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APLICAÇÃO NEGRO DE FUMO PIROLISADO PARA A FORMAÇÃO DE COMPÓSITOS POLIMÉRICOS COM FIBRAS VEGETAIS E AVALIAÇÃO DE SUAS PROPRIEDADES MECÂNICAS

PYROLYZED CARBON BLACK APPLICATION FOR THE FORMATION OF POLYMERIC COMPOSITES WITH VEGETABLE FIBERS AND EVALUATION OF THEIR MECHANICAL PROPERTIES

APLICACIÓN DE NEGRO DE HUMO PIROLIZADO PARA LA FORMACIÓN DE COMPUESTOS POLIMÉRICOS CON FIBRAS VEGETALES Y EVALUACIÓN DE SUS PROPIEDADES MECÁNICAS

Renato Reis Monteiro
Doutor, Universidade Estadual de Santa Cruz, Brasil
E-mail: rrmonteiro@uesc.br

Celso Carlino Maria Fornari Junior
Doutor, Universidade Estadual de Santa Cruz, Brasil
E-mail: celso@uesc.br

RESUMO

As fibras vegetais apresentam-se como alternativas promissoras para o desenvolvimento de materiais compósitos sustentáveis, devido à sua abundância, baixo custo, biodegradabilidade e propriedades mecânicas relevantes. No entanto, a eficiência desses materiais está diretamente relacionada à qualidade da interface fibra/matriz, que ainda representa um desafio significativo na engenharia de compósitos. Neste contexto, o presente trabalho investiga a incorporação de negro de fumo pirolisado em uma matriz de resina poliéster isoftálica, com o objetivo de avaliar sua influência na interação interfacial e nas propriedades mecânicas de compósitos reforçados com fibras de sisal e coco. Considera-se que a polaridade dos constituintes e os mecanismos de ancoragem mecânica desempenham papel fundamental na adesão interfacial, influenciando diretamente o desempenho estrutural do material. As fibras foram previamente tratadas em solução alcalina de hidróxido de sódio (NaOH), visando à remoção de impurezas superficiais e à modificação de sua estrutura morfológica. Os compósitos foram produzidos com e sem a adição de 1% em massa de negro de fumo, sendo posteriormente submetidos a ensaios mecânicos de flexão. Os resultados indicaram que a incorporação das fibras promoveu melhorias significativas nas propriedades mecânicas em relação à resina pura. Adicionalmente, a presença de negro de fumo contribuiu para o aumento do módulo de elasticidade, da resistência à flexão e da energia absorvida até a ruptura, especialmente nos compósitos reforçados com fibras de coco. Esses resultados sugerem que o negro de fumo atua como agente de reforço e modificador interfacial, desde que adequadamente disperso na matriz polimérica.

Palavras-chave: Sisal, Coco, Compósitos poliméricos.

ABSTRACT

Vegetable fibers have emerged as promising alternatives for the development of sustainable composite materials due to their abundance, low cost, biodegradability, and relevant mechanical properties. However, the efficiency of these materials is directly related to the quality of the fiber/matrix interface, which still represents a significant challenge in composite engineering. In this context, the present study investigates the incorporation of pyrolyzed carbon black into an isophthalic polyester resin matrix, aiming

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to evaluate its influence on interfacial interaction and on the mechanical properties of composites reinforced with sisal and coconut fibers. It is considered that the polarity of the constituents and the mechanisms of mechanical anchoring play a fundamental role in interfacial adhesion, directly influencing the structural performance of the material. The fibers were previously treated in an alkaline solution of sodium hydroxide (NaOH), aiming at the removal of surface impurities and modification of their morphological structure. The composites were produced with and without the addition of 1 wt% carbon black and were subsequently subjected to flexural mechanical tests. The results indicated that the incorporation of fibers promoted significant improvements in mechanical properties compared to pure resin. Additionally, the presence of carbon black contributed to an increase in the modulus of elasticity, flexural strength, and energy absorbed until fracture, especially in composites reinforced with coconut fibers. These results suggest that carbon black acts as a reinforcing agent and interfacial modifier, provided it is properly dispersed within the polymer matrix.

Keywords: Sisal, Coconut, Polymeric composites.

