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ORIGINAL PAPER

Effect of Amino Silane Grafted Cellulose and Kenaf Fibers in Mechanical, Impact Toughness and Drilling Characteristics of Epoxy Resin Composite

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Abstract
This research focused on effect of surface treated kenaf fiber as reinforcement and cellulose as filler in epoxy composites for mechanical, impact toughness and drilling characteristics. The laminates for this study are prepared by hand lay-up process and characterization was performed by preparing suitable samples with respect to ASTM standards. The mechanical characterization results show an increase in tensile strength of around 60% for silane treated reinforcement composites, while as-received reinforced composites give only a 45% increment for composite designation EKC2 (Epoxy + kenaf + Cellulose). Similarly, flexural and interlaminar shear strength (ILSS) tests show enhanced properties by 53% and 18%, respectively with a maximum impact resistance of 7.22 J for silane treated reinforcements. Drilling characterization shows smooth drilling from top to bottom due to the silane treatment and there is no delamination occurs. Such improved, strengthened materials are used in various industrial and household applications as well as from its drilling characteristics, it is clear that these natural fiber composites can be used in the automotive sector and for structural uses.

Keywords Grafting · Silane treatment · Natural fiber · Drilling test · Mechanical characterization

1 Introduction

Researchers from all around the world were really attracted to natural bio-polymers because of their different characteristics and multiple applications in a number of different fields. Among the many biopolymers discussed, cellulose is a versatile, renewable, affordable, plentiful, and well-studied bio-based polymer, whose contents in diverse lignocellulosic biomass range from a few percent to as high as 98% [1]. Similarly, natural cellulose, amongst the most naturally abundant polymers, has been widely used in chemistry, biology and biomedical sciences because of its renewability, biocompatibility, and biodegradability. Cellulose is a water-insoluble polymer with a rigid linear structure. Controlled cellulose biosynthesis allows arrangement of extensive linear chains which can be aligned side-by-side, creating fibers of great mechanical strength. Because the anhydroglucopyranose unit of cellulose contains reactive hydroxyl groups, it could be used to design specific polymeric materials. Consequently, there are many hydrogen bonds in its molecular structure. As a result, cellulose is difficult to dissolve in typical organic and inorganic solvents, limiting access in processing, modification, and application [2, 3]. The modification of cellulose by graft polymerization, which has been described in a variety of ways, provides a substantial pathway that combines the benefits of combining natural cellulose with synthetic macromolecules in a wide range of possible applications, including new materials for drug delivery devices, coatings, sorption agents, and membranes [3, 4].Numerous monomers have been grafted from cellulose via silane treatment and to enhance the surface of nanocellulose, a variety of processes can be performed, including the use of coupling agents, such as amino silane reagents. Aminosilanes possessing primary, secondary, and tertiary amines were also silane reagents that have been utilized for cellulose modifications. They have a hydrolytically

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sensitive core that can generate a silylated surface by reacting with hydroxyl groups or silanols [5, 6].

Many different natural fibers are employed in polymer matrix composites because they are economical, nontoxic, readily available, and satisfying to researchers as a biodegradable alternative to synthetic fibers [7, 8]. Natural fibers contain plant fiber such as cotton, hemp, jute, flax, kenaf, ramie, sisal, pineapple leaf fiber, and bagasse. Agrebi et al. [9] investigated the dielectric characteristics of pineapple leaf fiber and kenaf fiber reinforced phenolic hybrid composites treated with silane. The use of silane increased the interfacial adhesion between pineapple leaf fiber/kenaf fiber and phenolic resin, according to the authors, which will aid in the development of high-performance kenaf fiber/pineapple leaf fiber reinforced polymer composites for industrial applications. The dynamic mechanical behaviour of kenaf cellulosic fiber biocomposites: a comprehensive review on chemical treatments was done by Asyraf et al. [10]. Kenaf (Hibiscus cannabinus L.) is a versatile hibiscus species that is used to manufacture engineered timber, clothes, packing material, rope, and twine, according to the author's research. Kenaf is mostly composed of cellulose (about 70%), which accounts for its superior mechanical properties. To improve the fiber characteristics of kenaf fibers, they are chemically treated before being mixed with other polymer resins. The review also unveils other chemical treatments (e.g. zein and amino acid) and combined treatments on the fiber to improve the biocomposites' dynamic mechanical behaviour. Tee et al. [11] investigated the thermal characteristics of poly (lactic acid) composites with thermally grafted aminosilane onto kenaf produced cellulose. The thermal stability of the cellulose was improved after it was grafted with a pre-hydrolyzed APS coupling agent, according to the author. Natural fibres' non-homogeneous qualities, along with their anisotropic features, make natural fiber composites more difficult to machine than metal composites. Poor surface finishing, significant matrix removal, delamination, fiber pullouts, and subsurface damages were reported in traditional machining processes such as milling, turning, and drilling [12].Critical aspects of natural fiber composite manufacturing processes were investigated by Mei-po et al. [13]. The author came to the conclusion that poor wettability, poor bonding and deterioration at the fiber/matrix interface (a hydrophilic and hydrophobic effect), and fiber damage during the manufacturing process are the main reasons for composites' weakening. Dharmavarapu et al. [14] examined in the research, the role of silane surface treated Kevlar fiber and nano-silica and there failure analysis on inter-laminar shear strength and drilling process was studied. In the drilling process, the author determines that surface-modified epoxy composite designations have the highest dimensional stability than as-received designations. After the drilling operation, optical microscope fractography show no fiber pull-out or edge damage. Hence it's suggested that silane treatment on natural fiber is more effective than the non-treated fiber for machining process.

