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Synthesis & Characterization of Cotton Fiber Reinforced Epoxy-CNSL Hybrid Resin Composite Material

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ABSTRACT

Received: 18 Dec 2024 Revised: 10 Feb 2025 Accepted: 28 Feb 2025 Introduction: The natural fibers in composite materials have grown significantly in recent years, mainly because of their outstanding environmental benefits like recyclability, renewability, and biodegradability. The paper is interested in the experimental study of hybrid composites mechanical characteristics. The hybrid composites are strengthened with cotton fibers and hybrid resin matrix with cashew nut shell liquid and epoxy. For optimum hybridization to improve the CNSL epoxy IPN interpenetrating network, enhanced mechanical properties of the composite were used. Five hybrid composite samples were prepared using different weight percentages of cotton fibers, from 0 to 40%, and the samples underwent impact, flexural, and tensile strength testing. Sample 5 with 40 wt% cotton fibers showed the highest tensile strength of all and had an improvement of 8.3% over other samples. The increase resulted from higher adhesion of fibers in the matrix as well as distribution of stress through the formation of IPN. With cotton fiber concentration enhancement, the flexural strength is enhanced to 35% because of the hybrid resin's capacity to absorb energy under load circumstances. Impact testing demonstrated a 40 wt% increase in absorbed energy for composite 40 wt% cotton fiber materials, clearly demonstrating the synergistic effect of hybrid matrix systems on strength and impact toughness. Hybridization of cotton fibers with CNSL and epoxy resins seems to offer a real innovative route toward the achievement of lightweight, high-strength composites that can be used in structural, aeronautical, and automotive applications.

Keywords: Cotton fibers, Epoxy resin, CNSL, Composite, SEM, and Mechanical Tests.

INTRODUCTION

Natural fibers are recyclable, exhaustible, and biodegradable substances which facilitate the creation of composite materials [1-2]. In the contemporary trend of developing green composite materials, the sisal and flax natural fibers are used as alternatives to manufactured fibers like glass fiber. Natural fiber manufacturing enhances corrosion resistance and surface-related tribological properties, contributing to an eco-friendly environment. Fiber-reinforced composites derived from plant and animal sources continue to be extensively utilized in non-structural and structural tasks. The primary constituents are lignin and cellulose [4, 5, 6]. Natural fibers exhibit several disadvantages, including increased water consumption, reduced inner surface contact, instability at elevated temperatures, and incompatibility of certain fibers for matrix reinforcement [7]. Modification of fiber surfaces is crucial for enhancing adhesion with the matrix and decreasing moisture content through chemical processes [8]. Pure epoxy resin is combined with natural fibers to form straw-like structures. They significantly improve the composite material's tribological and mechanical properties. To enhance the natural fibers performance, appropriate coupling agents can impart hydrophobicity, or they may be coated with suitable resins. The rigidity of cell walls, the coarse structures, and the lengths of fibers in plants all differ in the manner in which the cells are interconnected [9]. To develop new composite materials, natural fibers may undergo chemical or physical treatment to enhance their adhesion to the matrix [10]. Chemicals are employed in physical treatment to eliminate natural fiber constituents, including wax, lignin, and oil. They enhance the fiber's adhesion to the polymer matrix and increase surface roughness [11, 12].

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Currently, extensive study is conducted utilizing hybrid natural fillers in polymeric materials. These fillers are ideal for cost-effectively improving mechanical properties. The primary rice producers globally are China, India, and Indonesia. [13]. The natural fiber strengthened composites' over metallic and synthetic fiber-based materials regarding weight and environmental impact have recently attracted significant attention. [14–17]. When it comes to performance, natural fiber-reinforced composites (NFRCs) outperform synthetic fibers like glass fiber, in addressing environmental concerns, as evidenced by research utilizing life cycle assessment (LCA). [18, 19]. A significant limitation that constrains the natural fiber composites' utilization in industrial and biomedical sectors is their diminished strength. [20, 21]. This constraint could be brought about by the laminates' insufficient fiber-matrix interaction. To improve mechanical qualities and address strength-related challenges, researchers have employed various strategies to strengthen the interfacial connection between the matrix and the reinforcing material. According to Ejazet al. [24], the incorporation of nanoparticles is a prevalent technique. To enhance interfacial bonding, an alternative strategy suggested in various studies [26-28] involves treating fibers with an alkali or NaOH solution. The amalgamation of two distinct polymers to enhance the qualities of the constituent resin is another strategy suggested by Hiremath et al. [29] and implemented in this study. The matrix is demonstrated to significantly enhance mechanical properties. Turcsan and Meszaros [29] observed an increase of 17-21% in the mechanical characteristics of fiber when examining resin strength [30]. Chakraborty et al. [31] examined the IPN structure using unsaturated bonding between jute and mortar (PS3 sample) and enhanced the fiber's compressive and flexural strengths by approximately 16% and 9%, respectively. The challenges presented by greenhouse gas emissions will be tackled by enhanced research and development of renewable materials. Entirely green biocomposites will address many recyclability concerns. Although partially green epoxy matrices with reduced fossil fuel content currently exist [32], both flexural and impact strengths were found in this novel composites which were 10% and 7% superior, respectively. A multitude of researchers are focused on polymer hybridization [28, 29, 33, 34]. Although limited information exists regarding the application of this technique in the analysis of natural fibers [35-38], there is a paucity of research examining its impact on the properties of synthetic fibers [39-45].