1. Introduction

The use of plant fibers in the development of functional composites constitutes a promising alternative for the manufactured goods industry, mainly due to the environmentally appropriate supply of raw materials derived from renewable production cycles. The search for new sources of raw materials capable of meeting the growing demand of the industrial sector is a concern for both engineering and public policy makers. It is estimated that, by 2050, the world population will reach approximately 10 billion inhabitants, imposing significant challenges on production organization and on the sustainability of the current production–consumption model (Gazzoni, 2017; Macedo and Júnior, 2017).

In this context, the use of plant fibers in the development of new materials for engineering applications represents a promising alternative, both due to the availability of renewable raw materials and their low cost, as well as the reduced environmental impact associated with their production. Fibers of plant origin present characteristics that allow their physical and structural modification, enabling the production of ropes, yarns, woven structures, and fabrics for different applications (Forcelini et al., 2025).

However, the efficiency of these materials is directly related to the quality of the fiber/matrix interface, which still represents a significant challenge in composite engineering. In this context, the present study investigates the incorporation of pyrolyzed carbon black into an isophthalic polyester resin matrix, aiming to evaluate its influence on the interfacial interaction and on the mechanical properties of composites reinforced with sisal and coconut fibers.

2. Literature Review

Fillers are commonly added to polymers in order to improve specific properties, applications, and processing costs. Carbon black is a widely used filler in polymeric materials due to the extremely small size of its particles, which results in a very high surface area. A primary particle with a diameter of 50 nm has an area of 7.9×10^{-15} m², corresponding to approximately 8.244 m²/g. As a consequence, carbon black exhibits high reactivity and adsorption capacity. More graphitic carbon black particles present a moderate surface energy ranging from 20 to 40 mJ/m², with a strong tendency toward agglomeration. These particles are composed of carbon, which presents chemical affinity with most polymeric matrices. Carbon black acts as a stabilizing agent against UV-vis radiation and weathering for a wide range of thermoplastic materials. Furthermore, carbon black can generate significant effects, especially within the matrix and at the fiber/matrix interface when positioned between the phases. It acts as a nano/micrometric particulate filler, a matrix reinforcing agent, and a potential interfacial modifier, in addition to increasing the effective roughness of the matrix and reducing interfacial failures.

Carbon black particles are obtained through the pyrolysis of aromatic oils, forming a family of products with different combinations of particle size and structural arrangement. The presence of carbon black improves properties such as morphology, rheology, and mechanical performance (Sarafpour; Pircheraghi, 2024). In the rubber industry, black carbon is widely employed because it improves mechanical properties and wear resistance, especially in tire manufacturing. The high surface area exhibited by carbon black provides highly effective behavior in most polymers, since it allows greater contact and chemical interaction with the polymeric matrix (Jakab; Blazsó, 2002). Several studies have been conducted on the production of composites reinforced with plant fibers from different sources such as banana, coconut, ramie, jute, flax, sisal, sugarcane, among others, demonstrating that these fibers can act as reinforcing fillers, improving the properties of the composites (Venkatarajan; Athijayamani, 2021).

However, plant fibers present some disadvantages when compared to synthetic fibers, such as lower thermal stability, lower impact resistance, limited addition content, and difficulties related to interaction with the polymeric matrix, especially regarding

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fiber/matrix interfacial coupling. In addition, they exhibit high water absorption and incompatibility with certain classes of polymers (Fornari, Monteiro, 2025). For these reasons, the industrial sector has not yet devoted adequate attention to large-scale applications using cellulosic fiber composites, which limits the broader use of these materials (Baghaei; Skrifvars, 2020; Shah, 2013). In order to achieve strong interaction between the fiber and the polymeric matrix, the resulting forces between both materials must be compatible so that mutual interaction occurs, forming a balance of attractive electromagnetic charges. In the case of plant fibers, they contain a considerable amount of lignin and cellulose, in which hydroxyl groups are arranged in groups of three for each glucose ring. This gives the fiber a polar character and may promote strong interaction bonding sites due to the high polarity of the group. On the other hand, the physical and morphological characteristics of the fibers may also act as negative factors in the interaction with the polymeric matrix, either due to the external coating present on the fiber or because of defect sites (Gamstedt, 2023). The combination of plant fibers with the polymeric matrix may generate a variety of different functions depending on the type of matrix and the condition of the fibers.