Based on the previous research, the silane treatment on natural fibre and particle has numerous advantages than the as-received reinforcements. Both the kenaf and cellulose have inevitable applications in the composite industries since they have high potentially, according to the previous literatures. It is emphasized that further research is needed to determine how silane treatment affects the mechanical, impact toughness, and drilling properties of kenaf fiber and grafted cellulose biocomposite. Thus, the current study focuses on how silane treatments affect cellulose and kenaf fibers when they are reinforced with resin. Mechanical and impact toughness, as well as drilling characteristics, are also revealed in this investigation. In this the composite laminates were manufactured using a hand layup procedure and the characterization was done according to ASTM standards. These environment friendly novel material-reinforced composites could be used to create beneficial items in both the household, structural and industrial sectors like door panels for automotive sector as well as in furniture and circuit boards as domestic purpose.

2 Materials and Methodology

2.1 Materials

Figure 1 shows the materials specifications used in this present study. The triethylenetetramine (TETA, Huntsman India Ltd. Mumbai., HY951), a low viscosity aliphatic amine having viscosity of 20 cps and density of 0.98 g/cm^3 was used as a curing agent for epoxy resin. Similarly, 3-Aminopropyltrimethoxysilane (APTMS) was used as silane grafting agent purchased from Sigma Aldrich USA. Figure 2 shows the real images of (a) Kenaf fiber and (b) cellulose microcrystalline powder.

2.2 Amino Silane Grafting of Natural Fiber and Cellulose

The amino silane treatment was used to prepare the grafted kenaf fiber and cellulose particles; in this procedure, the fiber and cellulose particles are briefly dipped separately in individual containers in an ethanol–water solution. A mixture of 95 percent ethanol and 5 percent water was gently stirred for 10 min. The appropriate amount of silane coupling agent, typically 2 percent concentration, was added drop wise to achieve a homogenous mixture, followed by 5 to 10 min of gently stirring. The fibers and cellulose particles were soaked for 10 min in an ethanol–silane solution. Through a manual process, pouring off the excess solution