Despite significant advancements in composite materials, most of their substantial research potential remains untapped in the integration of natural fibers and hybrid resin systems to achieve optimal mechanical performance. Most contemporary research investigations primarily concentrate on either synthetic or natural fibers, with few investigating the hybridization of both natural fiber reinforcement and hybrid resin matrices. Another area of focus is the strengthening of cotton fibers, cashew nut shell liquid (CNSL), and epoxy to create high-strength composites. Limited research exist that demonstrate the synergistic benefits of these resources. Although hybrid resins have been shown to improve toughness and impact resistance, the mechanisms of interpenetrating polymer networks (IPNs) are not yet well comprehended, particularly regarding their influence on composites' impact, flexural and tensile properties. Furthermore, significantly less research has been conducted on this topic concerning high-performance sectors such as automotive, aeronautical, and structural industries. The aforementioned research gaps are anticipated to be addressed through comprehensive investigations into the effects of hybridization on both reinforcement, specifically cotton fibers, and the matrix, comprising CNSL and epoxy, regarding the composite materials mechanical properties. Hybrid composite materials were synthesized by integrating natural cotton fibers with a hybrid CNSL/epoxy resin matrix. The hybridization technique is tailored for the development of interpenetrating polymer networks (IPN) between epoxy resins and CNSL. The impact, tensile, and flexural strength, of the produced hybrid composites have been examined. It has evaluated the optimization of the fibre-matrix mix to enhance mechanical performance. A comprehensive microstructural investigation employing techniques like SEM is performed on the fiber-matrix interface to evaluate adhesion levels and fiber pull-out.

REINFORCING PHASE

The qualities of the material are enhanced when reinforcing chemicals are added to the resin. Cotton fibers are utilized as reinforcing agents in composite materials to enhance their various qualities as shown in below Table 1.

2025, 10(34s) e-ISSN: 2468-4376

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Table 1. Fiber with resin properties.

Materials	Density(g/cm3)	Strength of Tensile (MPa)	Tensile Modulus (GPa)	Elongation (%)
Cotton fiber	1.50	300	5.5	3.0
Epoxy resin	1.16	60	4.1	1.5
CNSL	1.01	44	2 - 4.5	2.7

2.1. Hybrid Composite Fabrication

This study uses the compression moulding technique to fabricate hybrid composites. The 300 mm by 300 mm square plates with a 3 mm thickness were created from the composites. We created five distinct types of composites, each with differing proportions of matrix and fibers. Table 2 delineates the precise fiber compositions for each composite. Each composite was subjected to a load of about 1500 psi for thirty minutes during the curing process before removal from the mold. Figure 1 depicts the fabrication of a composite material. Five specimens have been prepared for this study. The specimens comprise different weight percentages (wt. %) of cotton fibers combined with a mixture of epoxy resins, hardener, and CNSL.



Figure 1. Laminate composite process

Table 2 shows the composition of hybrid composites with thickness, reinforcement matrices and resins combined with CNSL (wt. %).

Table 2: Composition of hybrid composites

Samples	Thickness (mm)	Reinforcement	Matrix Composition	
		Cotton fibers (%)	Epoxy resin (%)	CSNL (%)
1	3	0	85	15
2	3	10	75	15
3	3	20	65	15
4	3	30	55	15
5	3	40	45	15

2025, 10(34s) e-ISSN: 2468-4376

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MECHANICAL TESTING

Tensile Testing are performed using a computer-controlled electronic UTM through a 100 KN load cell limit and speed 2 mm/sec. Every specimen tested in ambient temperature and pressure condition. Figure 2 & figure 3 showshybrid samples with standardsfor tensile & flexural testing.

