viscosity caused by the increase in the percentage of hardener. The increase from 10 to 15% in the amount of hardener improved workability, promoting better resin dispersion and reducing porosity. However, the increase from 15 to 20% resulted in an excessive reduction in viscosity, which led to the formation of regions with different resin concentrations and, consequently, variations in pore concentrations, explaining the high standard deviation observed for this formulation.

The results of the mechanical tests are presented in the graph in Fig. 2. A trend of improvement in flexural strength is observed when observing the values of RA10 (20.3 MPa), RA15 (21.0 MPa), and RA20 (22.9 MPa). The best result of the compression test was observed for the composition with 15% hardener (142.1 MPa), as RA10 presented a resistance of 95.2 MPa while RA20 resisted 120.0 MPa.

According to Salehi and Rash-Ahmadi [18], by increasing the weight percentage of the hardener in relation to the epoxy matrix, an increase in the amount of cross-linking between the epoxy molecules and the hardener is observed, resulting in an improvement in material resistance. Additionally, there is an increase in the number of hardener molecules present. This increase in the amount of hardener molecules expands the possibility of forming more covalent bonds with the epoxy resin molecules.

The loss of compressive strength observed when increasing the amount of hardener from 15 to 20% can be explained by the exothermic reaction that causes the material to heat up during the curing process. This effect is more intense when a greater

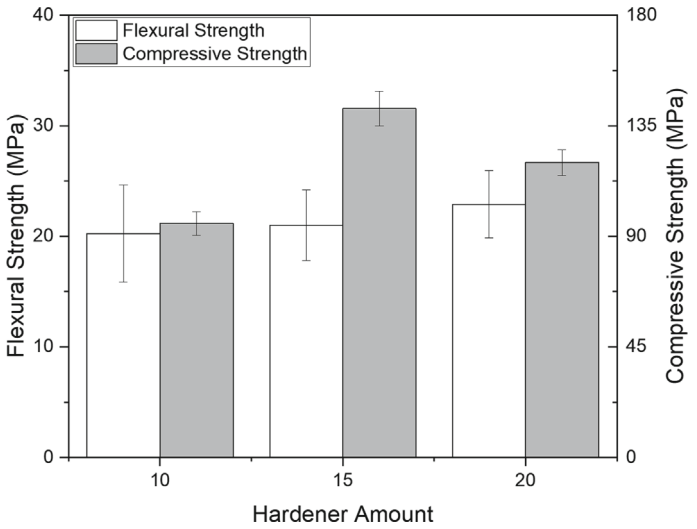

Fig. 2 Results of mechanical properties as a function of phr

amount of hardener is used, which can harm the performance of the composites [19, 20].

Chiod Filho and Rodrigues [17] classify rocks according to their flexural strength as very low (<6.0 MPa), low (6.0–10.0 MPa), medium (10–16 MPa), high (16–20 MPa), and very high (>20.0 MPa). The three rocks are classified as very high resistance.

NBR15845-5 [16] presents a minimum requirement of 100 MPa for compression of natural granites, while NBR15845-6 [15] requires a minimum resistance of 10 MPa in flexion of natural granites, RA10, RA15, and RA20 present resistance to compression and flexion higher than the values required by standard.

Gomes et al. [13] produced artificial stones using granite residue and epoxy resin, and with 10% hardener, the stone exhibited a resistance of 32 MPa, a value that is higher than those found in the present study. The authors utilized a wider granulometric range, resulting in a material with better packing and lower pore content. They attributed the high resistance to the low percentage of voids, which can act as stress concentrators—an observation that aligns with the apparent porosity results for stones RA10, RA15, and RA20.

Demartini et al. [19] developed an artificial marble composed of 15% by weight of epoxy resin and 85% of dolomitic marble residue. The final product achieved a flexural strength of 33.93 MPa and a uniaxial compressive strength of 96.49 MPa. The authors attributed these results to satisfactory adhesion between the waste particles and the resin. Although the flexural strength may have surpassed that of the present work, it is evident that the increase in hardener content in RA15 and RA20 resulted in a compressive strength more than 50% higher.

Conclusions

The stones developed in this research have a void content that must be considered regarding their final application. The hardener content did not result in noticeable changes to the physical properties and flexural strength. However, the compressive strength improved by 35% when the hardener content was increased from 10 to 15%, which can be attributed to the greater amount of cross-linking.

The values obtained exceeded the requirements set by technical standards and application specifications, resulting in a material with high mechanical properties and low density.

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EVA and Ramie Fiber: 3D Filament Manufacturing and Additives

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Abstract 3D printing with filament has advanced considerably, allowing the manufacture of complex objects with high precision. However, printing flexible materials represents a challenge due to the strength and high deformation characteristics of these materials. This study proposes the development of a reinforcement for the EVA polymer, using ramie fiber, in order to facilitate 3D printing using the FDM method, maintaining the flexibility of the material. The hot melt extrusion process was carried out in a mini-extruder, where the polymers were mixed in different proportions with reinforcement with natural fiber at 10%, 20%, and 30%. The results showed variations in the apparent density and tensile strength of the different polymer mixtures, at a speed of 500.0 mm/min and a capacity of 1 kN. The data obtained in the work were analyzed statistically and significant differences could be observed in the mechanical tensile test.

Keywords Additive manufacturing · Fused deposition modeling · 3D printing

Introduction

3D printing has experienced significant advances, pushing technology to new heights of precision and detail [1, 2]. The use of manufacturing generates greater fidelity in production, a simple workflow, reduced material waste, and the possibility of using sustainable materials [3, 4]. However, sustainability in Industry 4.0 is still in its early stages, requiring more studies to consolidate this emerging technology [5].

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The growing demand for sustainable and efficient processes has driven the addition of by-products to the polymer matrix for 3D printing, contributing to the valorization of waste and the saving of raw materials [6]. Recent studies demonstrate the success of this approach, such as the use of natural fibers in composites, where the Fused Deposition Modeling (FDM) technique stands out for its applicability in the production of biocomposites [3, 6, 7]. Examples that result in sustainable filaments for 3D printing include the use of flax fibers [8], bamboo [8], jute [9, 10], pineapple [11], soy [12], palm [13], and rice husk [3, 14], in all cases combined with PLA.

Ramie fiber (Boehmeria nivea), extracted from the stem of the plant, stands out for its high tensile strength, surpassing 1000 MPa, which is superior to other plant fibers such as hemp, flax, and cotton. Belonging to the family of long fibers, with an average length of 150 to 200 mm, ramie is renewable, non-abrasive to processing equipment, safe to handle, and incinerable at the end of its life cycle, allowing for energy recovery. Additionally, composites reinforced with natural fibers like ramie exhibit excellent tensile and flexural strength, greater ductility, impact resistance, and hardness, while combining high stiffness and strength with low weight and excellent corrosion resistance [15–18]. Despite its notable properties, such as good durability, absorption, and brightness, its use has decreased due to competition with cheaper and higher quality synthetic fibers. However, ramie remains valued in segments such as textile manufacturing, rope making, paper pulp for banknotes, and other industrial products, continuing to be a promising option in a context of growing demand for sustainable composite materials [17, 18].

Current studies in the literature that used ramie fiber in a continuous format, being introduced into the 3D printer hot end along with the filament, did not go through a process of mixing and extruding a filament with the incorporated fiber [19–23]. It was also observed that there was a significant increase in tensile and flexural strength [23] and that there was an alteration in the fracture behavior of the biocomposites, changing to brittle fracture. Based on these findings, the current research extends its focus to explore the use of ramie fiber in short fiber formats in EVA polymer as a matrix, using an extruder to achieve a homogeneous mixture. The goal is not only to contribute to the broader use of ramie fiber as a by-product, thus reducing its environmental impact, but also to investigate the potential effects it may have as a reinforcement for EVA.

Printing flexible materials is a challenge within additive manufacturing; this type of material is characterized by resistance and high deformation [24]. The challenge of printing flexible objects using 3D printing technologies with filament represents an exciting frontier in expanding the capabilities of these technologies, allowing for the creation of objects that can bend, twist, and flex without losing their structural properties. This opens doors to innovative applications in sectors such as footwear, flexible medical devices, and even robotics, where flexibility is a crucial characteristic [25].

Ethylene–vinyl acetate (EVA), a polymer known in the industry as a versatile material, is widely used for its flexibility and durability, being employed in a variety of applications, from sports to industrial coatings. It is not marketed with a 1.75 mm diameter. The authors [26, 27] point out the difficulty in using pure EVA and research

the addition of commercial polymers or drugs to increase strength and the possibility of using the polymer. Recently, ethylene and vinyl acetate copolymers (EVA) with the addition of ABS, recently arrived on the market, has been presented on the market. However, there is still no data in the literature on this material for 3D printing by filaments, and one of the disadvantages of this filament is the 2.75 mm diameter, which requires specific and high-cost printers for use. Increasing the diameter for this material has been one of the solutions found so far.

Materials and Methodology

Materials

The selected materials were the ethylene–vinyl acetate (EVA) copolymer from BIOART and ramie fiber short fiber formulations. The short fiber was only sieved to remove large particles and larger fibers using a sieve with a 0.15 mm opening, 100 Mesh. The filler amounts used were 10%, 20%, and 30% respectively and 0.5 g of carbon dioxide.

Hot Melt Extrusion Process

Initially, the ramie fiber and EVA polymers were mixed in a Mini-extruder—Haake MiniLab III—Thermo Scientific, where the mixture was induced to melt by heat. The mixture was fused at 200 °C for 20 min to achieve a homogeneous blend. This mini-extruder can handle up to 8 g of material per mixture.

This approach was considered more suitable for our experimental setup. About 1000 m of filament was produced for each composition. Following the manufacturer's instructions, the mini-extruder was set to 205 °C and 20.0 RPM for 20 min before starting the extrusion process. During the extrusion process, the filaments were vacuum-sealed.

Density of Material and Apparent Density

The ramie fibers and EVA were weighed on a precision balance with an accuracy of 0.001 g, brand InnerScan, at LAMAV/UENF. Using the length and diameter data, it was possible to calculate the volume of each fiber, and then the density of the ramie fiber was determined.

The diameter measurements were carried out using a profile projector, available at LAMAV/UENF. Five measurements were taken, and subsequently, the filaments

were rotated 90º for additional measurements. This approach was important for obtaining an average and thus determining the density of the filaments.

Tensile Testing

To determine the mechanical properties of the filament, its tensile strength was investigated using an Instron universal testing machine model 5582 at LAMAV/UENF, with a speed of 500.0 mm/min and a capacity of 1 kN. The preparation of the test specimens for the test was based on the recommendations of ASTM D638 [28] and ASTM D3822-07 [29]. Each filament was cut to a length of 160 mm, and adhesive tape and aluminum foil were attached to each of its ends, reducing the effective length of the test specimen to 100 mm. The purpose of this reduction was to protect the fibers from damage that could be caused by the grips of the universal testing machine and to prevent slippage.

Microstructure Characterization

This was performed using Scanning Electron Microscopy (SEM) with a Tescan Mira FEG SEM.