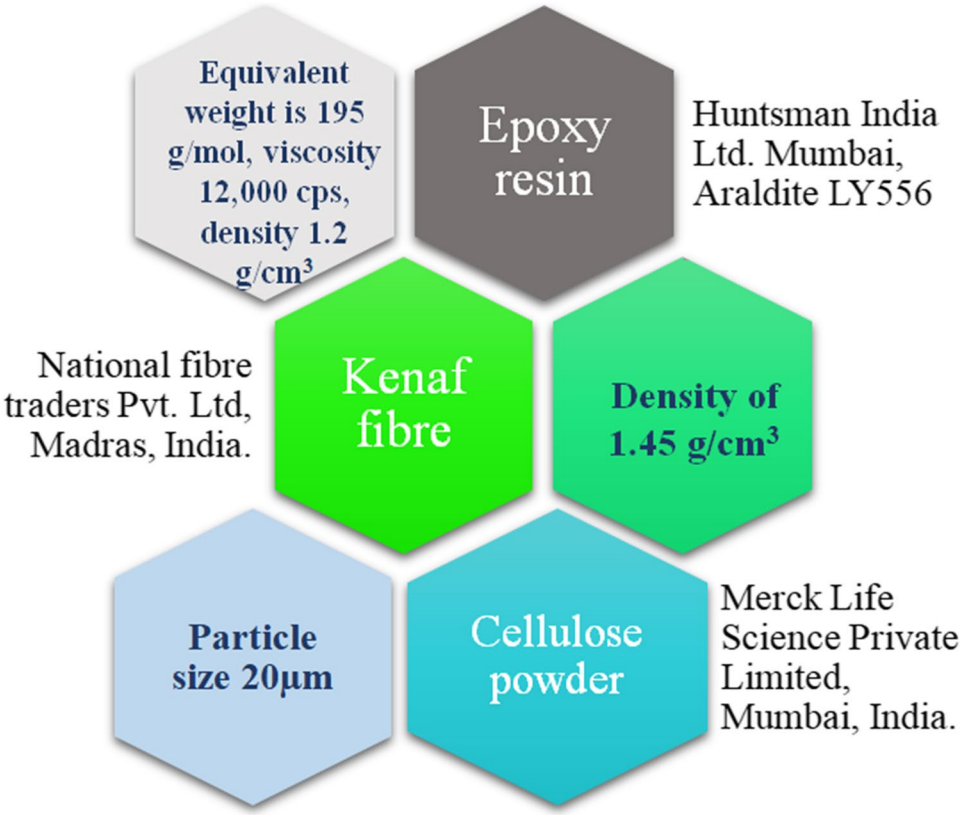


Fig. 1 Material with specification used in this study

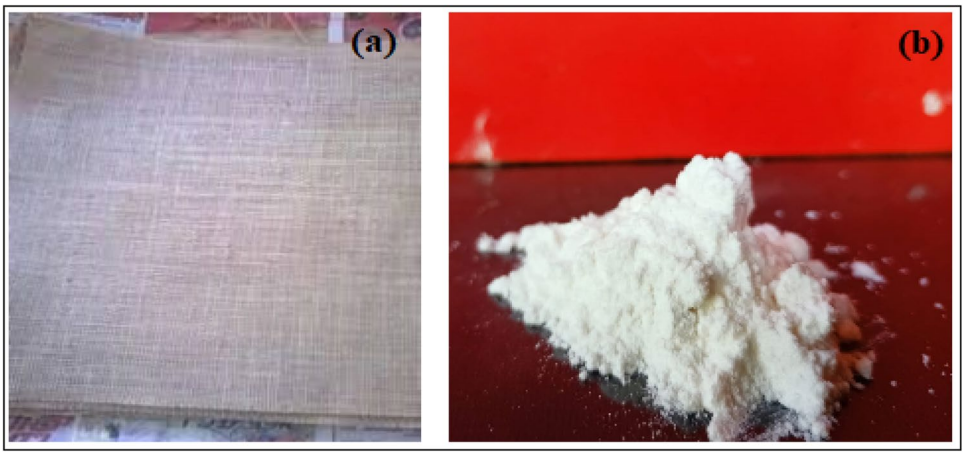


Fig. 2 Real image of (**a**) Kenaf fiber and (**b**) cellulose powder

was done, and the precipitated fiber and cellulose particles were removed from the aqueous solution. The precipitated fiber and particles were washed transiently with ethanol to remove excess silane and dried with 110 °C for 10 min in oven to remove moisture and forms Si–O–Si structures as shown in Fig. 3 [12]. The FTIR spectra for silane treated cellulose and fibres were represented in Fig. 3(a) and (b) respectively. This FTIR spectrum ensures the presences of amine after silane treatment.

2.3 Laminate Fabrication

Hand lay-up process was used to create natural fibre particulate composite laminates for this study. First, a generous coat of wax with a thickness of roughly 3 mm was applied to the mould. At room temperature, an epoxy resin was combined with cellulose particles of different volume percentages in a hardener. This viscous colloidal suspension was thoroughly stirred before pouring. After