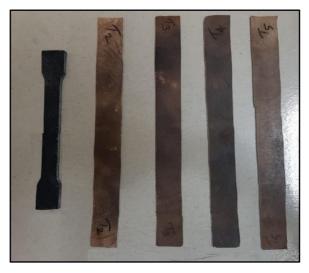


Figure2: Hybrid samples prepared according to ASTM D3039 standard (Tensile testing)

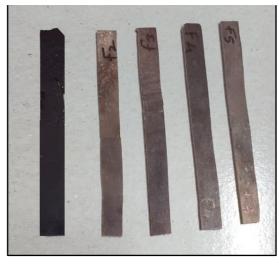


Figure3: Hybrid samples prepared according to ASTM D7264 standard (Flexural testing)

An ASTM D256 Semi-Automatic pendulum impact tester which is equipped with a 14 kilogram of load arm is utilized to perform the Izod impact test. Each composite sample has a V-notch machined on one face with a 2 mm depth at 45 degrees using a shaper machine. Figure 4 shows hybrid samples produced in accordance with the standards set by the ASTM for impact testing.



Figure 4: Hybrid samples prepared according to ASTM D256 standard (Impact testing)

RESULTS AND DISCUSSION

> Uniaxial tensile test

Figure 5 illustrates a comparison of hybrid resin strengthened with cotton fiber composites' tensile strengths against other hybrid sample composites. Sample 5 exhibits the most strength. Significant enhancements in tensile strength of 8.3% occur as the content of cotton fibers is elevated from 0% to 40%. The enhancements in uniaxial tensile strength result from the interwoven phase development of hybrid matrices. When two thermoset polymers were

2025, 10(34s) e-ISSN: 2468-4376 https://www.jisem-journal.com/

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hybridised, interfacial contacts were formed, leading to enhanced tensile characteristics. Forming the IPN structure was made easier by strong interlocking connections between CNSL and epoxy.

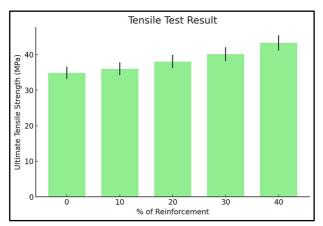


Figure 5: Tensile test result Graph of ultimate tensile strength Vs percentage of reinforcement

The inclusion of CNSL, cotton fibres, and epoxy resin improved the hybrid composites' tensile strength. The following are six possible reasons that may have contributed to the strength during the hybridization process along with proper discussions. Hybridization of CNSL and epoxy initiates the interpenetrating polymer network formation, which enhances the mechanical properties of the composite. An IPN is formed when two polymers coalesce and interlock at a molecular level. In this case, the polymers used are CNSL and epoxy. The IPN structure will exhibit high tensile strength because of better load distribution and transmission of stress among matrix and fibres. The superior mechanical properties result from better compatibility and stress dissipation throughout the interface of fiber-matrix because of interaction between the two resins. Cotton fibres, CNSL, and epoxy matrix all contribute to the improvement of the fibres and matrix's interfacial adhesion. Therefore, the transmission of stress to the stronger fibres from the weaker matrix is significantly influenced by the strong adhesion of fibre and matrix, which is a critical factor in tensile behaviour. The cotton fiber sserves like a load sustainable reinforcements, while the hybrid matrix enhances the adhesion of fibers with the surrounding resin, and, hence, the tensile strength increases. A synergistic interaction occurs when the two resins interact with each other when CNSL is combined with epoxy. In this case the one matrix vulnerability is covered by the other strengths. CNSL possesses good thermal stability and resistance toward wear; epoxy provides toughness as well as structural integrity. The composite has greater tensile strength than when used alone for each resin when used together. Hybrid matrices show the synergistic effect mainly because the mutual entanglement and interaction of the two phases result in a much stronger and tougher material. Improved load transfer efficiency within the composite is a result of the hybridization of cotton fibers with CNSL and epoxy. Higher contents of cotton fibers in sample 5 mean that there is a better transfer of loads between resin matrix and fibers. The reason for this is that fibres and matrix have stronger interactions as regards stress distribution that reduce the concentration of stresses; hence there will be a greater tensile strength. Hybrid matrix contains distributed Cotton fibers that ensure uniform stress distribution. Cotton fibers have flexibility as well as toughness. This prevents higher stress concentrations occurring at all points in the matrix. In increasing cotton fiber content from 0 to 40%, tensile strength for the composite is increased. This indicates that fiber reinforcement plays a key role for ensuring the distribution of tensile stresses over the matrix and strengthening the structure. The introduction of cotton fibers raises the composite's tensile properties because of the relatively high amount of strength to weight associated with natural fibers. Cotton fiber is light yet very strong and contributes to a higher volume fraction of fibers within the hybrid matrix, thereby enhancing the composites mechanical performance as a whole. Usually, an increase in volume percent of fibers increases the hardness and tensile strength. The number of fibers increases to carry the load as the fibers are stiffer and possess greater tensile strengths[46-51].

