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Investigation on mechanical properties of NAOH treated hybrid bio composites for lightweight materials

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Abstract: Lightweights are in demand, highly-stable as new materials for specialized purposes emerge and the demand for natural fiber-based composites is driven by the desire for more sustainable, lightweight, Environmental Sustainability, Cost-Effectiveness and environmentally friendly materials across various industries. However, it's important to note that while natural fiber composites offer many benefits, they also have limitations and challenges, such as moisture sensitivity and variability in material properties. With this work, we hope to produce a hybrid composite using epoxy as a resin and flax and coir fibres as reinforcement. Treating fibers with a 5% sodium hydroxide (NaOH) solution is a common process known as alkali treatment or mercerization. This treatment is often used to modify the properties of natural fibers, particularly plant-based fibers like hemp, flax, jute, cotton and coir. Using different ratios of fibres and resin, six samples were created. Tensile, flexural, impact, and hardness tests were used to assess the presentation of the composites. In evaluation to the other samples, specimen after alkaline treatment (A6) made of 70% matrix and 30% fiber had a substantially higher tensile strength of 245.67 MPa and a modulus of 28.43 GPa. The material exhibited a flexural strength of 155.75 MPa and a modulus of 34.13 GPa. Additionally, other notable properties were observed, including an impact strength of 36.21 kJ/m2, a hardness rating of 79.82, and a low moisture absorption rate of only 2.65%. The hybrid composites outperform several other composites described in the literature when it comes to their mechanical properties. Tests for water immersion and surface morphological analyses were also conducted.

1. Introduction

The need to improve materials' durability, rigidity, density, and affordability while retaining sustainability has arisen as a result of the manufacturing industry's rapid growth. There are several industries that use composite materials that are made with natural or synthetic fibres, including the aerospace, automotive, biomedical, construction, mechanical in nature, marine, etc. (Rowell *et al,* 2008; Jabri *et al.,* 2013). Natural fibres come in a variety of forms, each with unique qualities that affect how they are used in a variety of contexts. Due to the fact that natural fibers, even when derived from the same plant species, can exhibit a wide variety of differences in their qualities due to a range of factors, particularly the circumstances and location of their harvesting (Sadashiva *et al.,* 2023; Elidrissi *et al.,* 2012). Examined, and those features were explained, of natural fibers utilized as reinforcement (Balaji *et al.,* 2023). Evaluating mechanical characteristics of composites made of natural fibers involves a comprehensive analysis of numerous variables that can impact the overall recital of the composite material. These variables interact in complex ways, and understanding their effects is crucial for designing and engineering high-quality composites made by natural fibers. They had outlined the benefits, restrictions, and uses of various natural fibers (Sadashiva et al., 2022). The assertion that natural fiber composites are a superior substitute to synthetic fiber-reinforced composites and older technical materials is a perspective that has gained attention due to the sustainability and unique properties of natural fibers. However, it's important to note that the suitability of natural fiber composites depends on the specific application, performance requirements, and desired characteristics (Malviya *et al.*, 2020). The several fibre composite manufacturing procedures, as well as a review of the wide variety of fibres, their qualities, categorization, and functioning (Rajak *et al.*, 2019; Elidrissi *et al.*, 2012). The versatility of natural fiber composites is evident in their use in various industries such as automotive, construction, consumer goods, sporting goods, packaging, and more. However, it's important to note that the suitability of natural fiber composites varies based on the specific application requirements and the inherent limitations of natural fibers. For certain applications, a hybrid approach that combines natural and synthetic fibers can be adopted to achieve the desired balance of properties (Alkbir *et al.*, 2019). Different layers of flax fibers are arranged at various orientations (0°, 30°, 45°, 60°, and 90°) relative to the principal axis of the composite. This variation in orientation is done to mimic different loading conditions and understand how fibers respond to stress in different directions. This research involving flax fiber-reinforced polymer composites and different fiber orientations contributes to our understanding of composite mechanics and performance. Azzaoui *et al.* proposed perfect substitution materials for bone repair should have good biocompatibility and a suitable biodegradation rate as well as higher mechanical property to support the growth of new bone tissue (Azzaoui *et al.*, 2016). The findings can inform material selection, processing techniques, and design considerations for applications ranging from automotive parts to sporting equipment, where understanding anisotropic behavior is crucial for optimizing performance (Prasath *et al.*, 2019). The hybrid composites provide a pathway to unlocking the full potential of fiber-reinforced polymer composites by capitalizing on the strengths of both natural and synthetic fibers. The resulting materials can address a wide spectrum of application requirements while considering factors like performance, sustainability, and cost-effectiveness (Sivasubramanian *et al.*, 2016). Several novel composites consisting of natural fibres were created, and by adjusting the proportions, their physical and mechanical behaviour was examined. The findings showed that the composites created were superior in terms of hardness, tensile strength, flexibility, and impact resistance (Sadashiva *et al.*, 2022; Verma *et al.*, 2013). The processing of flax fibres with an emphasis on epoxy-based composites. Enhancing the bond among flax fibers and epoxy resin is a critical aspect of optimizing the mechanical performance and overall properties of flax fiber-reinforced epoxy composites. Chemical fiber treatments can play a significant role in enlightening the adhesion among the fibers and the resin matrix. These treatments modify the fiber surface, making it more compatible with the resin and promoting better bonding (Weyenberg *et al*, 2003). Studied how well hybrid composites function in engineering applications. The combination of epoxy resin, satin-woven sun hemp fiber, chitosan, and de-oiled cashew nut shell cake demonstrates a creative and innovative approach to designing hybrid composites with the aim of achieving specific mechanical, environmental, or other performance goals (Jayabalakrishnan *et al*, 2023; Akartasse *et al.*, 2022). Studying the effects of ageing conditions, such as exposure to water and ultraviolet (UV) radiation, on the mechanical and physical properties of short sisal and polypropylene composites is essential for understanding how these materials behave over time and under various environmental conditions (Joseph *et al*, 2002). Examining mechanical properties such as hardness, tensile strength, flexural strength, and impact strength in the investigation of the short sisal and polypropylene composites under different ageing conditions provides valuable insights into how these materials respond to external factors. Studying the mechanical and acoustic characteristics of hybrid composites made from coconut and bagasse fibers is a multifaceted research effort with implications for material development, sustainable practices, and tailored composite design. It is evident from the aforementioned literature reviews that very few researchers have studied flax and coir composites. The hybrid composites constructed of natural fibres like flax and coir have widely studied, we cited for example, Narendar *et al.* (2014) developed coir pith/nylon fabric/epoxy hybrid composites by using hand layupand compression moulding method. In order to construct hybrid composites employing flax and coir fibers, an effort has been undertaken in this work. The mechanical properties of the bio composites are significantly influenced by the matrix material that has been used. The choice of epoxy resin, with its strong mechanical properties, chemical resistance, and modifiable characteristics, supports the research project's aim to create hybrid composites with desirable mechanical performance and durability. Additionally, epoxy's compatibility with natural fibers enhances the potential for achieving a strong fiber-matrix interface, which is crucial for optimizing composite properties. The tools and procedures are shown in more detail in the next section.