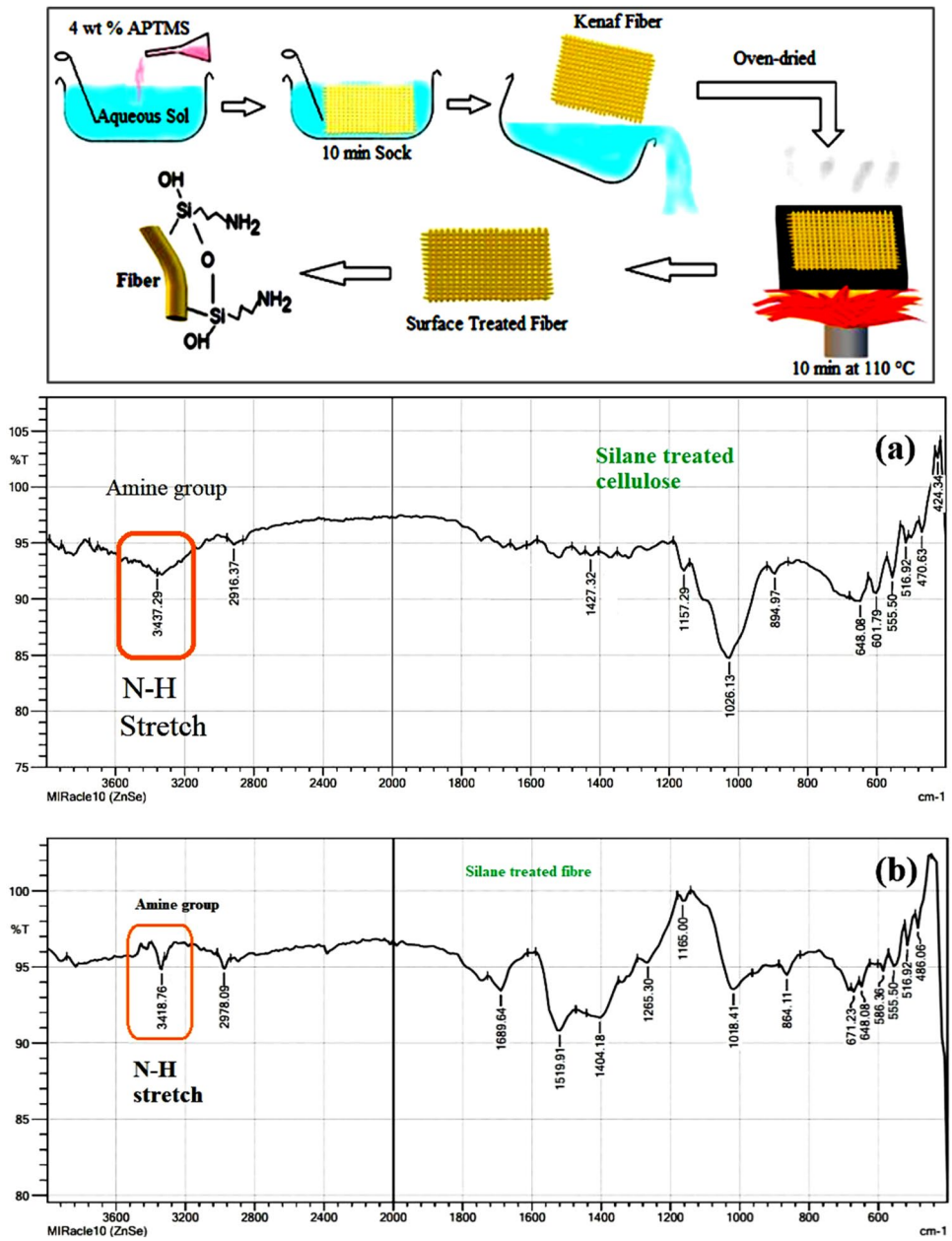


Fig. 3 Silane surface treatment on kenaf fiber and cellulose with FTIR spectra

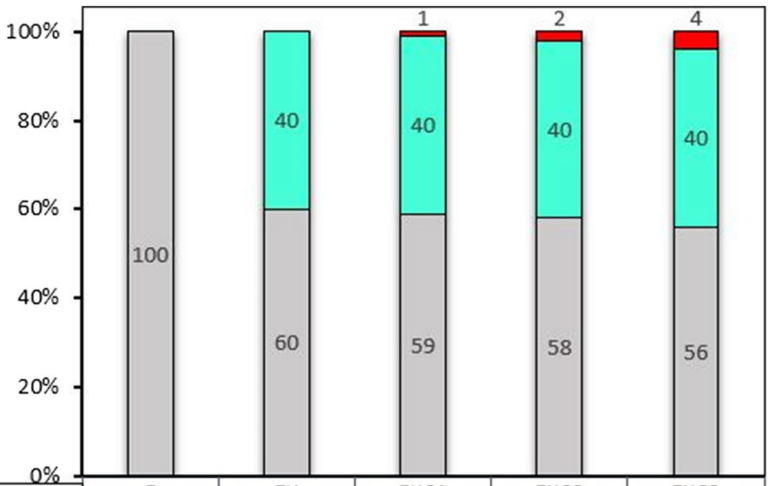
that, a homogeneous combination of resin and filler was created and poured into the mould. 40 vol. % of Kenaf fiber mats were laid one by one in the poured resin and gently pressed. Cotton roller was used to release trapped air bubbles between two layers. The extra resin was manually cleaned out, and gravity force was used to achieve a consistent composite thickness. The curing was done at room temperature for about 24 h and post cured at 120 °C using a hot oven for 48 h. Figure 4 shows the final product of fabrication process [13]. This laminates were fabricated for both as-received and silane treated fiber as well as particles. Figure 4 shows martial composition and designation for various composites fabricated.

3 Characterization of Composites

After curing in the oven, the epoxy biocomposites are next subjected to a close inspection to look for any potential surface defects. Once passing visual inspection, composites are machined. Appropriate ASTM test specimens were cut from cured composite panels using a Maxiem water jets, 1515, KENT, abrasive water jet machine.

3.1 Mechanical Testing

The tensile, flexural, and inter-laminar shear strength (ILSS) of bio composites were tested based on ASTM-D 638, 3039,



Composite designation	E	EK	EKC1	EKC2	EKC3
Cellulous particles vol.%	0	0	1	2	4
Kenaf Fiber vol.%	0	40	40	40	40
Epoxy vol.%	100	60	59	58	56

Fig. 4 Composite designations for various martial composition. Note: E-Epoxy resin, K-kenaf fiber, C-Cellulose particles

Table 1 Process parameters used for drilling

Spindle Speed rpm	Constant feed m/s	Flutes diameter mm	No. of flutes
1000	1.5	8 and 4	2

790 and 2344 respectively. A universal testing machine (INSTRON 4855, UK) with traverse speed of 1.1 mm/sec was used for analysing the composites fabricated. The Impact strength of bio-composite was tested based on ASTM-D 256 using a mini impact tester of loading capacity 20 J (Krystal equipment Ltd., India). Five in distinguishable test specimens were tested to find the average.

3.2 Drilling Characterization

Throughout the drilling operation, the drilling process was used to determine laminate damage, which was then followed by microstructure analysis. For this work, the process parameters were a radial drilling machine (HMT, India) with 1000 rpm spindle speeds. The drill tool was manufactured of HSS. Holes were drilled with maximum speed of 1000 rpm with constant feed of 1.5 m/s (Table 1).

3.3 Fractography

A scanning electron microscope HITACHI, S-1500, JAPAN has been used to examine the fractography of damaged surfaces of the kenaf fiber and cellulose particle epoxy composites. Before scanning, the fractured surfaces of the samples were coated with gold to prevent charging and improve image. An optical microscope (Moticam L 312) with a 25 × lens zooming capacity was used to analyse the surface morphology of the drilled holes.

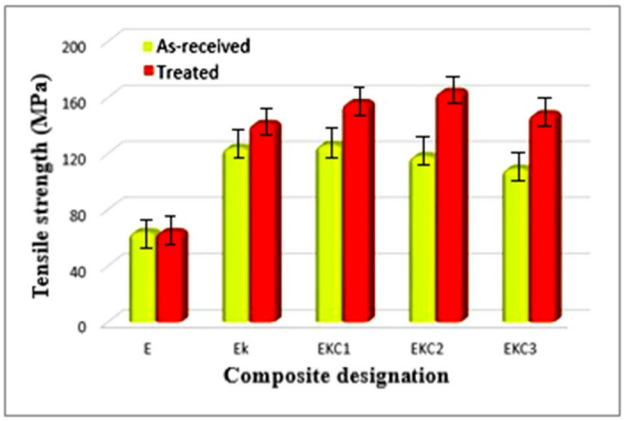


Fig. 5 Tensile properties of various composite designations

4 Results and Discussion

4.1 Mechanical Properties

The tensile test results for as-received and surface-treated kenaf fiber, as well as cellulose microcrystalline particles for various composite designations, are shown in Fig. 5. The tensile strength of composite designation E is low, at around 64 MPa. AS composite designation E contains