The formation of plant fibers begins with the organization of cellulose chains, in which sets of macromolecules associate to form the first nanofibers. These macromolecules align predominantly in the longitudinal direction, intertwining at specific angles that vary according to the plant species. Subsequently, lower molar mass molecules, such as hemicellulose, position themselves between these nanofibers, maintaining their spacing and preventing structural collapse. As larger groups of nanofibers become organized, lignin acts in the packing and protection of these regions, sealing the fibrous structure. The aggregation of these structures results in the formation of microfibers, which in turn give rise to cellulose macrofibers (Fornari Junior, 2017).

This fibrous structure is composed of repetitive anhydro-D-glucose units, responsible for providing potential chemical sites for interaction with other materials. However, the high polarity of the cellulosic structure, associated with the abundant presence of hydroxyl groups, gives the fiber a highly hydrophilic character, which may hinder compatibility with polymeric matrices that are generally hydrophobic (Dallocchio et al., 2023). This polarity mismatch represents a limiting factor for the formation of

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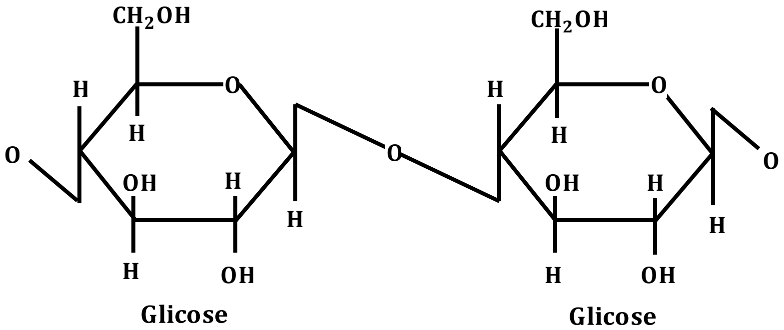
efficient interfacial bonds, compromising the mechanical performance of the resulting composites (Li et al., 2015). The large number of hydroxyl groups present in each glucose ring highlights the density of polar sites in cellulose. Figure 1 presents the planar structural formula of the cellulose molecule.

Figure 1 – Representation of the cellulose molecule containing two glucose rings



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Chemical interactions between materials are governed by attractive electromagnetic forces, which are responsible for the formation of effective chemical bonds between elements. Electronegativity, defined as the ability of an atom to attract electrons from adjacent atoms, plays a central role in this process and appears to explain the behavior observed in the composite containing carbon black and plant fiber. According to the Pauling scale, carbon presents an electronegativity of 2.55, while hydrogen and oxygen exhibit values of 2.10 and 3.50, respectively (Leopoldo, 2009). Thus, oxygen tends to attract hydrogen, resulting in stable bonds with displacement of electron density. Following this reasoning, carbon also presents potential to interact with hydroxyl groups, since its electronegativity is relatively close to that of hydrogen, suggesting the possibility that carbon black particles within the polymeric matrix were able to promote interaction among the composite components, as evidenced by the increase in mechanical properties.

This study was dedicated to evaluating the conditions of cellulosic fibers treated in NaOH alkaline solution in order to remove the external fatty layer present on the fiber surface. In addition, 1 wt.% N339 carbon black was incorporated into the composite with the objective of improving the anchoring between the plant fibers and the isophthalic resin polymeric matrix.

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As plant, or cellulosic, fibers, the research employed sisal and coconut fibers in the production of the composites, which were manufactured individually, with and without the presence of carbon black, in order to evaluate the mechanical properties regarding the influence of each component present in the composite.

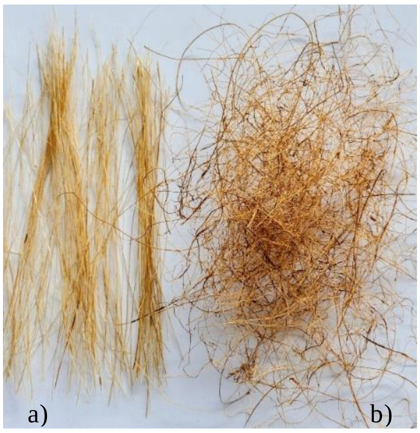
3. Materials and Methods

3.1. Fiber Treatment

Coconut and sisal fibers were manually separated in order to make them more homogeneous in terms of thickness and appearance. Coconut fibers were used in their natural size, while sisal fibers were cut to a length of 200 mm. The selected fibers were treated in a NaOH solution (20 g/L) for a period of 60 minutes in a ball mill, where they were modified simultaneously by mechanical and chemical action, which, according to the literature, alters the chemical and physical morphology of plant fibers (Fornari Junior, 2017). The solution/fiber ratio was 200 mL/5 g, based on previous studies already carried out.