Results and Discussion

The analysis of the density of ramie fibers and EVA revealed distinct values that reflect the material characteristics of these components. Ramie fibers exhibited a density of 1.14 g/cm^3, which is significantly higher than the density of EVA, measured at 0.90 g/cm^3. This difference in densities can be attributed to the intrinsic properties of the materials: ramie, being a natural material with a denser and more compact structure, shows a higher density compared to EVA, which is a polymer with a lighter and more porous structure. The lower density of EVA contributes to its lightweight and flexible properties, while the higher density of ramie indicates its capacity to provide structural reinforcement and additional strength when incorporated into EVA.

The average apparent density of various mixtures of ethylene–vinyl acetate (EVA) with ramie fiber varies according to the proportion of ramie. The analysis of the mixtures with ramie fiber, presented in Table 1, shows that this average density changes based on the EVA proportion.

The tensile strength results for the EVA and ramie fiber mixtures show a clear trend with the variation in EVA proportion. At 0% EVA, the tensile strength was 7.3 MPa $\pm$ 1.3. As the proportion of ramie increased to 10% and 20%, the tensile strength also increased, reaching 8.2 MPa $\pm$ 3.04 MPa and 8.39 MPa $\pm$ 3.27 MPa, respectively.

Table 1 Apparent density of different mixtures

Sample	Apparent density
100% EVA	0.00090 ± 0.00021
With 10% ramie	0.00086 ± 0.00006
With 20% ramie	0.00087 ± 0.00008
With 30% ramie	0.00128 ± 0.00031

This increase is attributed to the improved interaction between the EVA matrix and ramie fiber reinforcement, leading to better load distribution. A significant increase was observed at 30%, where the tensile strength rose considerably to 14.35 MPa ± 3.27 MPa. This result suggests that a higher proportion of ramie contributes to a more cohesive matrix, which can better support applied loads and enhance overall strength. Thus, while the addition of ramie up to 20% shows incremental improvements in tensile strength, the 30% proportion seems to optimize strength, indicating the need for a balanced ratio of matrix and reinforcement to maximize material performance.

This observation is corroborated by the maximum strain data, where at 10% ramie, the maximum strain increased to 411% ± 34%. However, this increase is reduced with further addition of the reinforcing fiber, with 20% and 30% ramie showing slight reductions in maximum strain to 391% ± 35% and 329% ± 31%, respectively.

The microscopy of the sample containing 0% ramie fiber, compared to pure EVA, revealed a frayed filament with a shell-like appearance (Fig. 2a). With the addition of 10% ramie fiber, the fibers were observed to be integrated into the polymer chains, exhibiting an elongated structure (Fig. 2b). Additionally, using EDS, titanium dioxide particles were visualized as being incorporated into the material (Fig. 2c, d).

In Fig. 3a, b, the interface between the polymer matrix and the fiber reinforcement can be observed, showing delamination caused by the mechanical test, which affects the integrity of the fiber-matrix interface. The elongated chains of the EVA polymer are visible, demonstrating plastic deformation consistent with the results shown in Fig. 1, highlighting the polymer's ductility.

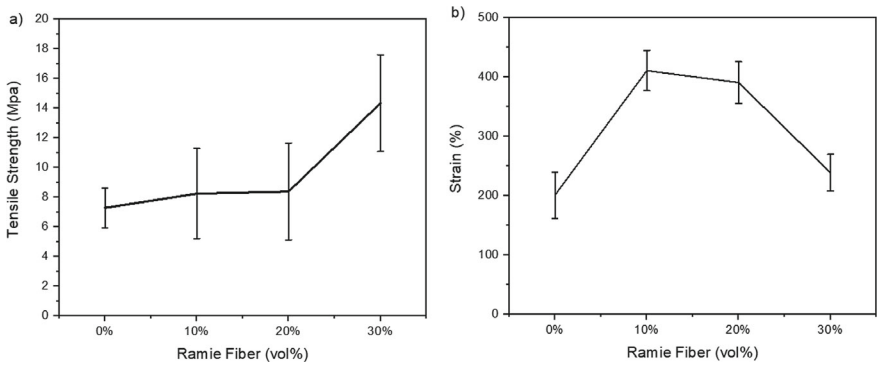

Fig. 1 **a** Tensile strength mixtures with ramie fiber; **b** Strain

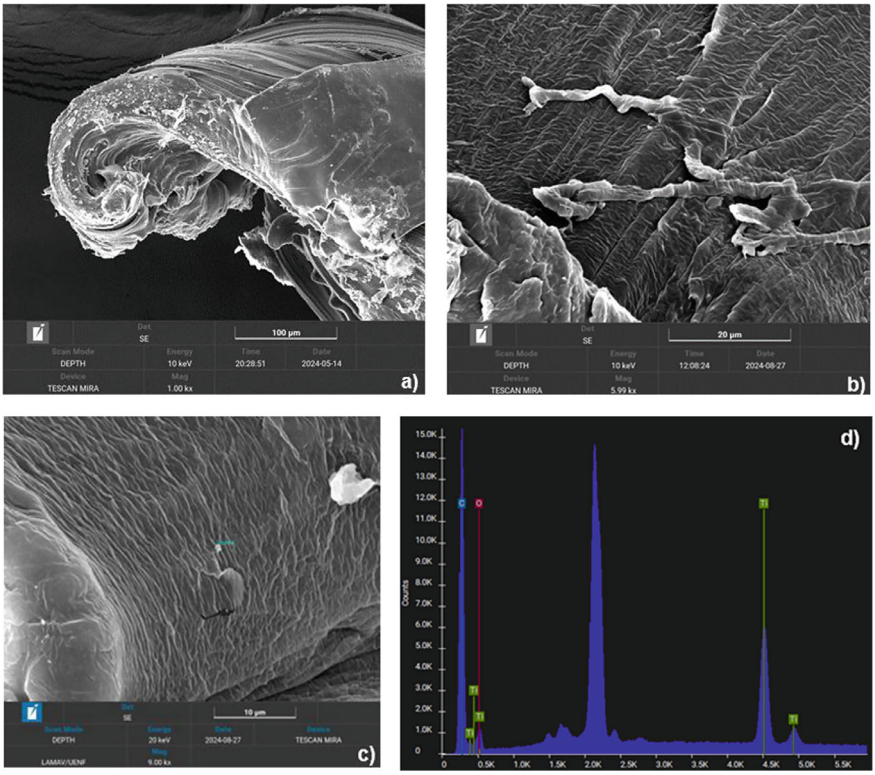

Fig. 2 SEM micrographs of fractured surface for EVA-RAMI samples at magnifications of **a** 0%, **b** 10%, and **c** Sample with titanium dioxide and **d** EDS

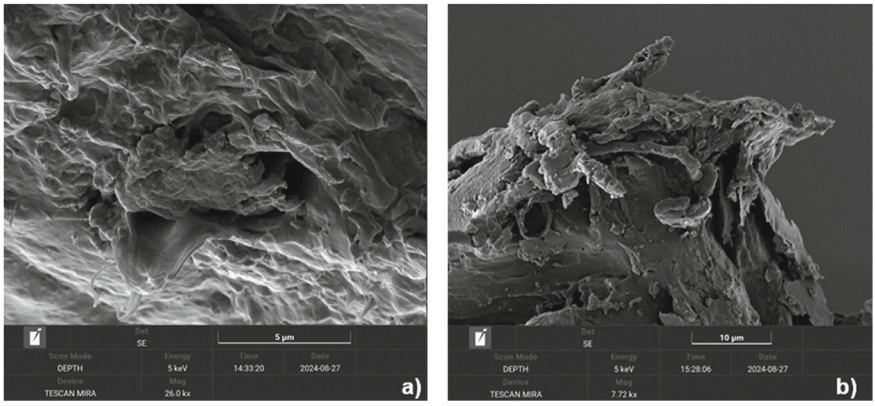

Fig. 3 SEM micrographs of fractured surface for EVA-RAMI samples at magnifications of **a** 20% and **b** 30%

The analysis of the results reveals a significant interaction between the EVA matrix and ramie fiber, particularly in terms of density and mechanical strength. The apparent density varied according to the ramie fiber content, with the sample containing 30% ramie showing the highest value (1.28 g/cm^3), reflecting the reinforcing effect of the fiber on the composite structure. This increase in density correlates with the improvement in tensile strength, which reached 14.35 MPa at 30% ramie, indicating better load distribution and matrix cohesion with fiber addition. However, the higher fiber concentration affects the maximum strain, with the 30% sample showing a significant reduction in flexibility (329% strain). Scanning electron microscopy (SEM) confirmed good fiber-matrix integration up to 20% ramie, but at 30%, delamination areas were observed, indicating potential failures in the fiber-matrix interface. The EDS analysis, shown in Fig. 2c and d, highlights the incorporation of titanium dioxide (TiO_2) particles within the composite material. While TiO_2 can enhance UV resistance and mechanical properties, its presence may also contribute to localized stiffening of the matrix. Additionally, the peak at approximately 2.3 keV in the EDS spectrum likely corresponds to sulfur, indicating the presence of sulfur-containing compounds.

Conclusions

This study investigated the reinforcement of EVA with ramie fiber to enhance its suitability for 3D printing using the FDM method while maintaining the material's flexibility.

The incorporation of ramie fibers into the EVA matrix resulted in significant changes in both density and mechanical properties, particularly tensile strength and strain.

The results suggest that ramie fiber can be an effective reinforcement for EVA in 3D printing applications, particularly when a balance between flexibility and strength is required. The optimal fiber content for maximizing mechanical performance appears to be 30%, which provides the greatest tensile strength without excessively compromising the material's flexibility.

Future research could explore further optimization of fiber content and investigate other natural fibers to enhance the properties of EVA-based composites.

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Evaluating Impact Resistance of Epoxy Composites Incorporating FGD Gypsum

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Abstract In recent decades, the rapid growth of industrial production has significantly increased the amount of industrial waste, posing considerable environmental challenges. One such waste, FGD gypsum, generated in the desulfurization process of combustion gases in thermal power plants, presents potential for sustainable reuse to mitigate environmental impacts. The objective of this study is to evaluate the impact resistance of epoxy composites with different proportions of FGD gypsum. This study investigated the feasibility of incorporating FGD gypsum into epoxy systems, with 0, 10, 20, 30%vol. The impact test was of the Izod type, carried out according to procedures established by ASTM D256. The results were analyzed using analysis of variance and Tukey test, aiming to verify whether the effects were significant. Through this work, it is possible to observe the influence of the addition of residues in epoxy systems, as well as investigate the effects on impact resistance.

Keywords Composites · Sustainability · Mechanical properties

Introduction

Industrial production is one of the pillars of the modern economy, responsible for transforming raw materials into finished products. In Brazil, this sector recorded a growth of 8.4% in April 2024 compared to the same month in 2023 [1]. However, this

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process generates a significant amount of industrial waste, which can be classified into two main categories: hazardous waste (Class I) and non-hazardous waste (Class II). Hazardous waste includes materials containing flammable, toxic, or corrosive substances, while Class II waste encompasses materials with characteristics such as combustibility, water solubility, or biodegradability [2]. Proper management of this waste is essential to minimize environmental impacts and promote sustainability.