2025, 10(34s) e-ISSN: 2468-4376 https://www.jisem-journal.com/

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> Scanning Electron Microscopy test

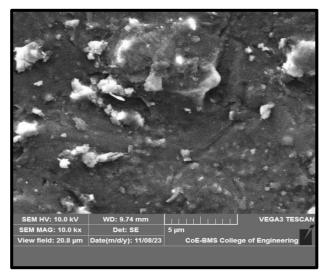
Tensile fracture mechanism of hybrid cotton fiber reinforced CNSL/epoxy composites is very crucial for material behavior, particularly mechanical behavior. In composites reinforced with fibers, the matrix is a load-transferring member in that it distributes the applied load to the fibers, which act as the most significant load-carrying members. If weak binding of fibre and matrix exist, the overall load transfer efficiency drops sub optimally, leading to the suboptimal mechanical performance.

Figure 6 and 7 are the tensile fracture surfaces of the cotton/CNSL epoxy composites through the micrographs of SEM. Figure 6 gives the composite fracture surface, with which there are several fiber pullouts shown in the magnified images; it signifies vulnerable adhesion of CNSL/epoxy matrix and cotton fibers. The micrograph shows a rather smooth surface with minimal existence of fiber-attached matrix resin. Micrographs show clean fibre surfaces, which means there isn't much interfacial bonding. Fiber pull-out occurs when the matrix and fibers bonding is incapable of transferring a load from the fibers to the matrix, causing early fiber debonding and subsequent failure of the composite under tensile load. This would eliminate a poor composite mechanical characteristics, since the tensile-induced stress is not efficiently distributed in both fiber and matrix phases.

The matrix and cotton fibers adhesion is inadequate because of insufficient chemical or physical interaction at the interface. Lack of bonding severely reduces the ability of the matrix to achieve effective tensile load transfer with subsequent failure by fiber pull-out as in Figure 6.

Figure 7 shows a contrast cotton/matrix blend with fibre pull-outs, which are relatively much smaller and fiber surfaces appear to be cleaner. In the composites, tensile strength is most improved where the cotton fibers comprise 40 weight percent and CNSL/epoxy as matrix comprises 60 weight percent. Higher concentration of fiber increases the available surface area for bonding at fiber-matrix interface and increases interfacial adhesion bond. At the same time, there is stress transfer optimization from fibers to matrix, which leads to an enhanced mechanical performance. Better adherence of matrix's substance to the fibre surface is made possible by this increased surface area as evident by cleaner surface finishes on the fabric as shown in the micrograph, at decreased fiber pullout.

The highest improvement of 10 percent tensile strength seen in composite with 40 wt% of cotton fibers. It can also be attributed to enhanced interaction of matrix and fibers with further enhanced possibility of stress transfer. Hybridization of cotton fibers with the CNSL/epoxy resin is indeed showing synergy. In this case, the hybridized matrix has compensated for the weaknesses of individual constituents by making use of the strength of hybrids. The same trend is also witnessed in previous work wherein the enhanced bonding at the interface among FRPs provided enhanced tensile properties.



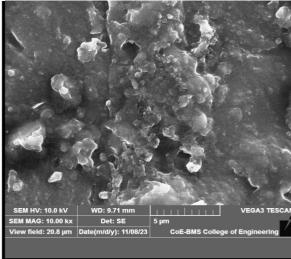
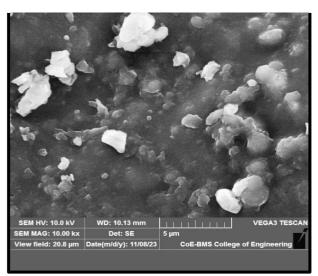


Figure 6. Tensile fractured surface at 10kx magnification (a) sample 2 (b) sample 3.