After treatment, the fibers were washed with abundant water and dried in an oven at 130 ± 5 ºC for 60 minutes. After thermal treatment, the fibers were cooled in a desiccator and then used for composite manufacturing. Figure presents the selected sisal and coconut fibers, and shows the composites produced with polyester resin reinforced with sisal and coconut fibers, without carbon black.

Figure 2 – Selected (a) sisal and (b) coconut fibers



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3.2. Specimen Manufacturing

The specimens were produced using isophthalic polyester resin supplied by Mekol, with viscosity ranging from 250 to 400 cps and thixotropy between 1.1 and 1.3, using MEKP initiator (Methyl Ethyl Ketone Peroxide) at a proportion of 1 wt.%, with a total of five specimens for each composite type and for pure polyester resin. The weight of each specimen was 11.3 g.

Coconut fibers were added at a proportion of 0.1 g for each mold cavity in order to maintain the same fiber content in all respective specimens. Coconut fibers were weighed due to their irregular length distribution and randomly arranged in the mold with uniform distribution. The composites reinforced with sisal fibers were manufactured using five fibers for each mold cavity, arranged longitudinally within the mold.

The carbon black was supplied by Birla Carbon, with an average particle size ranging from 30 to 32 nm, BET surface area between 90 and 102 m²/g, and apparent density of 0.48 g/cm³. The amount of carbon black added was 1 wt.% relative to the polyester/carbon black ratio. Carbon black was incorporated directly into the polyester resin and manually stirred for three minutes. After addition, the mixture was allowed to rest for fifteen minutes, followed by the addition of the initiator and further manual stirring for three minutes. The mixture was then poured into the mold cavities.

After a curing time of 24 h, the composites were subjected to a post-curing process for 60 minutes at 80 ºC. For each composite type, five specimens were produced.

3.3. Mechanical Properties

The evaluation of flexural stress and flexural modulus properties was performed according to ASTM D790 Method B. The tests were carried out using an EMIC DL 500 Universal Testing Machine equipped with a 500 kg load cell. The flexural test speed was set at 50 mm/min. The flexural modulus of elasticity and flexural stress at rupture were evaluated.

4. Results and Discussion

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The results of the flexural tensile mechanical evaluation tests were conducted according to ASTM D790 at a speed of 50 mm/min. The testing speed is an important parameter because it alters the rate of molecular adjustment and accommodation, directly affecting the variation in material strength. In practical terms, the variation in the mechanical strength of the material decreases with decreasing tensile speed. When the tensile speed is low, there is more time available for possible molecular rearrangements, resulting in improved internal displacement conditions within the material. Consequently, lower rupture resistance conditions are observed, reducing the mechanical strength of the material. In this case, part of the energy applied to the material is consumed by molecular displacements, and therefore the final resistance of the material tends to decrease (Fornari Junior, 2017).

The final strength of the composite depends largely on fiber/matrix adhesion, fiber volume fraction, fiber orientation, and the presence of voids and defects. The interaction bonds responsible for the formation and physical structure of the fiber are hydrogen bonds. The three hydroxyl groups present in the glucose unit act to attract and couple adjacent polymeric chains, maintaining them together through a balanced arrangement of attractive/repulsive energy. On the other hand, the cellulosic structures are surrounded and protected by lignin, which occupies the structural interstices. Both lignin and fibrous structures are disorganized and subsequently solubilized by the presence of the alkaline solution (Li, 2025).

After the beginning of the stacking process of the fibrous structures, starting from the first nanofiber that initiates the structure, the sequence of stacking results in increased thickness and simultaneously a greater number of mismatches among fiber populations. These mismatches or flaws are characterized as defects, which reduce the final strength of the fiber. Thus, the external structures and layers of the fibers are removed by the action of the NaOH alkaline solution, as classically reported in the literature, reducing the fiber diameter and partially modifying its morphology, as shown in Table 1. On the other hand, the removal of the external wax layer present in coconut fibers and the partial degradation of lignin and hemicellulose in sisal and coconut fibers constitute an important modification regarding the exposure of cellulose molecules, where the polar hydroxyl regions may interact with the carbon black-modified matrix (Fornari Junior, 2017). In addition to the removal of fiber components, the partial degradation of hemicellulose favors the release of micro- and nanofibers, increasing roughness and promoting greater interaction with the polyester resin (Li et al., 2015).