FGD gypsum is a byproduct of the Flue Gas Desulfurization (FGD) process, in which a lime slurry reacts with gases generated from coal combustion to produce coke in a metallurgical process. Lime-based products are efficient reagents for capturing contaminants, retaining SO_2 with an efficiency of up to 99%, thus contributing to atmospheric emission control [3]. This gypsum is chemically similar to natural gypsum, with FGD gypsum being purer (gypsum content above 96%) than natural gypsum (80%) [4]. The reuse of industrial waste provides environmental benefits by reducing materials and pollutants, as well as decreasing the use of natural resources [5]. It also contributes to a circular economy by transforming a byproduct into a valuable resource.

To mitigate these environmental impacts, research is being conducted in various countries to find new eco-efficient materials with suitable technological properties and durability for intended engineering applications [6]. Composite materials offer high strength-to-weight ratios and lower production costs [7]. The creation of composites is a promising approach to incorporate industrial waste, such as gypsum. They are used in various industries, including aerospace, automotive, and construction, where their unique properties provide significant advantages over traditional materials [8, 9]. Previous studies on gypsum-epoxy composites have demonstrated the feasibility of using this byproduct in engineering applications, showing that these composites can improve mechanical and thermal properties compared to pure resins [10, 11].

The need for their development arises from the quest for more sustainable, high-performance materials widely used in structural applications, distinguished by their excellent mechanical properties and lower density compared to traditional alternatives [12]. In this context, waste management provides benefits for both organizations and society, promoting the commercialization of these materials and generating revenue through recycling and sustainability practices [13], offering new solutions to longstanding challenges. When this material is also lightweight, it offers a significant advantage in terms of strength-to-weight ratio compared to conventional materials [14]. The use of composites with industrial waste helps reduce the volume of waste discarded, minimizing the environmental impact associated with these materials.

The main objectives of this approach include reducing the amount of discarded industrial waste and creating materials with enhanced properties. By utilizing FGD gypsum in epoxy matrix composites, the goal is to develop products that can replace conventional materials, resulting in a new material with unique properties and an improved combination of characteristics compared to the individual materials that comprise it [15]. The use of FGD gypsum in composites aims to develop products that can substitute conventional materials, offering environmental and economic benefits.

Moreover, the research and development of these composites could open new market opportunities and applications. Composite materials have a wide range of applications and are widely used in aerospace, naval, automotive, petrochemical, and electronic industries [16]. To mitigate these environmental impacts, research is being conducted in various countries to seek new eco-efficient materials with suitable technological properties and durability for the intended engineering applications [6]. The objective of this work is to evaluate the influence of FGD on the impact resistance of epoxy matrix composites.

Materials and Methods

The FGD waste was supplied by ArcelorMittal Brazil. The provided particle size was below 0.075 mm, with sieving performed to ensure material homogeneity, followed by drying in an oven at 120 °C. For the matrix, the epoxy system selected was Bisphenol A Diglycidyl Ether/Diethylenetriamine (DGEBA/DETA) with a phr of 16. The resin and hardener were purchased from Avipol under the codes SQ1005 and SQ3131, respectively, and were manufactured by Silaex. The volumetric ratios analyzed in the study included 0, 10, 20, and 30% of FGD gypsum relative to the resin volume. Seven samples were produced for each formulation using silicone molds. After an initial curing period of 5 days, the samples underwent a post-curing process for 3 h at 70 °C. Each sample was then sanded and notched using a broaching machine to meet testing guidelines. The dimensions of the test specimens are presented in Figs. 1, and 2 depicts the test specimens for each formulation.

After the preparation of the formulations, the samples were subjected to the Izod impact test, following the procedures outlined in the ASTM D256 standard [17]. The test was conducted on an Izod impact testing machine at the State University of Northern Rio de Janeiro in Campos dos Goytacazes (RJ), using a 22 J hammer. The significance of the results was evaluated through an analysis of variance (ANOVA), following the procedure established by Velasco et al. [18], with a significance level set at 5%.

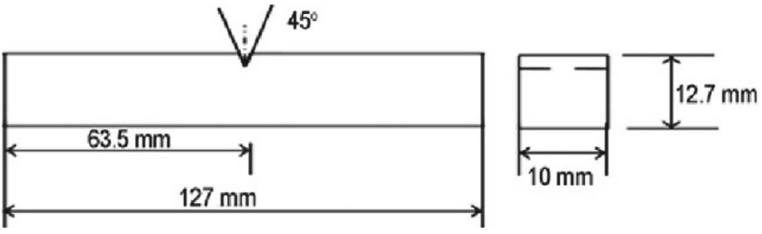

Fig. 1 Dimensions of the Izod impact test specimens [15]

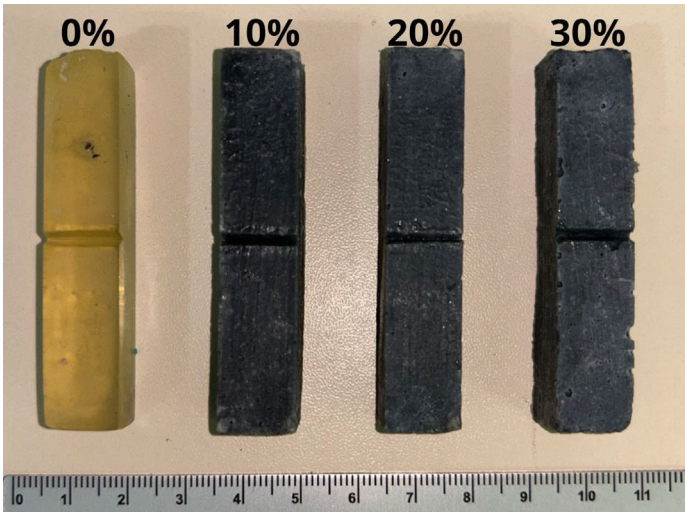

Fig. 2 Test specimens of each FGD gypsum formulation in relation to the resin

Results and Discussion

Figure 3 presents the notch resistance of the tested specimens. From this, it can be observed that the notch resistance did not vary significantly, which is confirmed by the P-values shown in Table 1.

Similarly, the impact resistance, presented in Fig. 4, aligned with the notch resistance, with non-significant variations, as the P-values presented in Table 2 are above the significance level adopted in this study.

From a technological perspective, when comparing this result with the literature, it is evident that FGD gypsum exhibits a more stable behavior, with no significant variations in strength due to the amount of particulates [19–21]. Velasco et al. [21] observed that one of the studied wastes showed a performance loss with the use of only 10%, a gain with 20%, maintenance of properties with 30%, and another gain with 40%. This was attributed to the material's behavior being the result of different mechanisms, with the final effect depending on the amount of waste.

In this context, FGD gypsum stands out as a waste that can be utilized more flexibly, allowing adjustments in the material's thixotropy according to the application, without concerns regarding variations in impact resistance. Furthermore, characteristics such as aesthetics or other properties can serve as references for selecting the ideal formulation. Moreover, the use of FGD gypsum has proven to be promising, as it allows for the replacement of a more expensive material with a waste product, without compromising the evaluated mechanical properties. Thus, by giving appropriate use to a material that would otherwise be discarded, socio-environmental and economic benefits are achieved, given that there is a partial substitution of the epoxy system with a waste material [22–25].

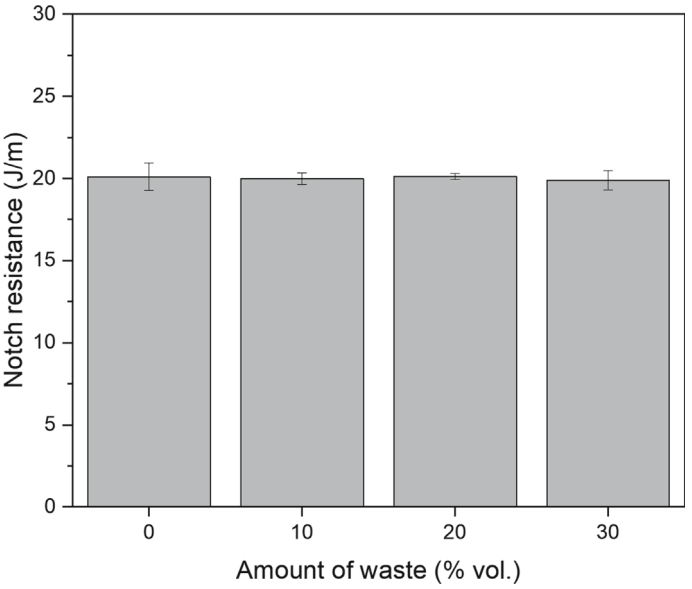

Fig. 3 Notch resistance x amount of waste

Table 1 Statistical analysis of notch resistance in comparison to the pure epoxy system

Waste amount (% vol.)	Notch resistance (J/m)	P-value (%)
0	20.09 ± 0.84	–
10	19.98 ± 0.36	79.48
20	20.11 ± 0.19	95.64
30	19.88 ± 0.59	65.87

Conclusions

The study shows that adding FGD residue to the epoxy system does not significantly affect notch or impact resistance, demonstrating stable mechanical performance across different concentrations. FGD's versatility allows for customization of material properties, such as thixotropy, without compromising strength. Additionally, the use of FGD gypsum offers economic and environmental benefits, enabling the replacement of more expensive materials while supporting the sustainable reuse of industrial waste.

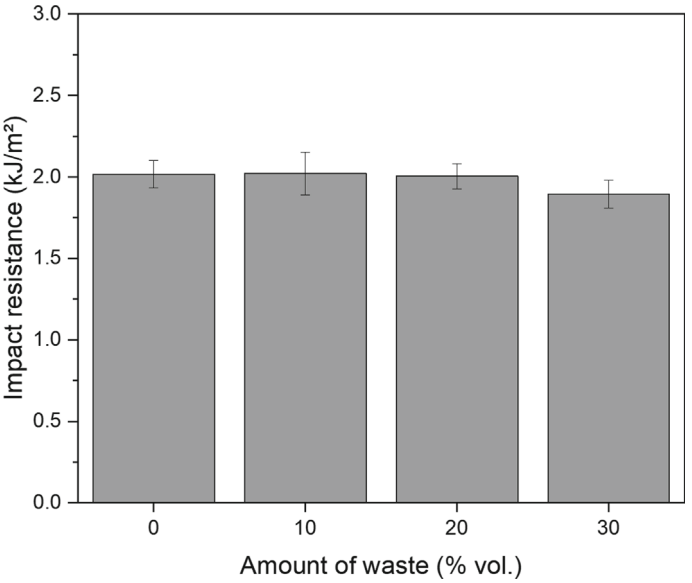

Fig. 4 Impact resistance x amount of waste

Table 2 Statistical analysis of impact resistance in comparison to the pure epoxy system

Waste amount (% vol.)	Impact resistance (kJ/m^2)	P-value (%)
0	2.02 ± 0.08	–
10	2.02 ± 0.13	95.52
20	2.00 ± 0.08	80.27
30	1.89 ± 0.09	5.20

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Incorporation of Coffee Grounds Powder as Reinforcement in Epoxy Resin for High-Performance Industrial Coatings: A Blue Ocean Strategy Analysis

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Abstract In some organic waste treatment facilities, coffee grounds are utilized for biogas production through anaerobic digestion processes. However, this method is expensive and complex. An innovative alternative involves transforming coffee grounds into a new product, a high-performance coating. This study explores a strategic analysis for this new product. The composite is created by adding coffee grounds in an epoxy matrix as reinforcement. Different proportions of coffee grounds were added to epoxy, and resulting composites evaluated for mechanical and thermal properties. Previous analysis shows some promising results for the material, but none of the evaluations of fabrication cost or revenue was done, so a Blue Ocean Strategy analysis identified market opportunities and compared the obtained product with today's market ones. Coffee grounds in epoxy offer a sustainable option, opening new market avenues with competitive performance and cost. This composite could impact industrial coatings, promoting eco-friendly practices.