2. Methodology

2.1 Materials and methods

Balaji Composites Pvt Ltd. Bengaluru, Karnataka, India, got the husk of coir fibres and the stem of flax fibres. The Hardener and Epoxy Resin were purchased from Ultra nanotech Pvt LTD in Bengaluru, Karnataka, India. The releasing agent utilised to create the composite samples was polyvinyl alcohol, which was provided by Balaji Chemicals in Bengaluru, Karnataka, India.

2.2 Extraction of flax fiber

Since the method of extraction used has a significant impact on both the quality and quantity of extracted fibres, the extraction process is crucial. Fibre extraction involves removing fibre from the chemicals that cement it, such as pectin or lignin, wax, resin, lipids, and other carbohydrates. Flax plant fibres are removed using a single method or a mix of mechanical and chemical retting techniques. The quality of the fibre to be recovered heavily influences the extraction technique choice. Fibres are now mechanically removed. Following harvest, the flax stalk goes through several extraction procedures, including retting and scutching. Flax is collected right before the seed is ready, then the seed and leaves are removed by a procedure known as rippling. Flax is then dried for a few days. After ripening, the plants are dispersed on grass and allowed to ferment for a few weeks under the influence of the weather. A variety of retting methods are used to remove the fibre from the stalks. In India, water retting is frequently employed.

Figure 1. Extraction of flax fibers.

Table 1. Physical and chemical composition of coir and flax fibers

Type of fibers	Cellulose (%)	Hemicellulose (%)	Lignin (%	Pectin (%)	Density(g/cm3)
Coir fiber	36-43	0.15-0.25	41-45	3-4	1.15-1.40
Flax fiber	65-70	5-9	50-55	4-5	1.4-1.45

2.3 Fiber treatment

Chemical treatment was applied to flax and coir fibres to improve their surface roughness and bonding properties. 24 hours at ambient temperature were spent submerging fibres in a 5% sodium hydroxide solution (NaOH). The NaOH treatment had an impact on the fibres' thermo physical properties. The fibres were given a chemical treatment, rinsed under running water to eliminate any alkali residue, and then allowed to air dry for 48 hours.