Table 1 – Average diameter of 50 sisal and coconut fibers, natural and treated in NaOH alkaline solution

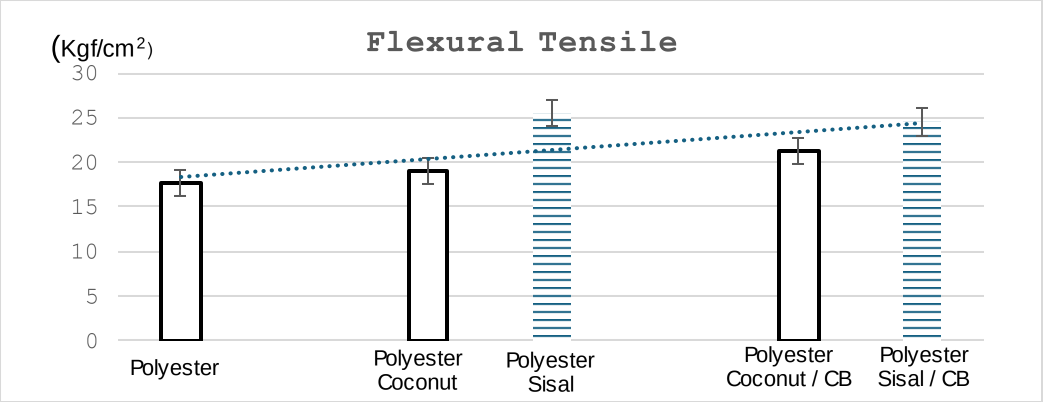
Average	Natural	Treated (25 ºC/60 min)
Sisal	0,115 mm	0,082 mm
Coco	0,182 mm	0,095 mm

As a consequence, the structures that were worn away actually correspond to the earliest fiber formations, resulting in a more precise packing of the nanofibers.

4.1. Flexural Tensile Tests

The flexural tensile test results for pure polyester and composites containing fibers with and without carbon black are presented in Figure 3. An increase in flexural tensile strength can be observed when pure fibers and fibers containing carbon black are added, increasing from 17 to 25 kgf/cm². For the sisal composite containing carbon black, the tensile strength was slightly lower than that of the composite reinforced with pure sisal fiber. In this case, the presence of carbon black acted as defect sites, probably due to the poor dispersion of carbon black within the polymeric matrix. However, for the composite reinforced with coconut fiber, the strength was higher, demonstrating that the presence of the fiber increased the material strength.

Figure 3 – Flexural tensile strength of composites with pure polyester, pure fibers, and fibers containing carbon black



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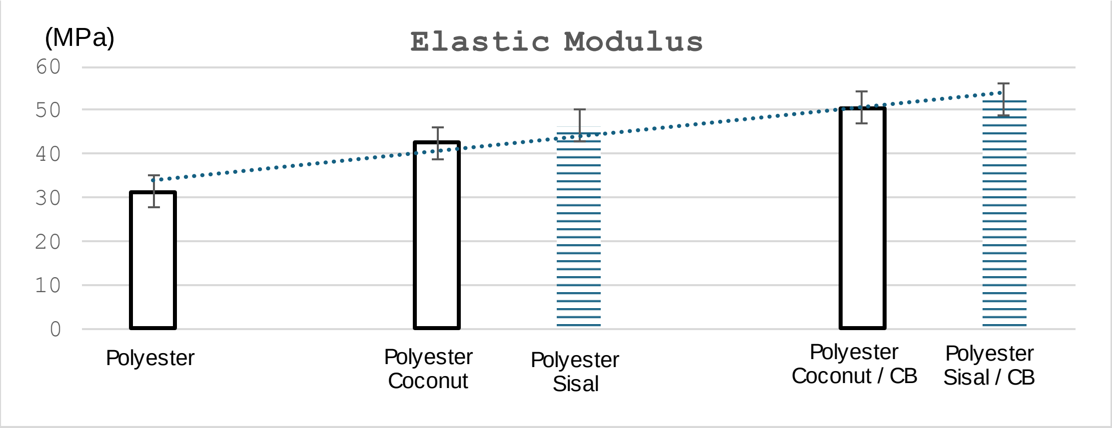
4.2. Elastic Modulus Tests

Figure 4 presents the elastic modulus results obtained for the composites. Regarding the modulus, a significant improvement was observed with the addition of pure fibers and fibers containing carbon black. The best results were obtained for sisal fibers with and without carbon black, although the difference compared with coconut fibers was very small.