Keywords Composites · Environmental effects · Sustainability

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Introduction

The increasing global demand for sustainable practices has driven innovation in various industries, including waste management and materials science. Among the myriad waste streams generated daily, coffee grounds represent a significant, yet often underutilized, resource [1, 2]. Traditionally, organic waste treatment facilities have employed coffee grounds in biogas production through anaerobic digestion processes [3]. While this method is effective, it is also associated with high costs and operational complexity, limiting its widespread adoption. Consequently, there has been a growing interest in exploring alternative, more economically viable applications for coffee grounds that not only mitigate waste but also add value to industrial processes [4–6].

One such innovative approach involves the transformation of coffee grounds into a high-performance coating material. By incorporating coffee grounds into an epoxy matrix as a reinforcement agent, researchers have begun to explore the potential of these composites to deliver competitive mechanical and thermal properties [7, 8]. The development of this composite material aligns with the broader trend of creating sustainable products that reduce environmental impact while maintaining, or even enhancing, performance standards [9].

In the context of this research, the focus has been on understanding how varying the proportions of coffee grounds within the epoxy matrix influences the composite's properties. Preliminary studies have shown promising results, indicating that these composites can offer desirable mechanical strength and thermal stability [10]. However, while the technical feasibility of the material has been explored, there has been a noticeable gap in the assessment of the economic viability of this innovation [11, 12]. Specifically, no comprehensive evaluations have been conducted to analyze the fabrication costs or potential revenue streams associated with this new product.

To address this gap, a strategic analysis rooted in the principles of Blue Ocean Strategy was conducted. Blue Ocean Strategy, which focuses on creating uncontested market spaces and making the competition irrelevant, provides a framework for identifying market opportunities that have yet to be fully exploited [13]. By applying this strategy to the development of coffee grounds-based epoxy composites, this study aims to uncover new market avenues and compare the performance and cost-effectiveness of this innovative material against existing industrial coatings [14, 15].

The potential impact of coffee grounds in epoxy extends beyond just technical and economic benefits. This composite material represents a sustainable option that can contribute to the promotion of eco-friendly practices within the industrial coatings sector. By diverting coffee grounds from traditional waste streams and repurposing them into valuable industrial products, this research not only opens up new market opportunities but also aligns with the growing global emphasis on sustainability and resource efficiency [16].

This study explored the strategic potential of coffee grounds-based epoxy composites, with a particular focus on their market positioning, competitive performance,

and cost structure. The findings from this analysis could have far-reaching implications, offering insights into how waste materials can be transformed into high-value products that support both economic and environmental goals [17, 18].

Experimental Procedure

The strategic analysis of the coffee grounds-epoxy composite aimed at identifying its market potential and positioning using the principles of Blue Ocean Strategy involved several critical steps. The procedure was designed to assess the composite's competitive advantage, market opportunities, and potential for creating uncontested market space.

The first step in the Blue Ocean Strategy analysis involved conducting comprehensive market research to understand the current landscape of industrial coatings. This included identifying and analyzing existing products, their performance characteristics, pricing, and market share. Data was collected through industry reports for the years 2019 to 2024, posted by the four biggest players in the coating industry and market surveys, and conducted using the survey tools from Facebook and Instagram. The surveys obtained a little more than 1,300 responses.

Competitive analysis focused on mapping out key players in the market, their product offerings, and their positioning strategies. Special attention was given to identifying gaps in the market that existing products do not address, such as sustainability, cost-effectiveness, and performance.

To create the coffee grounds-epoxy composite, a detailed comparison was made between the new product and current market offerings. The value curve analysis involved plotting key attributes of the composite, such as mechanical properties, thermal stability, and environmental impact, against those of existing coatings. This graphical representation that can be seen in Fig. 1's highlighted areas where the coffee grounds composite could potentially outperform traditional products.

The analysis aimed to pinpoint unique value propositions offered by the coffee grounds-epoxy composite, such as its eco-friendly nature and potential cost savings from using recycled materials. This step was crucial for understanding how the new composite could stand out in the market and create a differentiated value proposition.

A strategic canvas was developed, as can be seen in Table 1, to visually map the competitive landscape and the relative performance of the coffee grounds-epoxy composite compared to existing products. The strategic canvas included dimensions such as cost, performance, sustainability, and customer satisfaction. By plotting these dimensions, the analysis highlighted where the composite could create new demand and capture untapped market segments.

The strategic canvas helped identify potential "blue oceans"—market spaces where competition is minimal, and there is significant opportunity for growth. This involved evaluating how the coffee grounds-epoxy composite could fulfill unmet needs or offer superior benefits compared to conventional coatings.

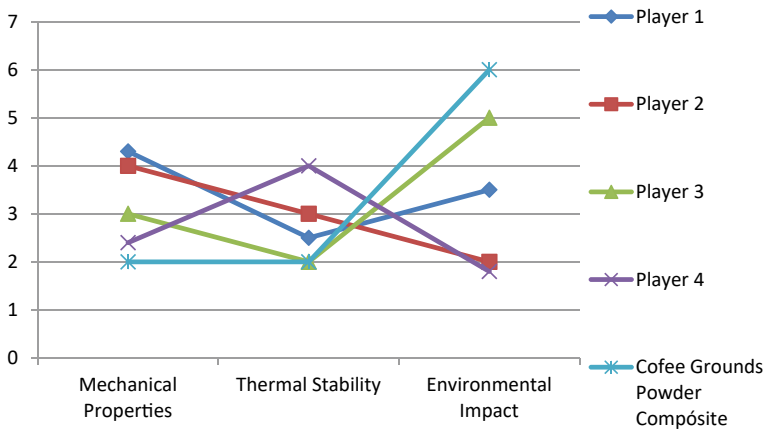

Fig. 1 The value curve analysis

Table 1 Strategic canvas table

Factors	Traditional high-performance coating	Eco-friendly coatings	Coffee grounds-epoxy composite
Cost	Medium–high	High	Medium
Environment impact	Low	HIgh	High
Mechanical performance	High	Medium	Low
Thermal stability	High	Medium	Medium
Market appeal (sustainability)	Low	High	Very high

Based on the insights gained from the market research, value curve analysis, and strategic canvas, a value innovation strategy was formulated. This strategy aimed to align the product's unique strengths with identified market needs to create a new market space. Key elements included proposing pricing strategies that reflect the composite's value while remaining competitive, and outlining marketing approaches that emphasize its sustainability and cost-effectiveness.

Strategic recommendations were developed to guide the positioning of the coffee grounds-epoxy composite in the market. These recommendations included targeted customer segments, potential partnerships, and promotional strategies designed to highlight the composite's unique advantages and capture market share.

Results and Discussion

The market analysis revealed a growing demand for sustainable industrial coatings, driven by increasing environmental regulations and consumer preference for eco-friendly products. Existing coatings primarily emphasize performance and durability but often lack a focus on sustainability and cost-effectiveness. The competitive landscape was characterized by a few dominant players with established products that are high-performing but relatively expensive and resource-intensive.

The coffee grounds-epoxy composite emerged as a unique offering with significant potential to address existing gaps in the market. Specifically, it provided a sustainable alternative by incorporating recycled coffee grounds, which positioned it favorably against traditional coatings that rely on non-renewable resources.

The value curve analysis illustrated that the coffee grounds-epoxy composite could outperform existing coatings in some key areas. While traditional products excelled in performance attributes such as mechanical strength and thermal stability, they generally fell short in terms of environmental impact and cost efficiency. In contrast, the coffee grounds composite offered comparable or superior mechanical and thermal properties while also being more sustainable and potentially more cost-effective due to the use of recycled materials.

The strategic canvas highlighted the composite's strong potential in creating a new market space. By addressing both the performance and sustainability needs of customers, the composite positioned itself in a "blue ocean" where competition was minimal, and there was significant room for growth. The analysis showed that the composite could differentiate itself through its eco-friendly nature and the innovative use of a waste material, thus appealing to a niche market that values sustainability.

The value innovation strategy identified several key opportunities for the coffee grounds-epoxy composite: The composite's use of recycled coffee grounds aligns with the growing demand for environmentally friendly products, offering a compelling value proposition for customers seeking to reduce their carbon footprint. Preliminary cost estimates suggested that the composite could be produced at a lower cost compared to traditional coatings, particularly if scaled up. This potential for cost savings could make it an attractive option for budget-conscious customers. The composite demonstrated competitive performance metrics in terms of mechanical strength and thermal stability, meeting or exceeding the standards set by current market offerings.

The financial analysis indicated that the coffee grounds-epoxy composite could achieve a positive return on investment, driven by its lower production costs and the premium that can be charged for its eco-friendly attributes. A detailed cost breakdown showed that the use of recycled coffee grounds reduced material costs significantly, while the overall production process remained efficient. The revenue potential was bolstered by the increasing consumer and regulatory focus on sustainability, which could drive higher demand for the composite. The financial projections suggested that with effective market positioning and scaling of production, the composite could capture a substantial share of the industrial coatings market.

Conclusion

Based on the analysis, the following strategic recommendations were made: Position the coffee grounds-epoxy composite as a premium sustainable product that offers both environmental benefits and competitive performance. Emphasize its unique selling points in marketing and sales efforts. Focus on industries and sectors with high sustainability requirements or those actively seeking to reduce their environmental impact. Potential target markets include automotive, aerospace, and construction. Explore partnerships with organizations and companies committed to sustainability to enhance credibility and market reach. Collaborations could also help in scaling production and optimizing cost-efficiency.

Overall, the study concluded that the coffee grounds-epoxy composite holds significant potential to create a new market space with minimal competition, offering both economic and environmental benefits. The strategic analysis supported the composite's viability as a sustainable alternative in the industrial coatings market.

In conclusion, the coffee grounds-epoxy composite presents a promising opportunity to disrupt the industrial coatings market by offering a sustainable, cost-effective, and high-performance alternative to traditional products. By leveraging its unique attributes and addressing key market needs, this innovative composite has the potential to drive significant impact and growth in the coatings industry.

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Izod Impact Tests on Epoxy Matrix Composites Reinforced with Montmorillonite Clay Particulate

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Abstract Montmorillonite clay is a material of great interest for research purposes, presenting an interesting economic and technological potential for use in polymer composites. Thus, this study evaluates the impact resistance of an epoxy matrix composite reinforced with montmorillonite clay particulates. Formulations containing 0%, 5%, 10%, and 20% volumetric fraction of montmorillonite clay were developed for Izod impact testing, using standardized test specimens. An increase in impact resistance is expected after the addition of montmorillonite clay particulates compared to the formulation composed only of epoxy.

Keywords Composite · Epoxy · Impact · Sustainability

Introduction

Clays are materials of great scientific relevance due to their physical and chemical properties. They are hydrated aluminum silicates with a layered structure that can be modified and used in various industrial applications. It's primarily composed of SiO_2 and Al_2O_3, along with other elements in smaller proportions, such as iron, magnesium, and calcium [1, 2].