2.4 Fabrication of composite materials

The ratios of the fibre and resin were changed to produce six different samples. While the other three samples received a NaOH solution treatment, three samples were left untreated. The hydraulic cold compression moulding technique was used to create the composite specimens (Marichelvam et al, 2021). Using a compression molding machine, specifically the model JRD CE 150D, to create the composite samples is a common and well-established method in the field of composite materials manufacturing. Compression molding is a versatile technique that allows for the production of various composite components with controlled pressure, temperature, and time parameters. Using conventional dies with measurements of 150 x 150 x 4 mm to create the composite samples further illustrates the standardized and controlled approach to sample fabrication. Employing a steel roller in the rolling process to maintain fiber homogeneity, enhance isotropic characteristics, and eliminate air bubbles is a strategic step that contributes to achieving uniform and high-quality composite materials. This method is often used in composite manufacturing to optimize material properties and ensure consistent results. Closing the die and applying hydraulic pressure for ten hours at room temperature is a notable step in the composite manufacturing process that allows for thorough consolidation of the composite materials. This extended duration of pressure application contributes to achieving proper resin matrix and fibres are bonded together, as well as minimizing voids and imperfections. Table 2 provides an illustration of the matrix weight, coir fibre and flax fibre weights.

Table 2. Samples of different composition

Specimen	Treated and untreated fibers	Matrix and fiber ratio	Flax and coir ratio	Matrix weight (g)	Coir weight (g)	Flax weight (g)
A1	Untreated	60:40	50:50	71.451	23.214	23.214
A2	Untreated	65:35	50:50	74.852	21.269	21.369
A3	Untreated	70:30	50:50	81.250	18.691	18.619
A4	Treated	60:40	50:50	71.451	23.214	23.214
A5	Treated	65:35	50:50	74.852	21.269	21.369
A6	Treated	70:30	50:50	81.250	18.691	18.619

3. Experimentation

3.1 Tensile test

Tensile tests being performed on the composite samples using a universal testing machine (specifically the INSTRON-6025 type Universal Testing Machine) at a speed of 1.5 mm per min is a standard technique for evaluating a mechanical properties of materials, including composites. This testing procedure provides valuable data on how the composite materials respond to tensile forces and how their properties are affected. The composite sample remained produced from the composite plate in accordance with ASTM D638 standards. Conducting the majority of the tests at room temperature and using an average sample size of 150 x 20 x 3 mm3, with a gauge length of 100 mm, to examine tensile strength, tensile modulus, and percent of elongation provides valuable insights into the mechanical behavior of the composite samples under normal operating conditions.

3.2 Flexural test

Performing flexural tests at a speed of 1.5 mm per min through a universal testing machine (UTM) in accordance with ASTM D790 is a standardized method for evaluating the bending or flexural properties of materials, including composites. This testing approach provides essential information about a material's behavior under bending forces and is widely recognized in industries and research settings. Using the Bluehill INSTRON Universal Testing apparatus to perform flexural tests ensures accurate, standardized, and reliable evaluation of a material's bending properties. The flexural testing were conducted in a room temperature environment. Using samples with dimensions of 150 x 24 x 3 mm3 mm for investigating flexural properties is a well-considered choice that provides valuable information about the material's behavior under bending loads.

3.3 Impact test

Performing impact tests on the samples in accordance with ASTM D256 standard is a crucial step in evaluating the toughness and resistance of the composite materials to sudden impact or shock loads. This test provides valuable insights into how well the composite samples can absorb energy and withstand impact forces. Using impact test samples with dimensions of 60 x 12 x 3 mm3 and performing the impact test according to requirements using an INSTRON CEAST 9050 impact-testing equipment demonstrates a well-defined and standardized approach to evaluating the impact resistance of composite materials.

3.4 Micro- Hardness test

A hardness test is a valuable method for evaluating a material's resistance to deformation or indentation caused by contact with a harder substance. This test provides insights into the material's mechanical properties and its ability to withstand localized stresses, wear, and long-term distortion. The Shore D hardness scale is specifically designed for harder materials, such as plastics, rubbers, and composites. It measures the material's resistance to penetration by using a pointed indenter. The test is standardized, reliable, and widely used across industries to evaluate material properties, ensure quality, and inform design choices.