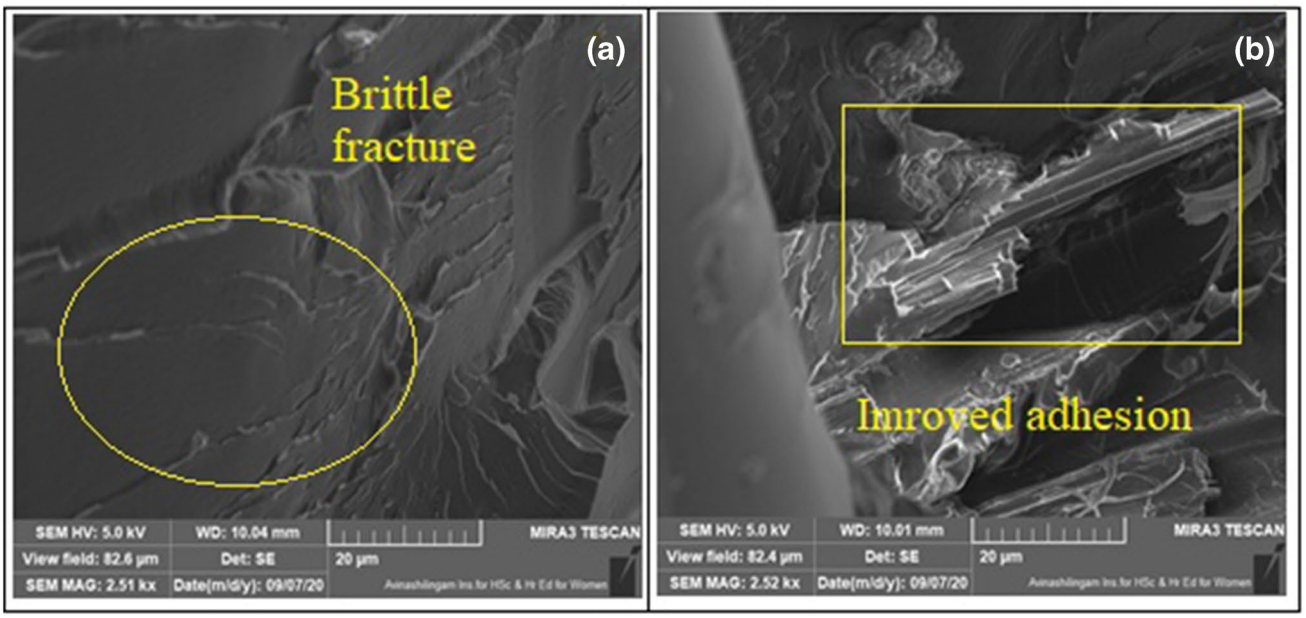


Fig. 6 (**a**) Brittle fracture for composite designation E and (**b**) Improved adhesion for composite designation EK

pure epoxy and absences of reinforcing materials causes such result. As illustrated in the SEM picture in Fig. 6(a), this composite designation demonstrates brittle fracture due to pure epoxy. However, when further as-received and surface-treated kenaf fibers were added by 40 vol.%, tensile strength increased by 48% and 54%, respectively. The composite designation EK represents this enhancement. This improvement is due to the kenaf fiber's load-sharing property, which distributes the load from the matrix and minimizes stress concentration [14]. However, as-received fiber has a lower tensile strength than fiber composites that have been surface treated. The reason for this is weak interfacial bonding between the fiber and the matrix material as received, but the surface-treated fiber's adherence to the epoxy matrix improves due to the silane treatment as shown in Fig. 6(b).

Similarly, the addition of cellulose particles to composite designations EKC1, EKC2, and EKC3 was increased by 1 vol. %, 2 vol. %, and 4 vol. %, respectively. For composite designations EKC1, EKC2, and EKC3, improved tensile strength was seen in the range of 49%, 45%, and 41%, respectively for as-received fiber and 58%60% and 56% for silane treated fibers. Because particles improve the bonding between fiber and resin, tensile strength increases. However, as the volume percent grew by more than 1vol. %, particle amalgamation was detected, resulting in a loss in tensile strength [15] for composite designations EKC2 and EKC3 as compared to EKC1. Silane treated cellulose shows less clustering effect than as-received cellulose particles.

The flexural test results for as-received and silane treated kenaf fibre, as well as cellulose microcrystalline

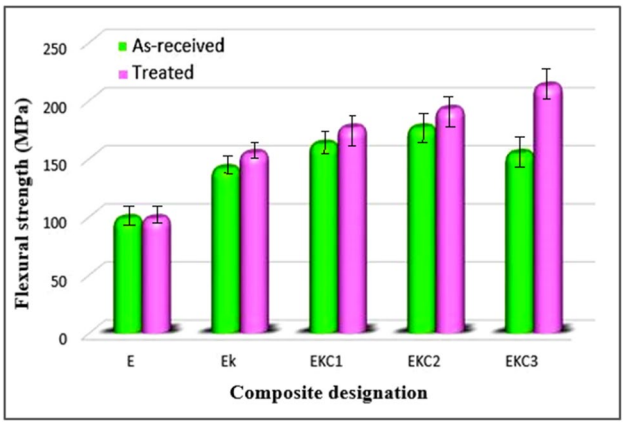


Fig. 7 Flexural strength for various composite designations

particles with epoxy matrix composites of varied compositions, are shown in Fig. 7. The composite designation E has a flexural strength of roughly 98 MPa because it is made entirely of epoxy and has no reinforcements. Furthermore, when 40 vol. % kenaf fibers were added, flexural strength increased by 30% and 36% for as-received fibre and silane treated fibre, respectively. This is due to the fiber's ability to withstand the bending load applied by the matrix material [16]. However, untreated fiber has lower values than silane-treated fiber. Because of the OH bond on the surface of silane-treated fibres, they interlock well with epoxy resin. However, further inclusion of cellulose particles enhances the flexural strength for composite designations EKC1, EKC2 and EKC3. It shows