2025, 10(34s) e-ISSN: 2468-4376

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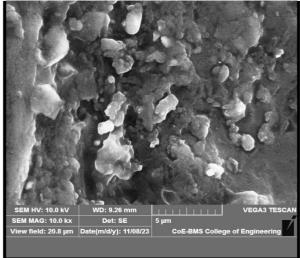


Figure 7. Tensile fractured surface at 10kx magnification (a) sample 4 (b) sample 5.

> Flexural test

The test is meant to reveal how well each composite sample's flexural strength. The flexural behavior of every sample composite with cotton fiber is shown in Figure 8. The sample with the highest strength is sample 5. When cotton fibers percentage enhances from 0% to 40%, the maximum improvement in flexural strength is 35%. This notable impact on flexural strength can be ascribed to the greater energy-absorbing ability of hybrid resin under load conditions. As a result, composites load transfer efficiency is raised. The interlocking action between the elements of the resin increases strength of composites. Such improvement in cotton fiber-reinforced CNSL/epoxy hybrid composites' flexural strength could be attributed to several significant reasons as provided below with an appropriate discussion.

In composite materials, adding flexural strength increases the load transfer efficiency between matrix and fibers. In the case of the cotton/CNSL epoxy composites, hybrid matrices improve more of the load transfer efficiency. A material composition with increased fiber concentration of 0% to 40% improves the material's ability to transfer flexibility across the composite structure. The strength of the composite is further enhanced by the cotton fibers, as these fibers provide better support to the matrix under bending loads. The energy-absorbing capability of the CNSL and epoxy matrix is very high when subjected to flexural loads, which leads to better resistance against bending forces. The hybrid matrix CNSL/epoxy also gives it a toughened structure with its toughness, which is able to absorb and dissipate energy effectively before failure. This hybrid matrix, in itself, is pretty much responsible for preventing brittle failure and leads to higher flexural strength. The relative increase in energy-absorbing capacities through the hybrid matrix is considerably responsible for the development of better flexural properties in a composite.

The interlock action between CNSL and epoxy resins forms a tightly bonded network that enhances the composite mechanical properties. This IPN would result in more stress transfer at the interface of fiber and matrix, which is important for enduring flexural deformation. It enhances the material's capability to resist bending forces through the formation of an IPN between hybrid resin components that contribute to increased flexural strength of composites. The inclusion of cotton fibers in the composite greatly enhances its stiffness and, by consequence, its flexural strength. The various natural fibers within the hybrid matrix are also capable of offering resistance to deformation under flexure as they function like load-bearing members. The growth of fiber content permits reinforcement fibers to come into contribution for the purposes of resisting loads of bending applied, hence increasing stiffness and more significantly raising the strength in flexure. At a content of 0% to 40% of cotton fiber, this directly contributes to more surface areas where bonding between fibers and the hybrid resin matrix can take place. A larger fiber-matrix interfacial area also contributes to better adhesion and, consequently, to enhanced flexural resistance within the composite. If the fiber-matrix bond is stronger, it aids in transmitting flexural load

2025, 10(34s) e-ISSN: 2468-4376

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applied to the matrix to the fibers, thus enhancing the flexural strength. Hybrid resin systems tend to minimize the formation of micro cracks and other defects in the composite during flexural loading. CNSL, being able to act as a crack Bridger, inhibits the cracks propagation in matrix. Hybrid matrix decreases such defects from appearance and, hence, it favors flexural characteristics as the structural integrity is preserved during bending. Crack resistance of the hybrid matrix is very important to enhance the total flexural strength within the composite material.

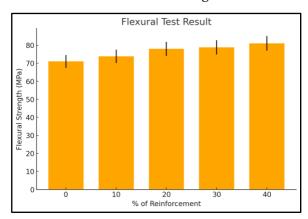


Figure 8: Flexural test result Graph of Flexural Strength Vs percentage of reinforcement

Izod impact test

Figure 9 displays the impact test outcome, which is used to evaluate each composite's capacity for energy absorption. When the cotton fibers percentage enhances from 0% to 40%, the maximum impact strength increases by 40%. The maximal strength needed to fracture the composites is the absorbed impact strength (kJ/m2), as determined experimentally. In contrast to the individual polymer matrices, because resin can support a larger impact load than single resin composites, it takes more energy to shatter.