3.5 Moisture Absorption test

The water absorption test is a vital tool for evaluating a material's interaction with moisture and its potential consequences. The test provides valuable information for material selection, design optimization, and assessing a material's suitability for applications exposed to environmental conditions involving water. The sample is immersed in water for a specified duration, often 24 hours. This immersion simulates exposure to moisture over time, which can be relevant in real-world applications. After 24 hours the sample is removed from the water, and its weight is determined again. The increase in weight indicates the amount of water the material has absorbed.

$$MAT = \frac{R2-R1}{R1} x100 \qquad \text{Eqn... (1)}$$

R1= Sample's initial weight, in g
R2= Sample's final weight, in g

3.6 Analysis of the materials' surface morphology

The scanning electron microscope is a versatile and powerful instrument that provides detailed images of a sample's surface at very high magnification. It is used across numerous scientific and industrial fields to study materials, analyze structures, and gain insights into the fine details of various objects and specimens. Using the JEOL JSM model 6390 scanning electron microscope (SEM) with an acceleration voltage of 20 kV to examine and analyze fractured samples from tensile, flexural, and impact tests is a standard practice in materials science and engineering research. The signals generated when electrons interact with atoms in a sample in techniques like scanning electron microscopy (SEM) and provide valuable info about the surface topography and chemical composition of composite materials.

4. Results and Discussions

4.1 *Tensile test*

Figures 2 and 3, highlighting the tensile strength and tensile modulus test outcomes for different specimens, is valuable for understanding the mechanical properties of the composite materials. The treated sample A6's ultimate tensile strength of 245.67 MPa indicates that it can withstand a significant amount of force before failure. A higher tensile modulus indicates that the material is relatively rigid and less prone to deformation under load. The treated sample A6's tensile modulus of 28.43 GPa suggests that it has high stiffness and is capable of maintaining its shape under stress. Based on the results, sample A1 untreated tensile strength and tensile modulus are both 189.02 MPa and 11.36 GPa, respectively.

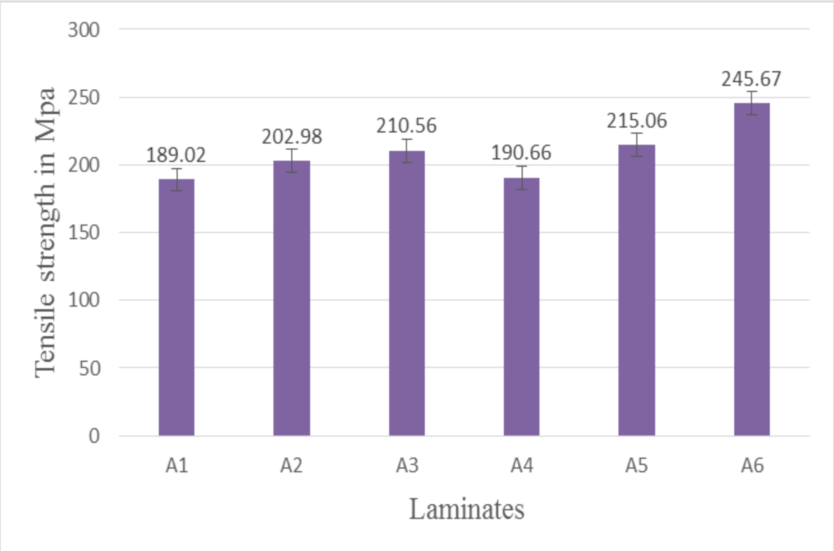

Figure 2. Tensile strength Vs Laminates

Tensile strength for the untreated sample A3 is 210.56 MPa. This is higher than samples 1 and 2, respectively, by 37.52% and 16.43%. The treated sample A6's ultimate tensile strength of 245.67 MPa. In comparison to the untreated sample of the same configuration, this is 20.47% higher. Additionally, sample A6 has greater tensile strength than samples A4 and A5. According to the results, sample A6 stands out from the other samples in terms of its characteristics. Compared to hybrid composites consisting of bagasse and jute fibre, which have a tensile strength of 49.08 MPa, (Marichelvam et al, 2022) A6's tensile strength is significantly higher. Observing the development of an ornate SEM (Scanning Electron Microscope) layer to examine the impact of surface treatment on the interface between flax and coir fibers is a significant approach in materials science and composite research. This technique provides visual insights into how surface treatments affect the interaction and bonding between different fibers, shedding light on the changes in microstructure and adhesion. This approach contributes to a deeper understanding of composite material behavior and guides the development of more effective and high-performance materials for various applications (Sadashiva et al., 2022). Figure 4, featuring SEM images of tensile fractures in flax and coir fiber hybrid composites with different compositions and treatments, provides valuable visual insights into the fracture behavior and structural changes of these materials.