around 39%, 44%, and 36% for as-received cellulose particles and for silane treated cellulose about 43%, 48%, and 53% for composite designations EKC1, EKC2, and EKC3, respectively. The reason behind this is that the cross-linking density of epoxy resin increases at a higher level due to the cellulose-OH group attraction obtained by silane treatment [17].

Figure 8 shows inter laminar shear strength for kenaf fiber and cellulose particles epoxy matrix composites. This figure shows both results for as-received reinforcements and silane treated reinforcements. As previously discuss composites designation 'E' possesses pure epoxy hence ILSS will comes 0 as their lack of reinforcements to transfer the load. However, kenaf fiber were included by 40 vol. % in epoxy matrix inter laminar shear strength increased around 18 MPa and 26 MPa. It shows the bonding between the fibers and matrix and also indicates that the breaking load of the specimen is increased by addition of fiber which transfers the load by shearing. Moreover, when cellulose particles inclusion is done by 1%, 2% and 4%laminates strength is increased. For composite designation EKC1, EKC2 and EKC3 enhancements are observed around 18%, 10% and 0% for as-received particles respectively. Similarly, for silane treated cellulose particles this increment is seen around 13%, 18% and 16% for composite designation EKC1, EKC2 and EKC3respectively. As-received particles shows lower values due to the lustring of particles but silane treated particles composites shows higher values due to the uniformly dispersion of particles as result of treatment. This enhancement in strength because of the existence of the filler particles which opposed the shearing of laminates [18].

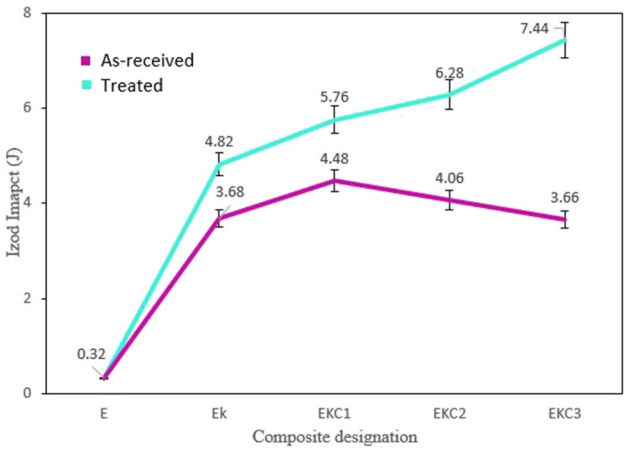


Fig. 9 Izod Impact testing characteristics

4.2 Izod Impact Test

It should be observed that the Izod impact toughness gives coherent results, as shown in Fig. 9. The impact toughness of pure epoxy resin is 0.32 J, which is relatively low shown for composite designation E. The lack of load-bearing micro-mechanisms of epoxy matrix materials is due to its inadequate energy absorbing capacity. When further inclusion of kenaf fiber by 40 vol. % is done the impact toughness of natural fiber composites increased dramatically. This increment is seen in composites designation EK around91% and93%for as-received and silane treated kenaf fiber respectively. The sudden load-bearing performance was increased by adding untreated kenaf fiber to the epoxy resin. But silane treated fiber shows higher increment