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Clays are divided into families according to the number of tetrahedral layers connected to octahedral layers, representing 1:1 clay minerals where there is one tetrahedral layer and one octahedral layer, and 2:1 clay minerals where there are two tetrahedral layers and one octahedral layer [3].

Montmorillonite clay belongs to the smectite family, with a dioctahedral molecular structure, and its chemical composition is described as $(Al,Mg)_2Si_4O10(OH)_2(H_2O)$, with a negative unit cell charge. One of the most notable characteristics of smectite clays, especially montmorillonite, is their ability to vary the basal spacing, making them popularly known as expansive clays [4].

This feature arises due to an intermediate layer of H_2O present in the molecular structure, which presents an interesting potential for replacing the water with some other material or additive [5].

The clays have notable physical characteristics such as good thermal resistance and typically exhibit high levels of mechanical strength, although with a low degree of deformation. Therefore, it is understood that their use in polymer matrix composites may help enhance various properties which, combined with the best characteristics of polymers, tend to yield more efficient and versatile materials [6].

In the literature, notable works include those by [8, 9], which propose epoxy composites with montmorillonite clay for different applications. Surip and Ismail's study focuses on an adhesive nanocomposite, where tensile strength, impact, and shear resistance were tested. Improvements were observed in all properties, with tensile strength enhanced by clay microparticles, while impact and shear strength improvements occurred with nanoparticles [7].

Meanwhile, Mustapha et al.'s work aimed to evaluate the thermal, mechanical properties, and water absorption of the composite in different formulations, with the clay volume fraction ranging from 0.5 to 2%. The results showed insignificant differences in flexural strength and modulus of elasticity, but a decrease in the composite's impact resistance [8].

Additionally, thermogravimetric analysis indicated improved thermal properties, as the material took longer to decompose compared to the reference. Regarding water absorption, the composite with a higher clay volume fraction absorbed more water compared to the clay-free composite [8].

The aim of this study is, therefore, to evaluate the impact resistance of an epoxy composite reinforced with fine particles of montmorillonite clay, through the Izod energy test, and thus contribute to future analyses of composites utilizing these materials.

Materials and Methods

The montmorillonite clay (Fig. 1a) was obtained from deposits located in the city of Campos dos Goytacazes, RJ, Brazil. The clay underwent a sieving process using a 0.15 mm sieve to collect only the finest particles. After sieving, the clay was dried in an oven at a temperature of 180 °C for 24 h.

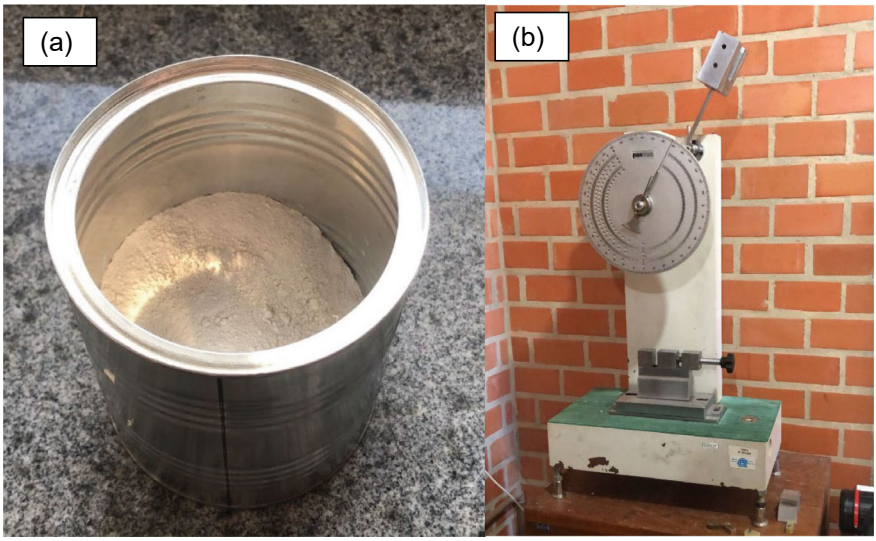


Fig. 1 **a** Clay after sieve (2024); **b** PANTEC XC-50 (2024)

Following this process, the clay was mixed with a commercial epoxy resin of the diglycidyl ether of bisphenol A (DGEBA) type and a diethylene tetramine (DETA) hardener in a stoichiometric ratio corresponding to Phr = 16. The mixture was manually stirred for about 8 min.

Simultaneously with the process of producing the test specimens, a scanning electron microscopy was performed using the Mira equipment from Tescan, available at the Advanced Materials Laboratory (LAMAV) of the State University of Northern Rio de Janeiro Darcy Ribeiro (UENF).

After preparing the test specimens, with approximate dimensions of 10 × 12x60 mm, they were stored to cure at room temperature for 120 h. The specimens were then sanded to improve their shape and surface finish.

Next, notches approximately 2 mm deep were made in the central region of each bar, according to ASTM D256 standards.

Finally, the test was performed using a XC-50 pendulum from the PANTEC brand, available at the LAMAV of the UENF, with a 22 J hammer, as shown in Fig. 1b.

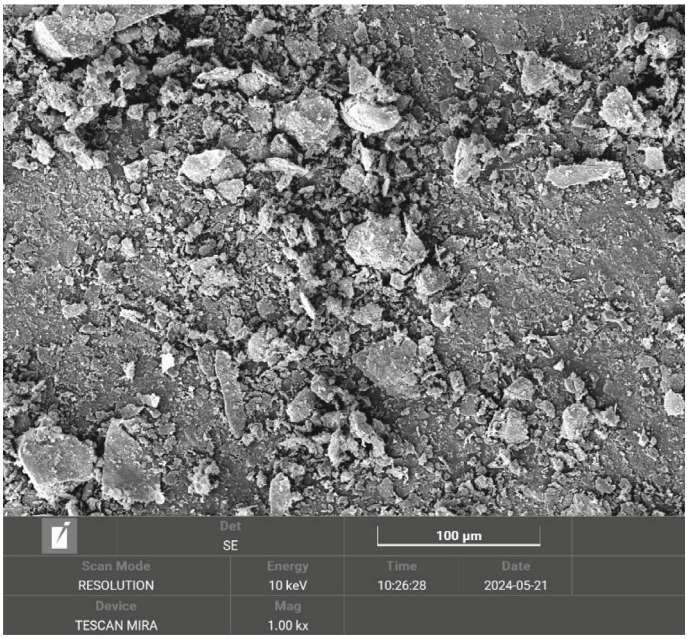


Fig. 2 Scanning Electronic Microscopy with a zoom of 1,000x

Results and Discussion

SEM

The SEM analysis reveals some interesting characteristics of the clay. By applying a 1000 × zoom (Fig. 2), small clay particles can be observed, as well as their polygonal shape.

At 5000 × zoom (Fig. 3), even smaller granules can be seen, while others form larger clusters, bringing together several other grains. In this way, it is possible to identify the behavior of the clay on its own, but also how it may behave within the composite mixture, forming these grain aggregation regions.

Izod Impact Test

In the Izod impact test, both the material's notch resistance and Izod impact resistance were evaluated. Table 1 presents the composite's notch resistance, which shows a decrease in resistance with the inclusion of the clay volume fraction.

This effect can also be observed in Graph 1, where it is possible to analyze that although there was a drop in resistance compared to the reference, the increase in the

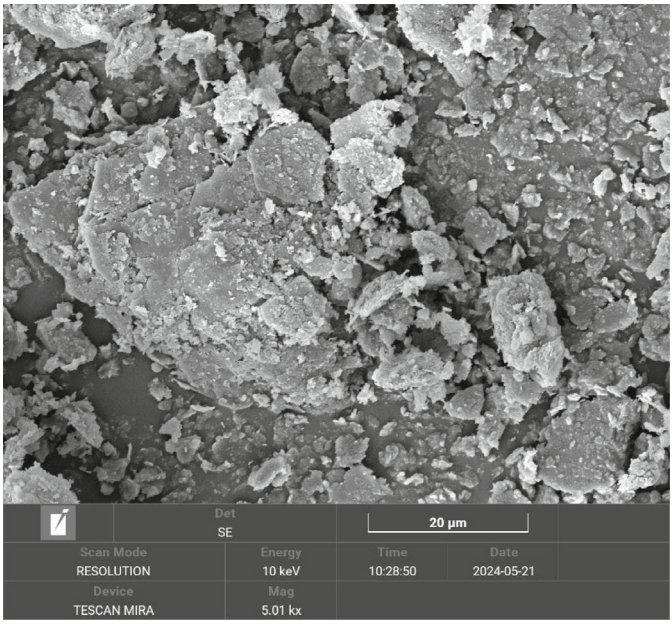


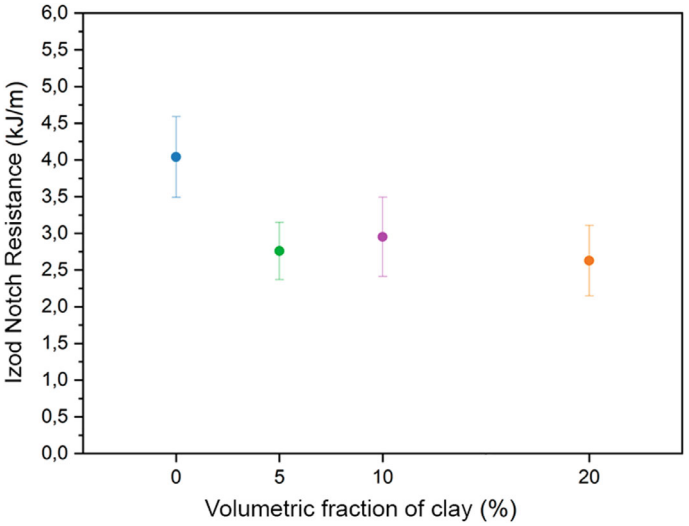
Fig. 3 Scanning Electronic Microscopy with a zoom of 5,000x

Table 1 Notch resistance from the composites

Volumetric fraction of clay (%)	Energy (kJ/m)
0	4.04 ± 0.55
5	2.73 ± 0.29
10	2.84 ± 0.41
20	2.62 ± 0.35

volume fraction did not show a trend of further decreasing resistance. This suggests that even increasing beyond the 20% proposed in this study, the resistance may remain stable. This reduction should be analyzed concerning the intended applications to determine whether the composite is viable for use.

Table 2 presents the Izod impact resistance, with results following the same trend as the notch resistance, although the decrease in values is less pronounced.