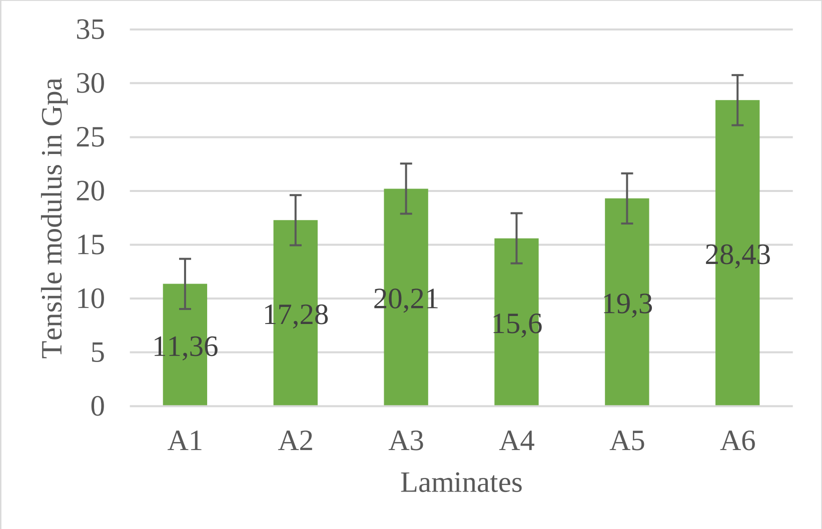

Figure 3. Tensile modulus Vs Laminates

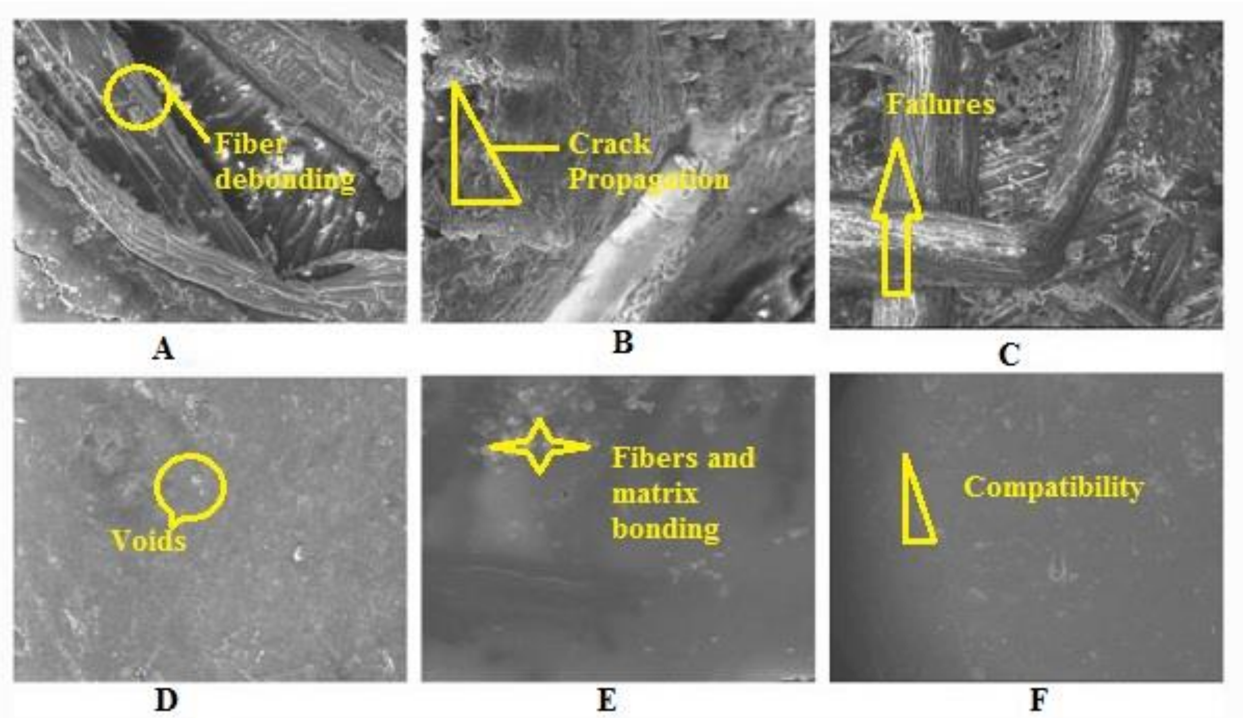

Figure 4. Tensile fracture of SEM images

The SEM images help researchers understand how fibers and matrix interact during failure, revealing crucial information about mechanical properties and composite performance. Flax and coir fiber hybrid composites combine the properties of both fiber types, and observing their fracture behavior helps evaluate the synergistic effects of this combination. SEM scans demonstrated that the fibre surface experiences typical structural changes. Additionally, it was confirmed that the NaOH treatment, which partially eliminated the hemicellulose, lignin, and other soluble materials, caused the fibre surface to become tougher. The SEM pictures of the untreated samples reveal substantial and uneven voids. The early failure of the untreated samples was brought on by the voids in the surface. Additionally, it has been found that the NaOH-treated fibres have improved coherence between the matrix and the fibre, which has led to superior tensile qualities.