The hybrid cotton fiber-reinforced CNSL/epoxy composite showed impact strength enhancement because of the following critical parameters relating to fiber-matrix interactions, energy dissipation mechanisms, and material characteristics. The following are six reasons for impact strength enhancement when cotton fiber concentration rises from 0% to 40%, and a discussion thereof is presented.

The hybrid resin system containing CNSL and epoxy has an improved energy absorption characteristic than individual resin systems. Two different types of resin combination will dissipate more energy in the impact, since each resin component can absorb and distribute stress differently. This leads to a higher impact strength since much more energy can be absorbed before fracture occurs in the composite. Hybridization of CNSL with epoxy yields a more intense and tougher matrix, which can support load because of greater impacts in magnitude, thereby enhancing the overall impact strength.

Cotton fibers increase the surface areas exposed to bonding between hybrid matrix and fibers. When cotton fibers are added, then the fibers and matrix interface are adhered strongly, so that stress can be better transferred upon impact. Robust adherence of the matrix and fibers is a prerequisite to absorb and distribute the energy transferred by an impact without premature failure of the composite. The robust interfacial bond reduces the probability of fibre pull-out and matrix cracking and hence increases the material's ability to repel impact loads.

The combination of CNSL and epoxy forms a synergistic effect that enhances the composites mechanical properties, with impact strength specially. CNSL was hard and flexible; it was bonded with epoxy, which had its structural rigidity and strength. The hybrid resin system, due to synergy, took in more energy under impact than single resin composites. Combining two different matrices balanced stiffness and toughness, and as a result, energy absorption for the hybrid composite was higher than that for single resins.

These cotton fibers are contributing more significantly to the toughness of the composite. Impact energy absorption of the composite has been increased by reinforcement due to cotton fibers when the content of fibers is raised from

2025, 10(34s) e-ISSN: 2468-4376

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o% to 40%. In the natural fibers like cotton, inherent toughness and flexibility are found, which are absorbing and dissipating the energy during impact. The fibers act as a crack-arresting agent during the impact event, thereby preventing crack growth and increasing the overall toughness of the composite.

The cotton fibers in combination with the hybrid resin system are very effective in preventing the propagation of cracks in the composite during an impact. Bridges crack quite well in the case of CNSL, where this additive stops the crack from growing. The inclusion of cotton fibers inside fills in the holes, essentially reinforcing the structure and thereby the crack cannot move further. As a result, the composite is tougher and can absorb more impact energy at the time of cracking. Crack propagation is significantly reduced and helps in improving the composite impact strength.

Cotton fibers, as a natural flexible fiber, are capable of absorbing the impact energy and dissipating it. Due to inherent flexibility, under impact, cotton fibers bend and deform without cracking and therefore the composite can take on higher impact forces. As the cotton fibre rises, the whole composite shows higher energy absorption as a result of an increase in impact strength. Flexing of the fibres and deformation on loading further allows them to take energy from impacts better than brittle, newly developed synthetic fibres.

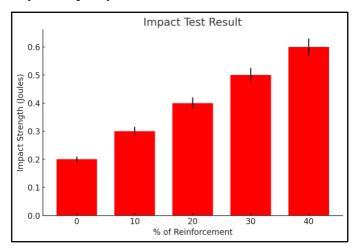


Figure9: Impact test result Graph of impact strength Vs percentage of reinforcement

CONCLUSIONS

This research looks into what changes when the inclusion of hybrid resin made of epoxy and CNSL to polymer materials that are reinforced with cotton fibre. Extensive testing and research have been doneto improve the composite specimen'sproperties. Here is list of the conclusions.

- The hybrid composite tensile strength expanded by 8.3% with the increase in concentration of cotton fibers from 0% to 40% concentration due to enhanced fiber-matrix adhesion as well as IPN formation.
- Flexural strength increase by 35% for the composite with 40 wt% cotton fibers, due to the absorption of more energy by the hybrid resin.
- The impact strength for 40 wt% cotton fibers containing composite was enhanced by 40% over a substantial increase in toughness with dissipation of energy within the hybrid resin system.
- IPN between CNSL and epoxy improved the mechanical property, thus showing better load distribution and stress transfer throughout the composite.
- Cotton fibers effectively participated in both crack bridging and toughening mechanisms, hence an overall improvement in the toughness and crack resistance was reported to be 10%.
- SEM analysis indicates a reduction in fiber pull-out and increased fiber-matrix adhesion within composites containing 40 wt% cotton fibers, therefore affecting the stress transfer and performance mechanically.

2025, 10(34s) e-ISSN: 2468-4376

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