The fracture surface analysis revealed that a small portion of the fibers was detached from the polyester + carbon black matrix. This indicates that the shear force was exceeded at the moment of rupture, meaning that the acting force between both materials was insufficient to maintain the fibers bonded to the polymeric matrix, resulting in partial or complete fiber pull-out. When the fiber is extracted from the polymeric matrix, a phenomenon referred to in the literature as "pull-out," the fracture surface reveals that strong chemical bonding interactions between the fiber and the matrix did not occur, allowing the fiber to be removed from the matrix during mechanical testing.

Figure 4 – Elastic modulus of composites with pure polyester, pure fibers, and fibers containing carbon black



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If the interaction between the fiber and the matrix had been more effective, the mechanical properties of the composite would have been even higher. Consequently, at the moment of specimen rupture, the matrix would remain adhered to the fiber, fracturing at another region of the matrix rather than at the fiber/matrix interface. This indicates that the composite may achieve higher flexural tensile strength values than those obtained in the present tests. The obtained values were relatively high because

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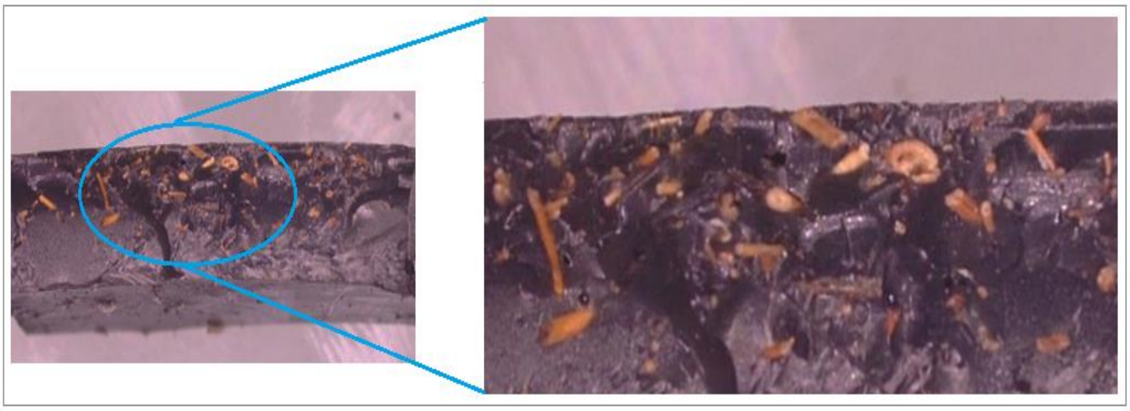
most fibers did not undergo pull-out, indicating a partially effective but positive action associated with the presence of carbon black in the composite matrix.

The incorporation of 1 wt.% carbon black into the polyester matrix promoted an increase in mechanical properties, attributed to the particulate reinforcement effect generated by the incorporation of carbon black particles. Mechanical testing clearly demonstrated a potential improvement in the fiber/matrix interface, conditioned to the adequate dispersion of carbon black within the matrix. Figure 5 presents the fracture surface image of the polyester + carbon black + coconut fiber composite, showing exposed fibers on the fracture surface.

Figure 5 – Fracture surface of the polyester + carbon black + coconut fiber composite



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The elastic modulus represents the resistance to internal displacement within the material, while the energy represents the total sum of forces exerted or accumulated by the composite until rupture during the flexural test. The results obtained for the energy values are presented in Figure 6 and Table 2

It can be observed from the table that the modulus increased in comparison with pure polyester when fibers were added and increased even further in the presence of carbon black. The highest value was obtained for sisal fiber containing carbon black. The increase in modulus reveals that material rigidity becomes greater when the fiber is present. This occurs because the chemical interactions formed between the polyester and the fiber surface arise from attractive forces between both components, resulting in a new composite material whose properties derive from the combination of its constituents