4.2 Flexural test

Figures 5 and **6** display the results of the various samples' flexural strength and flexural modulus. The treated sample A6 has a flexural strength of 155.72 MPa and a modulus of 34.13 GPa, making it stronger than other samples A1, A2, A3, A4, and A5. Flexural strength is 62.05 MPa and modulus is 21.39 GPa for the composite made with coir fibre. According to the results of the comparison, it can be said that the hybrid composites comprised of coir and flax fibre have superior flexural capabilities than many other composites discussed in the literature. Sample A6 has a flexural modulus of 34.13 GPa. In comparison to other treated and untreated samples, this value is higher. Sample Six (A6) has a substantially greater flexural modulus than the hybrid composites discussed in. 155.72 MPa is the flexural strength of A6. This is higher than the strength of composites made using rambans fibre (35.76 MPa) and Himalayan agave fibre (54.51 MPa) (Kumar et al, 2022).

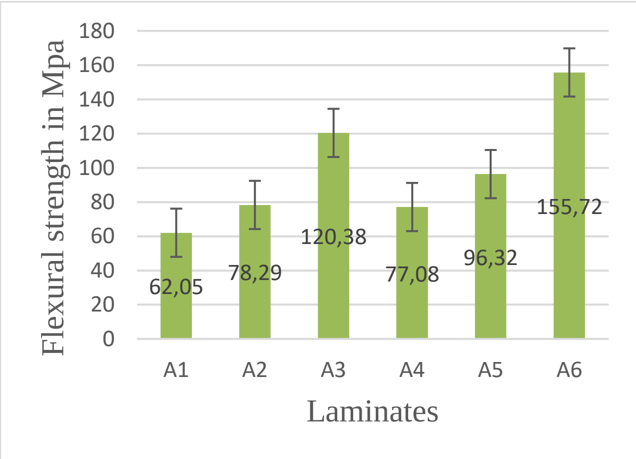

Figure 5. Flexural strength of various laminates.

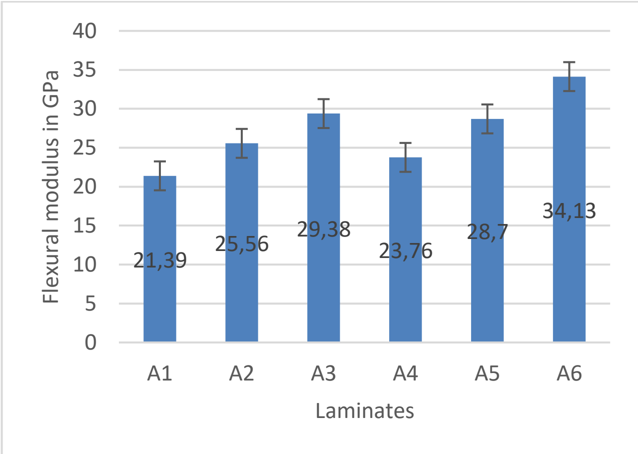

Figure 6. Flexural modulus of various laminates

Figure 7's SEM images of flexural failure samples provide essential visual information about the behavior of coir and flax fiber hybrid composites under bending loads. These images help researchers understand how composition and treatment influence flexural failure mechanisms, thereby contributing to the broader understanding of composite material behavior and guiding material design and application choices. Comparing treated and untreated samples sheds light on how surface treatment affects the composite's flexural performance. Changes in interfacial bonding can lead to different failure patterns. Observing more adhesive matrix particles in treated fiber composites through SEM images confirms the positive impact of the surface treatment on interfacial bonding. This enhancement contributes to better load transfer, improved mechanical properties, and enhanced overall composite performance. The interface between fibers and the matrix in a composite is critical for load transfer, stress distribution, and overall mechanical performance. Improved adhesion between fibers and the matrix is desirable as it prevents fiber pull-out and enhances load-bearing capabilities. The observation that the fibers are widely disseminated in the treated samples, leading to greater adhesion between the fibers and the matrix as evident in the SEM images, directly correlates with the upsurge in flexural strength of the treated samples. This insight highlights the crucial role that fiber dispersion and interfacial adhesion play in determining the mechanical properties of composite materials.