Graph 1 Izod notch impact resistance

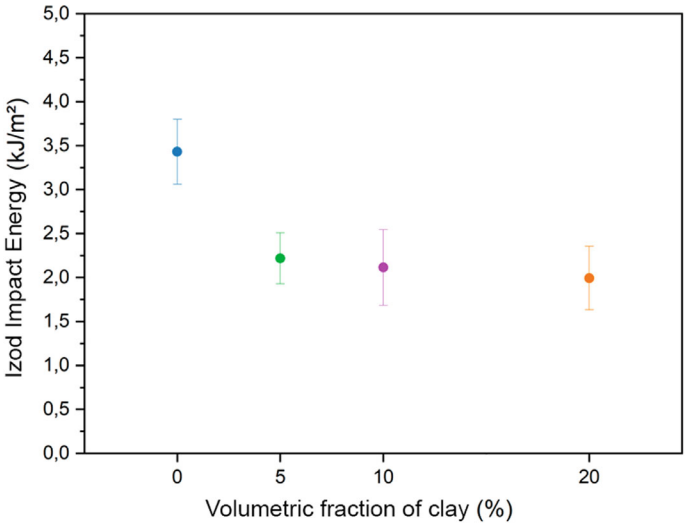
Table 2 Izod impact resistance from the composites

Volumetric fraction of clay (%)	Energy (kJ/m^2)
0	3.43 ± 0.37
5	2.47 ± 0.39
10	2.33 ± 0.54
20	2.09 ± 0.48

Similarly, Graph 2 supports the analyses mentioned above.

Compared to the research mentioned earlier, the decrease in values is also observed in the work of [9], but it differs significantly from that presented by [8]. This difference can be explained by the processing method used, where Surip and Ismail achieved improvements for a specific application using nanoparticles.

Thus, particle size and even mixing time may have had a negative effect on the material, which raises the need for further tests to determine if there would indeed be a difference by changing these factors.



Graph 2 Izod impact resistance

Conclusion

This research, therefore, presents the following conclusions:

- There was a decrease in resistance with the inclusion of clay, but the volume fraction did not significantly affect these values, suggesting that further studies can explore increasing the volume fraction.
- Particle size and the processing method can be adjusted to improve the obtained results.
- Further studies are necessary to assess the material's viability for each specific application.

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Structure and Properties of Two Natural Fibers from South America

Henry A. Colorado, Marc Meyers, and Sergio Neves Monteiro

Abstract Natural vegetable fibers used as reinforcements are increasingly used in polymer and cementitious matrices because of their main advantages when compared with the traditional engineered fibers: biodegradable, low carbon footprint, low cost, lightweight, and tough. This research presents results on some of the most representing fibers used in the region, some of them for centuries by local communities, from its mechanical properties to its chemical and microstructural features. Scanning electron microscopy and flexural strength results are presented. Information regarding the plants and in some cases their cultural use is presented as well. Properties confirm these fibers can substitute high-performance engineering fibers, in applications that include composites for vehicles, bullet protection vests, and green construction materials.

Keywords Natural fibers · Composites · Characterization

Introduction

Natural vegetable fibers have been used extensively for millennia worldwide by very separated and distinct communities [1]. From houses, armor, hunting, crafts, and daily products, indigenous peoples and other groups in Africa, Asia, and America mastered the use of them [2]. Particularly in South America, where there are still

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contacted and non-contacted indigenous communities living in the Amazon rainforest [3], it is possible to learn from them not only about the use of these fibers, but also about unknown fibers to us. Amazon rainforest and its area of influence, covering an area of about 6.7 million km^2 in several South American countries (Brazil, Bolivia, Peru, Ecuador, Colombia, Venezuela, Guyana, and Suriname), is the world's largest tropical rainforest, known to be a source of oxygen, fresh water, and biodiversity [4]. The Colombian Amazon covers approximately 40,300 km^2, which is around 35% of the country's total area of 1,142,000 km^2. This region includes the departments of Amazonas, Caquetá, Guainía, Guaviare, Putumayo, and Vaupés. Due to a reduction in conflict involving guerrilla groups and others, the Amazon is now open for exploration by tourists and scientists. This offers an opportunity to gain valuable insights into indigenous communities, whose knowledge is important to preserve and to educate people on sustainability and other important related areas [5].

Close to this area, the Andes Mountain range is also a source of diversity [6], covering areas from Venezuela in the north; the range passes through Colombia, Ecuador, Peru, Bolivia, Argentina, and Chile. These two regions in Colombia cover most of the territory and represent a huge natural source of diversity as well, food, fresh water, and the economy of the country. Thus, the number of species to investigate from the engineering point of view is huge, which can be filtered with the important knowledge from the local communities, the real experts on their uses and ecosystems [7].

In addition to the mentioned local applications, natural fibers are used in many sustainable products, which includes ballistic protection [8], composites for building and infrastructure [9, 10], automobile industry [11], and arts and decoration [12], among others. The fibers investigated in this research were selected from the Amazon rainforest and also from the Andes Mountain range, both in Colombia. From the Amazonia region, *Leopoldina piassaba* (locally known as Chiqui-chiqui) [13], used mainly in roofs and crafts by local indigenous communities, was investigated. Also, from the Andes Mountain range, Fique, *Furcraea cabuya* [14], a species of plant in the family *Asparagaceae*, was selected. Fique has been used for centuries also by indigenous peoples but modernly by farmers. These two fibers were also combined with epoxy resin to show the potential for engineering.

Materials and Methods

The samples collected for this research were obtained in Colombia from two very different regions, Amazonia, the rainforest influenced by the Amazon River, and the Andes Mountains range, both huge nature colossi involving many countries in South America. Figure 1a shows the extension of the Amazon rainforest on the top of the continent, while the Andes is on the left side. Figure 1b shows both areas in Colombia, with the squares in yellow showing the collection areas of Piassaba fiber in the forest, and the white square the collection area on the Andes for the fique plant.

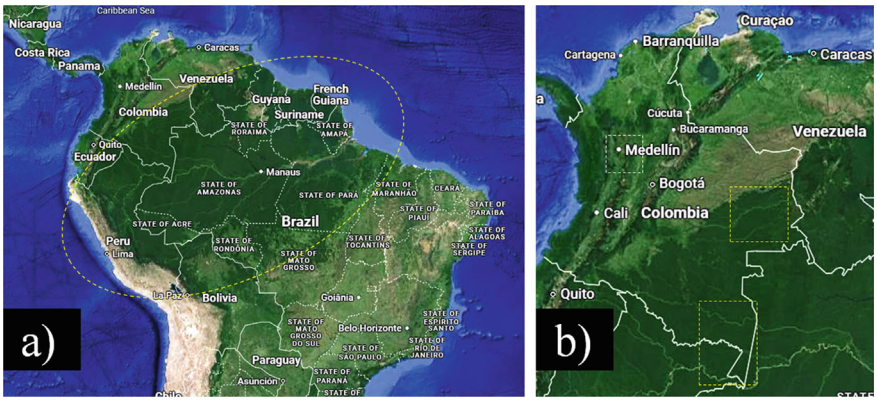

Fig. 1 **a** Amazon rainforest in different countries; **b** areas in Colombia where fibers were collected

Local communities process these two fibers for diverse products as mentioned above, so the fibers were directly purchased to a local Community in the jungle. These fibers were characterized with scanning electron microscopy (SEM), for which samples were sputtered in a Hummer 6.2 system (15 mA AC for 30 s) to obtain a thin film of gold necessary to be observable in the microscope, a JEOL JSM 6490LV used in high vacuum mode. In addition, composites with epoxy resin of 10 wt% of fibers were made manually, in order to test the flexural strength and compare its properties, aiming new application for both fibers.

Results

Figure 2a shows the Piassaba palm in the Amazonia region of Colombia. The fibers can be seen in the plan stem. Some of the most common applications are roofs for local houses and very different crafts, Fig. 2b and c respectively. The area corresponds to the Department of Guanía in Colombia, near the Inírida municipality, very humid and warm area mainly due to its low altitude (less than 100masl). The fibers of piassaba are dark and strong, ideal for structural applications. Because of this, these fibers are also used extensively in Colombia as brooms for sweeping the floor, a very typical product in homes, mostly because they are inexpensive, and resistant to abrasion and high strength. Today, this product has been more appreciated as it is 100% biodegradable and since it is made of 100% natural fibers (in combination with a wooden handle), and it is very environmentally friendly [15].

Figure 3a shows a Fique plant from an area near Medellin, a city located in the Anders Mountain range of Colombia. Samples were collected from plants located at 1900masl, in an area with a lot of rains and temperatures relatively low for Colombia, from around 15 to 20 °C. Some applications are also shown, a typical 50 kg fique bag, a rope, and a resin matrix composite fabricated; see Fig. 3b, c, and d respectively.

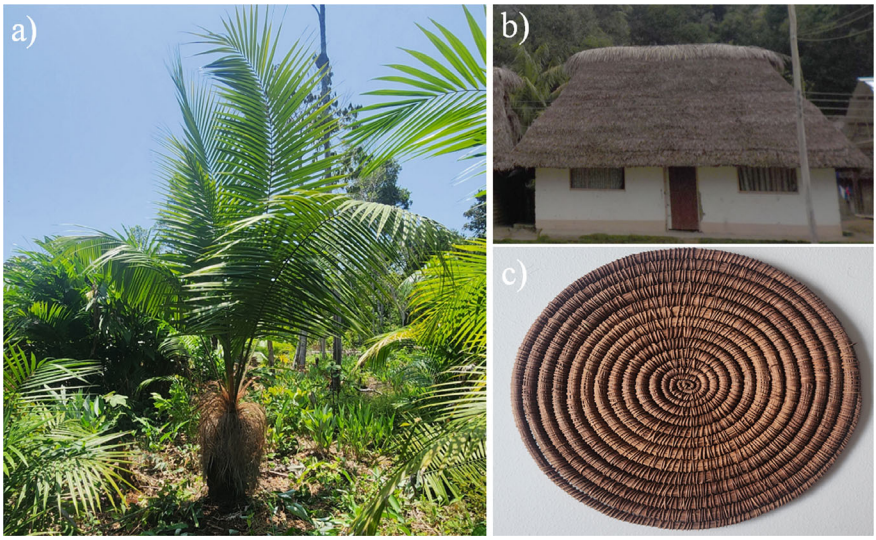

Fig. 2 **a** Piassaba palm and its most common applications, **b** houses, and **c** crafts

The fique fibers are located inside the leaves, placed along them, and extracted in an elaborate artisanal procedure.

Figure 4 shows SEM images for the piassaba fibers. Figure 4a is a lateral view of a fiber, where the small dots correspond to tiny silica particles inserted in the fiber surface, which clearly explain why the fibers have been used for centuries in brooms for sweeping the house or street, a very abrasive use. Figure 4b shows a detail of the complex surface microstructure, magnified in Fig. 4c, showing the silica particle, plan tissue, and spicules. The silica particle shows a radial symmetry like a flower.

Figure 5 shows SEM images for the fique fibers. Figure 5a is a cross-section view of the fiber, showing a clear cellular structure, which explains that this fiber is very flexible and light, ideal for many applications that combine high strength and deformation, such as packaging and armor. Figure 5b shows details of the microstructure, magnified in Fig. 5c to show the wall structure.

Figure 6 shows a summary of the flexural tests for the natural fiber composites, compared with respect to the neat epoxy resin. Clearly the fibers do not weaken the resin. These results are important for several reasons. First, 10wt% of fibers reduce the amount of epoxy resin, a material expensive both economically and environmentally. Therefore contributing to sustainability of the planet. Second, the composite properties are better than those to the pure resin, which via optimization and large-scale manufacturing can be improved further for products. Moreover, fique fiber did great, much better than piassaba, which is expected as the fibers used came from a selected and mastered process polished by years by farmers. This means that piassaba fibers and results can also be improved via fiber extraction and selection.