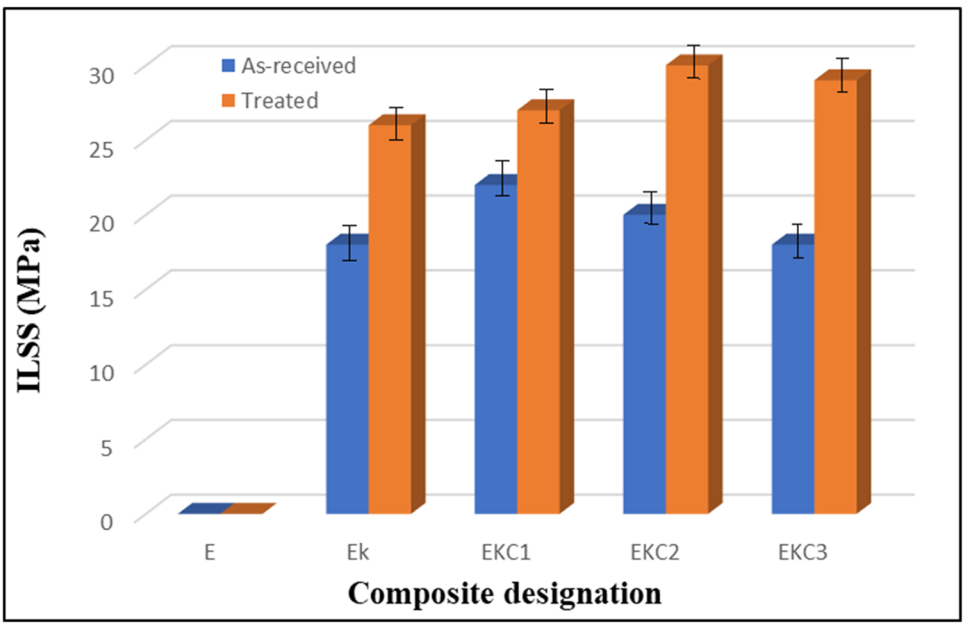


Fig. 8 Inter laminar shear strength for various composite designation

because it transfer load smoothly thought the resin system. The existence of fiber monitors a rapid load and minimizes the stress intensity factor inside the resin system, resulting in a higher rate of energy absorption [19]. However, by introduction of cellulose particles in natural fiber composites enhances the impact toughness for composite designation EKC1, EKC2, and EKC3. This increment is around 91% to 92% for as-received particulates composite and 94% to 95% for silane treated kenaf fiber. This improvement is the reason for improved bonding of fibre with matrix material as shown in Fig. 10(a), which could transfer the sudden load very smoothly throughout the matrix by inclusion of cellulose particles [20]. Cellulose particles reduced the voids between resin materials and improved the compactness. It leads to store high energy by improving interlocking mechanism [21]. For silane treated particles impact toughness is high due the surface treatment which improves bonding as illustrated in Fig. 10(b) than as-received cellulose particles.

4.3 Drilling Process

The kerf widths of 8 mm drilled holes and 4 mm drilled holes for as-received and silane treated reinforcements are shown in Table 2. As shown for composite designation E, the kerf width for 4 mm and 8 mm diameter holes is approximately 4.25 and 8.26 mm, respectively. The increase in kerf width is the reason for the lack of reinforcements in composite designation E, which consists of pure epoxy. This due to the vibrated penetration of drill bit material and the hard exclusion of polymer material [22, 23]. When using as-received kenaf fiber in composite, for 4 mm and 8 mm drill bits, it shows the kerf width is around 4.18 mm and 8.32 mm, respectively, and 4.09 mm and 8.14 mm when using silane treated kenaf fiber. Whenever the drill bit strikes the specimen's upper part with as-received fiber, the top layer is crippled, and fiber pull-out occurs [24, 25], as shown in Fig. 11(a) for the 8 mm drill bit. But in the case of silane treated kenaf fiber, the bonding between fiber and matrix is improved due to surface treatment, and hence smooth drilling from top to bottom is observed [26, 27] as shown in Fig. 11(b) for an 8 mm drill bit. Moreover, when cellulose particles are added at 1%, 2%, and 4%, the kerf width also shows accuracy with the smooth surface, and no crack is found inside of the hole. For as-received cellulose particles, the kerf width comes around 4.12 mm, 4.08 mm, and 4.06 mm after drilling by a 4 mm drill bit, and for 8 mm drill bit, it comes around 8.22 mm, 8.16 mm, and 8.11 mm. The variances in dimension are the reason cellulose particles increase the bonding but creating a clustering effect and improving the interfacial cracks as shown in Fig. 11(c) for a 4 mm drill bit. Silane-treated cellulose particles, on the other hand, dispersed uniformly across natural fiber composites,

Table 2 Kerf width for various composite designations

Composite designation	4 mm		8 mm	
	As-received	Treated	As-received	Treated
E	4.25	4.25	8.26	8.26
EK	4.18	4.09	8.32	8.14
EKC1	4.12	4.06	8.22	8.04
EKC2	4.08	4.03	8.16	8.06
EKC3	4.06	4.04	8.11	8.03

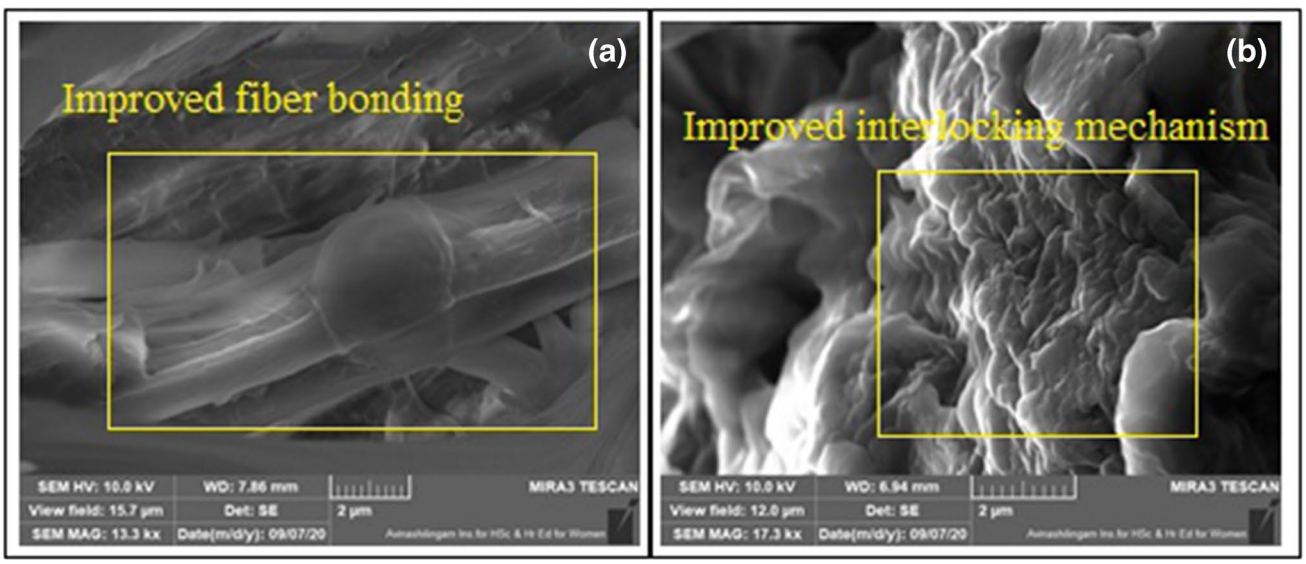


Fig. 10 (**a**) Improved fiber matrix bonding and (**b**) silane treated cellulose with improved interlocking ability