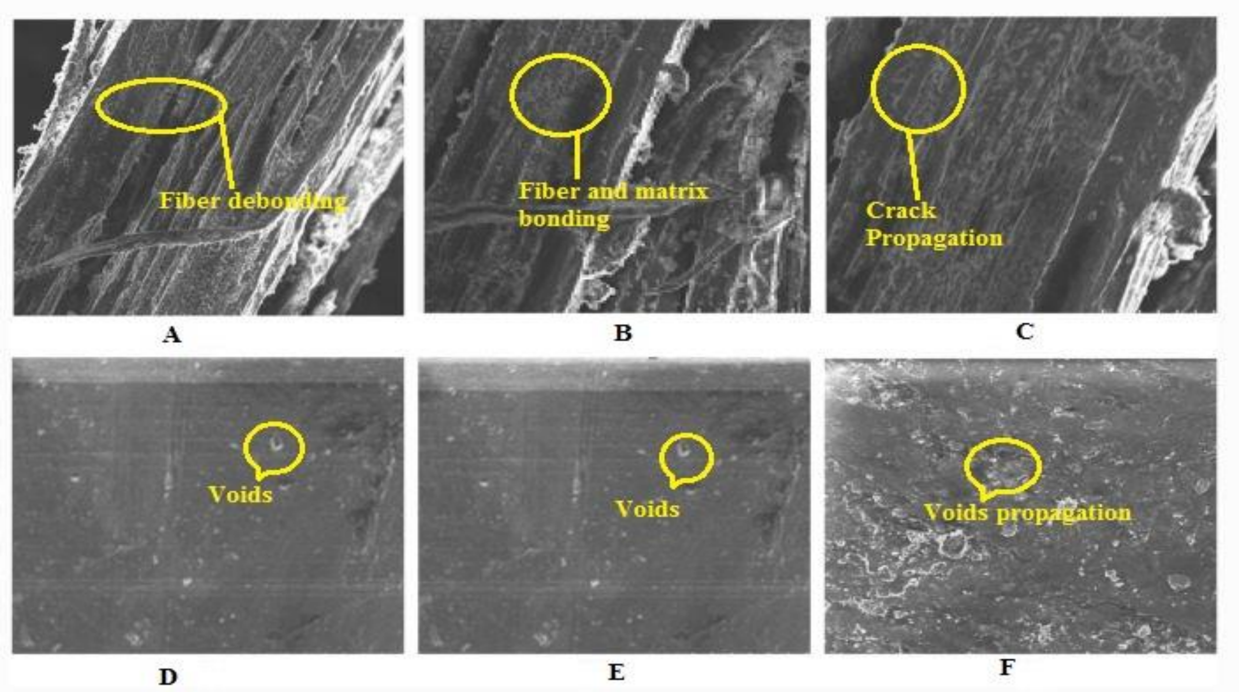

Figure 7. Flexural fracture of SEM images

4.3 Impact strength

The fibre and matrix composition affects the composites' impact strength in different ways. Figure 8 displays the samples' impact test outcomes. The clear observation from the figure that sample six (A6) has the highest impact strength, measuring 39.21 kJ/m², is a significant indication of the effectiveness of the treatment process on improving the material's ability to withstand sudden impact loads. Comparing this impact strength value with those of other hybrid composites, such as those consisting of sisal and kenaf fibers with an impact strength of 18.8 kJ/m², highlights the remarkable enhancement achieved through the treatment (Marichelvam et al, 2022).

The observation that untreated fibers and epoxy matrices do not form a strong bond is substantiated through the occurrence of voids, aggregates, and fiber pull-out on the fractured surface. These visual indications provide valuable evidence of the challenges in achieving effective adhesion and mechanical interaction between untreated fibers and the matrix. This suggests that the right methods for fibre extraction and treatment should be used. Better adhesion between treated fibres and epoxy matrices is detected in SEM images of treated fibres. The treated samples' impact strength was consequently increased.