As a future work, these fibers can be optimized in higher concentrations for structural applications aiming lower composite costs, light weight, and better strength,

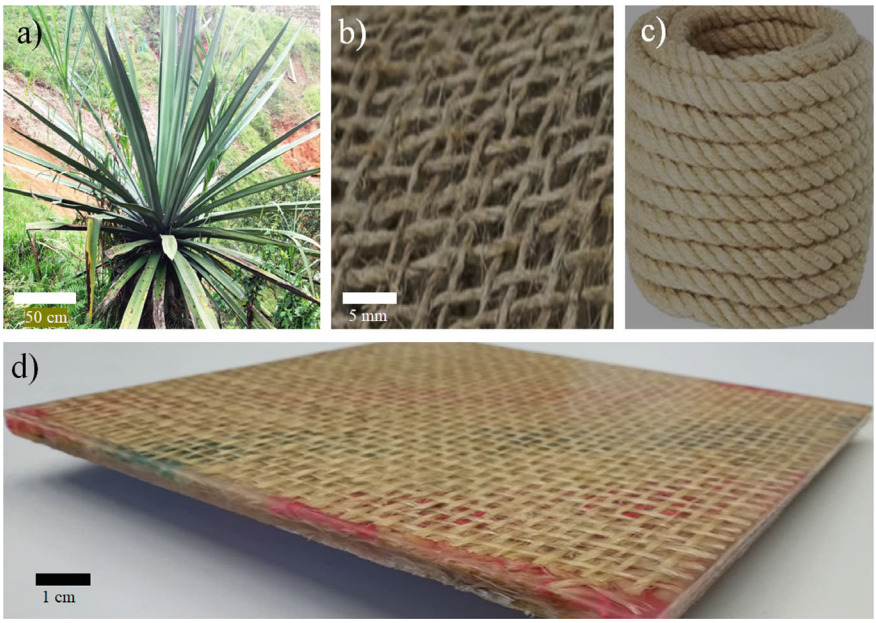

Fig. 3 **a** Fique plant and some applications, **b** fique bag, **c** ropes, and **d** composites

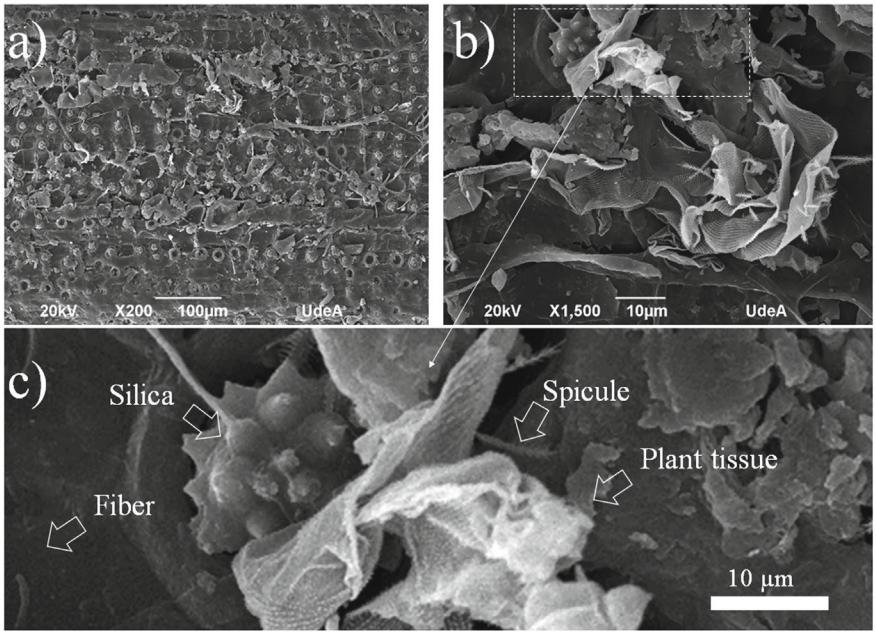

Fig. 4 SEM images corresponding to piassaba fibers: **a** side view, **b** microstructure, and **c** detail at higher magnification

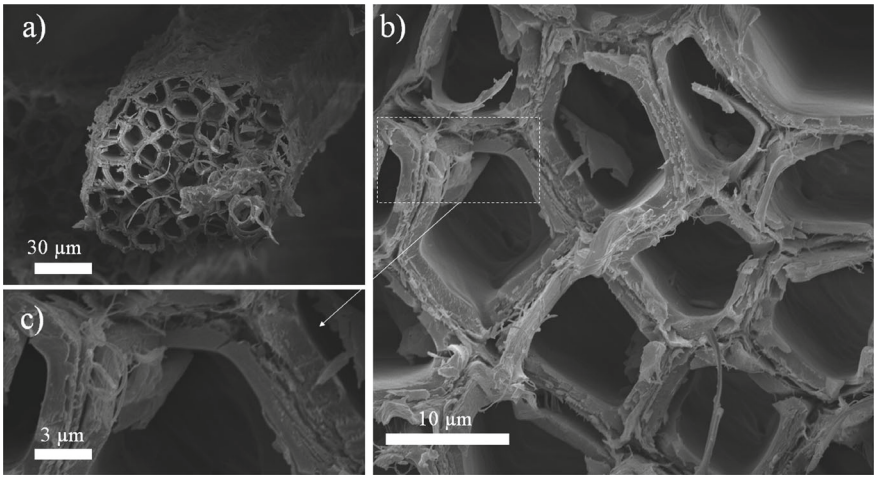

Fig. 5 SEM images corresponding to fique fibers: **a** cross-section view, **b** microstructure, and **c** detail at higher magnification

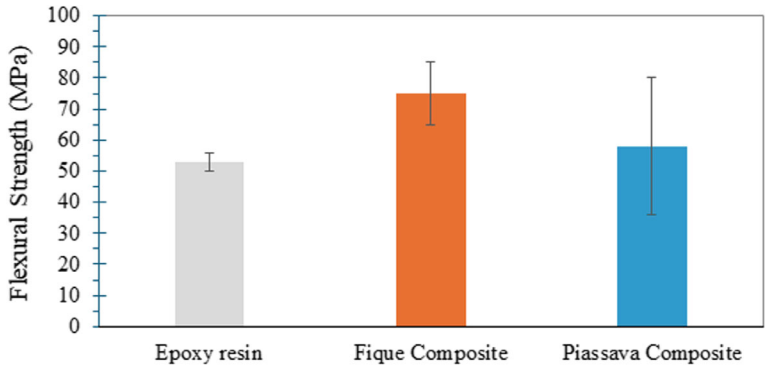

Fig. 6 Flexural strength of the composites made with fibers and epoxy resin

ductility, or deformation at break for energy absorption applications [16], or functional materials [17] via functionalization or additive manufacturing of polymer matrix vegetable fiber composites using filament makers for fused deposition modeling [18, 19] or VAT photopolymerization [20] techniques. A life-cycle analysis is also necessary for making a full balance of the process.

Summary

The current work has presented results regarding the use of two vegetable fibers from Colombia feasible to create an impact in the composite industry, one from the Amazon region, *chiqui-chiqui* fibers (*Leopoldina piassaba*), and another one from the Andes Mountain range, *fique* fibers (*Furcraea cabuya*). Both fibers showed a very distinctive microstructure, from a surface with tiny silica powders inserted into the plant tissue for piassaba, to a very light cellular structure for fique. The flexural results showed that both fibers can be used as structural material, either to decrease epoxy resin costs, to improve strength, or to use a nature material for a new application.

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Utilization of FGD Gypsum in Epoxy Composites: A Flexural Strength Evaluation

J. F. S. Souza, D. C. R. Velasco, J. L. D. C. Lirio, D. Souza, F. P. D. Lopes, and C. M. F. Vieira

Abstract The FGD (Flue Gas Desulfurization) gypsum, a byproduct of the desulfurization process of combustion gases in thermal power plants, is an example of industrial waste that can pose significant public health risks if not managed properly. In this regard, the aim of this study is to determine the influence of incorporating FGD gypsum on the flexural strength of epoxy matrix composites, aiming to generate an adequate disposal of waste and/or technological gains. Different formulations of waste were incorporated into a Diglycidyl ether of bisphenol A/Diethylenetriamine (DGEBA/DETA) matrix in varying proportions: 0, 10, 20, and 30% by volume. The flexural test was carried out following the guidelines of ASTM D790, the results being analyzed using an analysis of variance and Tukey test. Through this work, it is possible to observe the influence of FGD gypsum on flexural resistance, as well as the technological feasibility of its use.

Keywords Composites · Sustainability · Mechanical properties

Introduction

Due to growing concerns about public health and social well-being, the proper management of industrial waste has become a critical issue. The increasing demand, driven by population growth, leads to the expansion of industrial sectors [1]. According to Liu, sustainable supply chain management is undoubtedly a crucial

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topic in the twenty-first century [2]. The accelerated growth of industrial production has been one of the main drivers of global economic development. However, this progress is accompanied by a significant increase in industrial waste generation, which poses a major environmental challenge. It is projected that urban solid waste generation will increase from 2.3 billion tons in 2023 to 3.8 billion tons by 2050 [3].

The generation of industrial waste in the steel industry is a matter of great importance due to the significant environmental impact of this sector. Despite its relevance to the global economy, steelmaking is responsible for generating a large volume of waste in its production chain. In pursuit of sustainable development, the construction industry absorbs steelmaking waste by providing alternative raw materials and reducing the extraction of natural resources [4]. The environmental effects of this activity range from excessive consumption of natural resources to the generation of waste and pollutant emissions [5]. The construction industry is responsible for up to 50% of the total solid waste generated in Brazil, according to data from the study "Panorama dos Resíduos Sólidos no Brasil" conducted by the Brazilian Association of Public Cleaning and Special Waste [6].

In the thermoelectric industry, due to the flue gas desulfurization process during coal combustion, synthetic gypsum (FGD gypsum; Flue Gas Desulfurization) is generated as a byproduct [7]. The reuse of this material aligns with the principles of sustainable development, which refers to development capable of meeting the needs of the present generation without compromising the ability of future generations to meet their own needs [8].

The creation of composites with an epoxy matrix and industrial waste represents a promising approach for sustainable management. High-performance materials are designed by manipulating potential constituents and optimizing their arrangement [9]. Environmental issues highlight the need for a sustainable relationship between technological development and environmental conservation [10].

Flue gas desulfurization (FGD) gypsum is a byproduct of coal burning. This material is generated during the process of purifying flue gases to remove sulfur dioxide emissions, which contribute to acid rain and other environmental problems [11]. Addressing environmental pollution caused by the accumulation of FGD gypsum contributes to improving green and sustainable development [12].

Currently, composites belong to the fastest-growing class of materials, as they offer a wide range of applicable properties that can be "selected" by designers according to their needs [13]. The combination of an epoxy matrix with FGD gypsum not only contributes to reducing industrial waste but can also result in composites with enhanced mechanical properties. In recent decades, the development of these new technologies has brought greater safety, cost-effectiveness, and functionality [14]. Studies have shown that these composites can exhibit high strength and durability, making them a viable alternative to traditional materials [15–17].