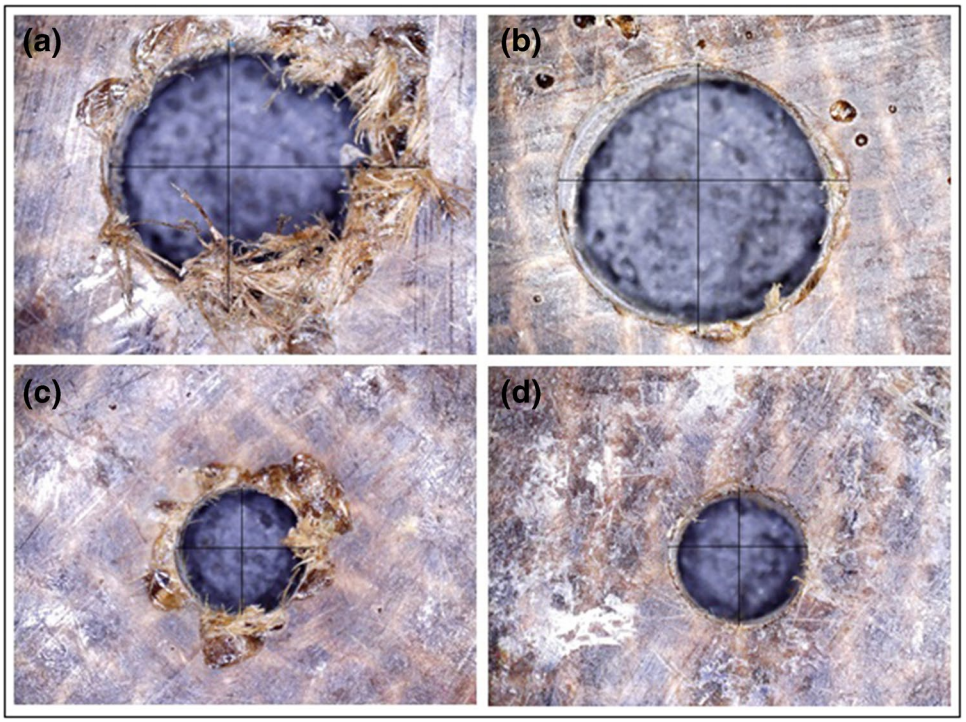


Fig. 11 Optical microscope images of top surface of the drilled portion

improving bonding. The kerf width for silane-treated cellulose particles drilled by an 8 mm drill bit was 8.04 mm, 8.06 mm, and 8.03 mm, indicating precise specifications. The drilled top surface for silane-treated cellulose particle composites is shown in Fig. 11(d) using a 4 mm drill bit. It demonstrates that there was no such delamination on the drilled hole edge. There was no detection of fiber pull-out, indicating enhanced fiber adherence to the matrix [28].

5 Conclusions

The amino silane grafted cellulose particles and kenaf fiber epoxy composites were fabricated using the hand layup process and characterized as per ASTM standards. After characterization, it is seen that the silane treated kenaf fiber composites show higher results than the as-received natural fiber composites. For mechanical characterization, as-received reinforced composites show values increased by around 49%, 44%, and 18% for tensile, flexural, and interlaminar shear strength, respectively. While silane treated kenaf fiber and cellulose particulate matrix composites give the highest results, with around 58%, 53%, and 18% for the same tensile, flexural, and interlaminar shear strength tests. The reason for this is weak interfacial bonding between the fiber and the matrix material due to as-received reinforcements, but the surface-treated fiber's improved adherence ability to the epoxy matrix due to the silane treatment. The Izod impact test shows the maximum impact resistance up to 7.44 J for silane-treated reinforced composites. For silane-treated particles, impact toughness is high due to the surface treatment, which improves bonding and leads to the storage of high energy by improving the interlocking mechanism. The kerf morphology of composites that were drilled, which included silane-treated kenaf fiber and cellulose particles by 2 vol. %, indicated superior dimensional stability. Peel off, pull-out, and a poor finish on the drilled surface are shown in the composite designations EKC1 for received reinforcements. As an outcome, it is suggested that for better performance of natural fiber particulate composites, silane treatment is necessary. And with such silane treatments improved in mechanical and drilling properties, composite materials could be used in automobile body parts and air products manufacturing industries.

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Authors' Contributions Kaliappan S, Arunadevi B – Research conduct and formation of results.

Sateesh N, Nookaraju B.Ch – Testing and drafting of manuscript.

Data Availability No data available to deposit as private.

Declarations

Ethics Approval Not applicable.

Conflicts of Interest/Competing Interests There is no conflict of interest by any form for this manuscript.

Compliance with Ethical Standards Yes.

Consent to Participate Yes.

Consent for Publication Yes.

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