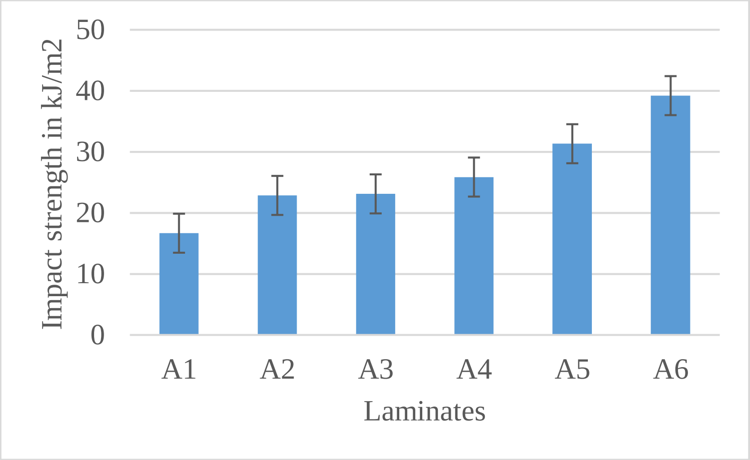

Figure 8. Impact strength of various laminates

4.5 Micro- Hardness test

The results shown in Figure 9 offer a quantitative measure of the material's hardness, reflecting its ability to withstand deformation from contact with a harder substance. The finding that sample 6 (A6) has a higher hardness value compared to the other specimens are significant and indicates that the treatment process has led to an increase in the material's resistance to indentation. The comparison between the hardness values of the hybrid composites made from bagasse and coconut coir fibers (59.23 HD) and Sample A6 (79.82 HD) further highlights the significant impact of the treatment process on enhancing the material's hardness (Marichelvam et al, 2021). Compared to other specimens like A2, A3, A4, and A5, the hardness rating for the A1 sample is 66.33, which is lower.

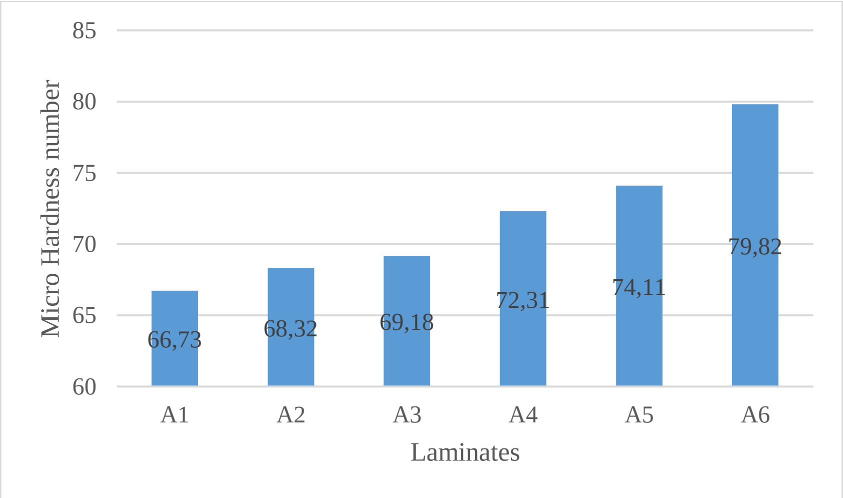

Figure 9. Micro hardness values of different laminates

4.6 Moisture Absorption Test

The data from Table 3, indicating that sample 6 (A6) exhibits the lowest water absorption properties, carries significant implications for the material's potential applications, especially in scenarios where water absorption resistance is important. Materials with low water absorption are less prone to developing issues such as warping, dimensional changes, or mold growth, leading to reduced. This property opens up opportunities for using this sample in applications where water absorption needs to be minimized for long-term performance and durability.

Table 3. Moisture absorption results of various laminates

Sl no	Specimens	Moisture Absorption %
1	A1	4.45
2	A2	4.36
3	A3	4.10
4	A4	3.88
5	A5	3.65
6	A6	2.65

5. Conclusion

Hybrid coir and flax fibre composite samples were created in the current investigation using epoxy as the matrix. We believe that this is the first application of the aforementioned fibres in the creation of hybrid composites that has been publicly published. Six distinct samples were created by adjusting the fibre and matrix. A variety of mechanical tests were carried out in accordance with ASTM standards to verify the performance of the samples. The treated sample with a matrix fibre proportion of 70:30 demonstrates improved mechanical characteristics, according to the results. The material demonstrated a higher tensile strength of 245.67 MPa and a modulus of 28.43 GPa. Furthermore, it exhibited a flexural strength of 155.75 MPa and a modulus of 34.13 GPa. Additionally, other noteworthy properties were recorded, such as an impact strength of 36.21 kJ/m2, a hardness rating of 79.82, and a low moisture absorption rate of merely 2.65%. The characteristics outperformed those of other fiber-based composites in comparison tests. Additionally, a surface morphological examination was completed. Although improved mechanical qualities were reached, it would be fascinating to build a product that was application-focused, which would be beyond the current work's scope. Future work on this project will focus on the hybridization of the aforementioned fibres with additional fibres in order to create more hybrid composites employing coir and flax fibres.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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