The objective of this work is to investigate the mechanical properties of composites reinforced with FGD gypsum in an epoxy matrix. The choice of epoxy resin is highlighted due to its high performance, attributed to properties such as chemical resistance, good thermal properties, high adhesion strength to various substrates, good electrical insulation, low shrinkage during curing, ease of processing, and good

mechanical properties [18, 19]. Additionally, the work seeks to explore potential applications of these composites in the construction industry and other sectors. To achieve these goals, a bending strength test will be conducted. This test aims to assess a material's ability to withstand bending and deformation without fracturing or failing [20]. Technological feasibility will be evaluated based on the performance gains of materials that would otherwise be discarded.

Materials and Methods

The epoxy resin system used was based on Bisphenol A diglycidyl ether (DGEBA), combined with a curing agent, diethylenetriamine (DETA), at a ratio of 16 phr. Both the resin and the curing agent were supplied by Avipol, under the product codes SQ1005 and SQ3131, respectively, and were manufactured by Silaex.

The waste used in this study is FGD gypsum (Flue Gas Desulfurization), a byproduct of the desulfurization process of gases from coal combustion. The gypsum was supplied by ArcellorMittal Serra, Brazil, with a particle size smaller than 0.075 mm. Before use, the material was sieved to ensure homogeneity and then dried.

The formulations were evaluated with 0, 10, 20, and 30% by volume of FGD. For each composition, seven samples were created, cured over five days, and then sanded to conform to testing specifications. To optimize the properties of the materials, once cured, the test specimens underwent a post-curing process at 70 °C for 3 h.

The flexural test was conducted using an INSTRON universal testing machine, model 5582, located at the State University of Northern Rio de Janeiro in Campos de Goytacazes, RJ, following the ASTM D790 standard [21]. For each formulation, 5 test specimens were produced, with 12.5 mm in width, 10 mm in height, and 63 mm in length. The test span was 50 mm, and the test speed was 0.5 mm/min.

Based on ASTM D790, the elastic modulus is calculated in the material's elastic region, where the behavior follows a linear pattern according to Hooke's Law. Figure 1 illustrates the typical stress–strain curve, highlighting the elastic region. The initial region AC does not represent the material's properties, being an artifact caused by specimen adjustment. For materials with linear (Hookean) behavior, the linear portion of the curve (CD) is extended to the zero-stress axis, defining point B as the corrected zero-strain point. The elastic modulus is then calculated by dividing the stress by the strain from this corrected zero point. This value provides a measure of the material's stiffness while it is still in the elastic phase, without undergoing permanent deformation [21].

The results were evaluated through an analysis of variance (ANOVA), following the methodology described by Velasco et al. [22], with a significance level set at 5%.

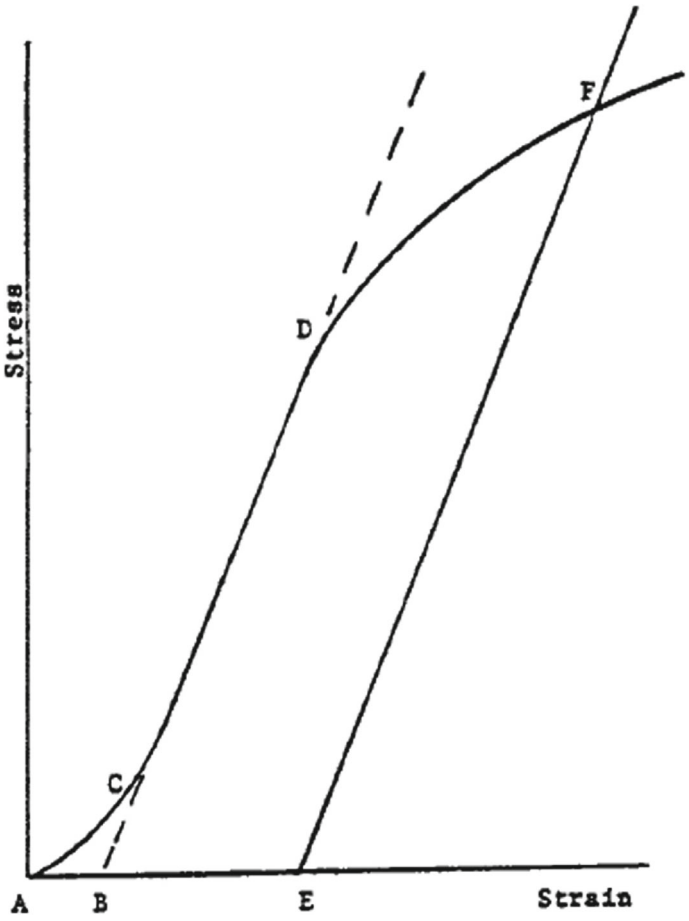

Fig. 1 Typical stress–strain curve [21]

Results and Discussion

Figure 2 shows the samples as they were removed from the molds (before the finishing and testing process). It is possible to observe that the insertion of particulates has generated a greater presence of manufacturing defects, especially in the formulation with 30%, which is a normal behavior predicted in the literature [23].

The elastic modulus of the formulations evaluated can be seen in Fig. 3. In this figure, an increase in the modulus of elasticity can be observed as a function of the amount of FGD added to the epoxy system, this behavior being in line with the literature [24].

Table 1 shows that the increase of elastic modulus is significant in all formulations, with an increase of up to 57% possible. This can be explained by the incorporation

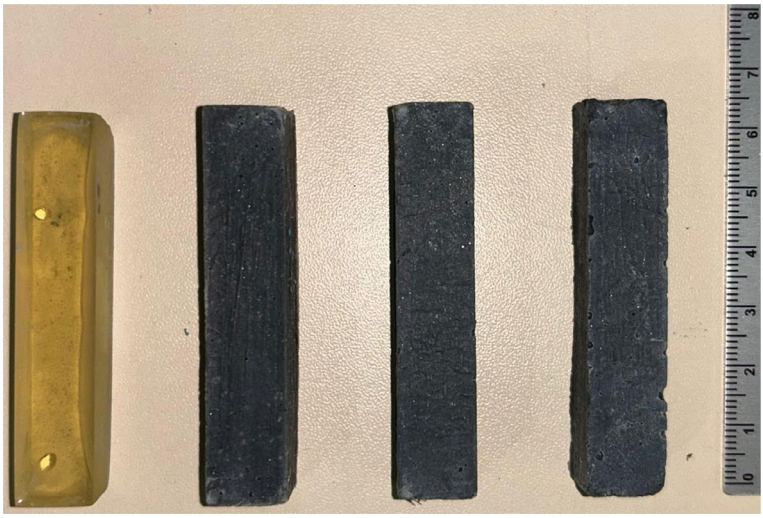

Fig. 2 Samples after demolding

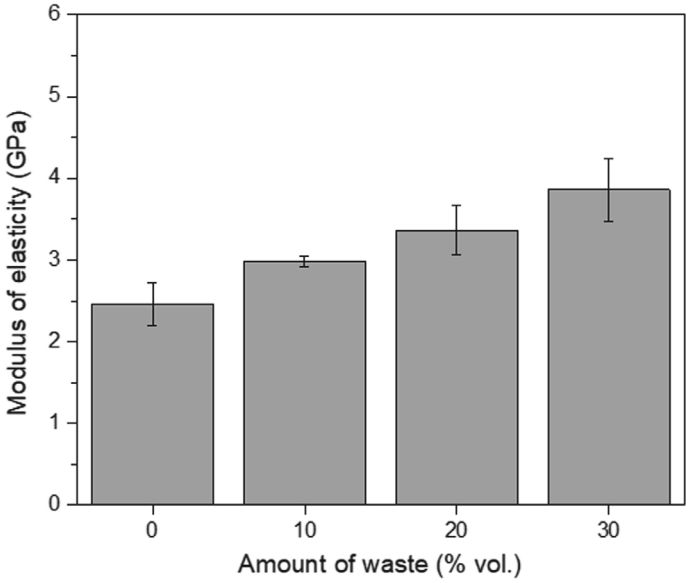

Fig. 3 Elastic modulus x amount of waste

of a stiffer material, which enhances the composite's rigidity by partially replacing a less rigid material and reduces the mobility of the remaining polymer chains [25].

Figure 4 shows that the flexural strength reduces with addition of particulates. This loss of performance is significant according to the analysis of variance, Table 2. This behavior is in line with that observed in several studies that insert particulates, and this is attributed to the particulates contributing to the insertion of defects that act as stress concentrators, being especially significant in applications such as tensile and flexural stress [23, 26, 27].

Although the observed loss of strength is not desirable, the observed values are still adequate for several applications, such as flooring and countertops, which require greater compressive strength (which tends to be less influenced by stress concentrators than tensile strength) [28, 29]. For such applications, the addition of waste can be beneficial from a financial and ecological point of view [30–32].

Table 1 Elastic modulus increase and ANOVA analysis relative to epoxy system

Waste amount (% vol.)	Elastic modulus (GPa)	Increase (%)	P-value (%)
0	2.46 ± 0.26	–	–
10	2.98 ± 0.07	+21%	0.2
20	3.36 ± 0.30	+37%	0.1
30	3.86 ± 0.38	+57%	0.0

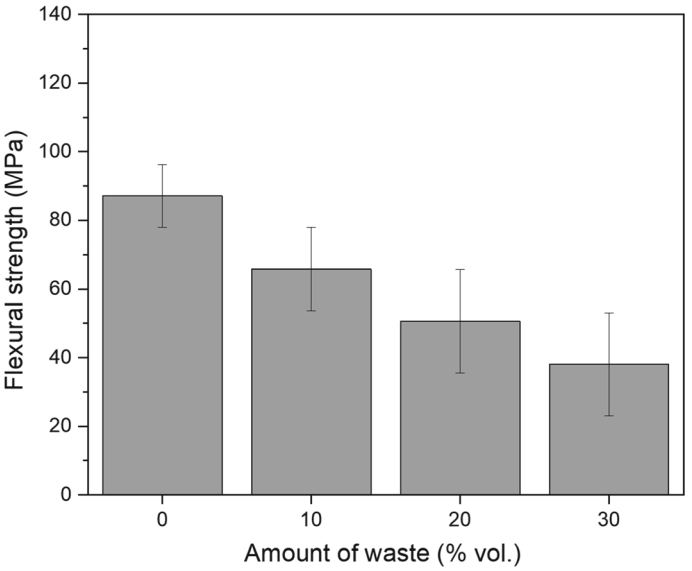

Fig. 4 Flexural strength x amount of waste

Table 2 Flexural strength and ANOVA in relation to the epoxy system

Waste amount (% vol.)	Flexural strength (MPa)	Decrease (%)	P-value (%)
0	87.15	–	–
10	65.86	24%	1.4%
20	50.58	42%	0.2%
30	38.05	56%	0.0%

Conclusions

The findings of this study indicate that the incorporation of FGD into the epoxy matrix leads to a significant enhancement of the modulus of elasticity, with increases of up to 57%. However, this improvement in stiffness is accompanied by a reduction in flexural strength. The introduction of particulates induces defects that act as stress concentrators, particularly under tensile and flexural loading conditions. Despite this decline in mechanical performance, the remaining flexural strength values are still within acceptable limits for applications such as flooring and countertops, where compressive strength is a more critical factor and is less affected by stress concentrators. Consequently, the addition of FGD in these composites represents a viable alternative, providing an optimized balance between mechanical properties, cost-efficiency, and environmental sustainability.

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Subject Index

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