Figure 6 – Energy absorbed by the composites until rupture

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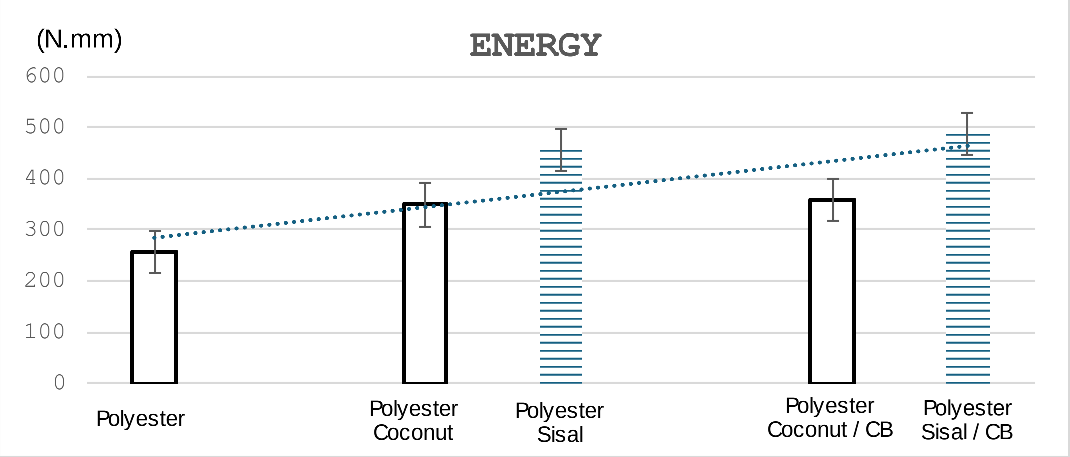

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Table 2 – Tensile strength, modulus, and energy values obtained in the flexural tensile tests, with respective standard deviation

	TENSILE STRENGTH (Kgf/cm^2)	Standard Deviation	**MODULUS** (MPa)	Standard Deviation	**ENERGY** (N.mm)	Standard Deviation
Polyester	17,64	1,2746	31,47	2,2587	256,45	33,9331
Polyester + Coconut Fiber	19,01	1,7379	42,56	4,8247	348,81	38,0181
Polyester + Coconut Fiber + CB	21,25	2,8508	50,62	2,9891	358,81	43,9766
Polyester + Sisal Fiber	25,52	3,9681	46,49	3,7248	456,03	41,8011
Polyester + Sisal Fiber + CB	24,53	2,1924	52,39	2,5021	488,23	22,9567

As a result, the macromolecular displacement within the material becomes restricted by the presence of the fiber, which presents a significantly high fiber/matrix coupling strength capable of withstanding the flexural tensile load up to the rupture point. Both composites exhibited increased modulus values; however, the presence of carbon black further increased the modulus compared with the composites without carbon black, indicating that a more effective interaction occurred due to the presence of carbon black.

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The absorbed energy value for the sisal fiber composite containing carbon black was higher than that obtained for the other materials. Similarly to the modulus behavior, the total energy required for rupture was also higher compared with the other composites. This most likely occurred due to the homogeneity of the polyester/fiber/carbon black mixture, which promoted an increase in reinforcement efficiency and reduced fragility points. When particulate agglomeration occurs, as in the case of carbon black, the properties tend to deteriorate; however, when good dispersion of the particles within the matrix is achieved, the properties improve significantly.

Specimens measuring 20 × 5 × 95 mm were tested using the three-point bending method, with a support span distance of 70 mm. For each composite type, including pure resin, five tests were performed.

5. Conclusion

The addition of both coconut and sisal fibers increased the flexural tensile strength, elastic modulus, and absorbed energy when compared with specimens produced using pure polyester resin. The addition of carbon black together with the fibers, manually dispersed for a period of three minutes in order to homogenize the polymeric matrix, demonstrated an improvement in the mechanical properties of the polyester matrix composite.

The results indicate that the presence of 1 wt.% carbon black may contribute to the increase in elastic modulus and absorbed energy, although confirmation of the interfacial mechanisms requires additional characterization analyses. The energetic interaction between the composite constituents was attributed to the specific energies of carbon, hydrogen, and oxygen, as well as to the morphological modification of the fibers promoted by alkaline NaOH treatment.

The physical reduction in fiber dimensions after alkaline treatment indicates that the fibers underwent considerable wear due to both lignin removal and the partial removal of nanofibers composing the fibrous bundle, increasing the conditions for mechanical anchoring and chemical interactions.

Plant fibers are natural products widely available in large territorial extensions, biodegradable, renewable, and characterized by low production cost. These fibers can

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be used in the manufacture of composite materials with polyester resin matrices for the development of commercial products with lower environmental impact and greater environmental sustainability.

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