Effect of alkali treatment on the mechanical and interfacial properties of woven jute and kenaf fiber reinforced epoxy composites

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Abstract. The natural fibers like jute and kenaf have been taken as sustainable reinforcement of polymer composites as they are low density, renewable and possess good specific strength. They, however, have minimal ability to bond to hydrophobic polymer matrices due to their hydrophilic character. This paper has used the woven jute and kenaf fibers as a subject to the alkali treatment by using sodium hydroxide (NaOH) solution to enhance interfacial bonding between fibers and a matrix in the epoxy-based composites. A hand lay-up procedure was used with compression molding to make composite. The mechanical properties, tensile, flexural and impact strength were checked according to the ASTM standards. The fracture surfaces were scanned using the Scanning Electron Microscopy (SEM) which was used to assess the fiber- matrix interfacial characteristics. The alkali-treated fiber composites were found to have immense enhancement on mechanical strength, as well as decreased void content, in comparison to the untreated samples. The data show that the alkali treatment is an effective method used to eliminate surface impurities, hemicellulose, and lignin, which increases the interfacial bonding and effectiveness of load transfer in woven natural fiber composites.

1 Introduction

Over the past years, natural fiber reinforced polymer composites (NFRPCs) have gained a lot of attention due to the world trend on sustainability of materials and green manufacturing technologies. The urgency to substitute synthetic fibers, including glass, carbon, and aramid, with environmentally friendly ones grew more topical with the environmental issues, depletion of fossil energy, and the necessity to have biodegradable materials. The natural fibers, which are formed by plants, animals, or minerals, have shown a lot of potential in the polymer matrices reinforcement due to biodegradability, low density, renewability, cost-effectiveness and relatively good mechanical properties. Jute, kenaf, among other types of natural fibers, have been the most popular candidate fibers to be used in semi-structural and

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industrial composite applications because of their abundancy, moderate strength, and easy processing ability [1].

The natural fiber composites are materials whereby polymer matrices, both thermosetting and thermoplastic are strengthened with plant based fibers. Applications of such composites solve the growing need of lightweight and sustainable composite materials in the automotive, packaging, and building, and aerospace sectors. NFRPCs have a reduction of 20-30 percent of weight when compared to traditional glass fiber composites, it also has reduced tool wear when processed, and reduced energy is spent through the production process. In addition, natural fibers have a much lower carbon footprint since their growing consumer carbon dioxide in the atmosphere counterbalances the production of the composite. Nevertheless, in spite of these strengths, the biggest weakness of natural fibers is that they cannot be used in conjunction with hydrophobic polymer matrix, high moisture absorption, and low thermal stability. All of these lead to the diminished strength of the fiber-matrix interfacial strength that results into early failure and ineffective performance in the long term. Therefore, it is essential to learn and enhance the surface properties of fiber in order to produce composites with excellent mechanical and environmental characteristics [2].

Lignocellulosic bast fibers (jute, Corchorus capsularis and kenaf, Hibiscus cannabinus) are mainly cellulose-based, hemicellulose-based, lignin-based, pectin-based and wax-based fibers. The tensile strength and stiffness are provided by the cellulose component, the hemicellulose has an influence on flexibility, absorption of moisture, and the degradation behaviour, and the lignin does the same. Jute usually has a cellulose content of 60-65, hemicellulose content of 12-14 and lignin content of 10-12, whereas kenaf has higher cellulose content of 70-72 and lower lignin content, which is translated to higher intrinsic strength and thermal stability. The two fibers have hierarchical microfibrillar structures and have many hydroxyl (-OH) groups on their surfaces rendering them very hydrophilic. In cases where these hydrophilic fibers are incorporated in hydrophobic matrices such as epoxy, the surface energy mismatch results in a poor wetting of the material, as well as, poor interfacial bonding. This incompatibility causes fiber pull-out, formation of voids, and stress debonding causing the poor transfer of loads. Thus, jute and kenaf require modification of their surface to be used as a successful reinforcement, it is necessary to change their chemistry and morphology in order to be bonded to the polymer matrix more effectively [3].

Surface modification of natural fibers is oriented to the changes in the chemical composition and the surface topography to obtain better compatibility with the polymer matrix. Different chemical, physical, and biological treatments have been experimented with such alkali treatment (mercerization), silane coupling, acetylation, benzoylation and plasma treatment. Out of them, the alkali treatment with sodium hydroxide (NaOH) is regarded as one of the most efficient, economical, and scalable approaches. Part of the alkali treatment process involves the removal of amorphous compounds including: hemicellulose, lignin, pectin and wax that increase the content of crystalline cellulose and release more hydroxyl groups. Surface roughness is also promoted by the elimination of surface impurities which increases mechanical interlocking with the polymer matrix. Hybrid composites, though they consist of two or more fibers of a different type, have attracted increasing attention since they enable fine-tuning of the mechanical performance characteristic of individual fibers through the balancing of their strengths and weaknesses [3]. Jute and kenaf fibers can be combined to produce composites that are of good balance in terms of stiffness, toughness and cost efficiency. Jute has better dimensional stability and stiffness compared to kenaf but the former has inferior impact and energy absorbing ability. Dispersed fibers and the enhancement of the isotropy of the laminate structure is also facilitated by hybridization. Nevertheless, to utilize hybrid synergy to maximum, the two types of fibers should demonstrate similar surface activity with the epoxy matrix. Thus, alkali treatment should appear to boost the

bonding attributes of both jute and kenaf at the same time resulting in the homogeneous interfacial bonding and elevated load-sharing ability [4].

The main aim of the current case study is to examine the impact of alkali treatment on the surface morphology and mechanical characteristics of woven jute and kenaf fiber reinforced epoxy composites. The objective of the research is to increase interfacial adhesion of the natural fibers with the epoxy matrix by modifying the surface and hence, increase the overall structural integrity and performance of the composites developed. To achieve this, woven jute and kenaf fabrics were treated with sodium hydroxide (NaOH) to eliminate any surface impurities e.g. hemicellulose, lignin and wax, which normally inhibit a good bond with the polymer matrix. The treated and untreated fibers were then used to make epoxy-based laminates using hand lay-up technique with compression molding. The mechanical characteristics of the composites such as tensile strength, flexural strength and impact strength using ASTM standards were checked to ascertain the effect of alkali treatment in the composites performance. Moreover, Scanning Electron Microscopy (SEM) was used to determine the interfacial bonding nature and fracture mode in order to relate microstructural features to the available mechanical properties [5].

2 Materials and methods

In this paper, the reinforcement was made under the form of woven jute and woven kenaf fabrics made of natural fibers because of their availability, renewability and beneficial mechanical properties. The fabrics were sourced to a certified manufacturer in India, in which the quality of the fibers was uniform and the weave density was consistent. Epoxy resin (LY 556) and hardener (HY 951) that is distributed by Huntsman Advanced Materials were chosen as the matrix system because it has very good adhesion, chemical resistance, and dimensional stability. The weight ratio was kept 10:1 of epoxy to hardener as it was recommended by the producer [6]. The process was carried out using sodium hydroxide (NaOH) pellets of analytical purity and distilled water in the neutralization and cleaning, and distilled water was employed for neutralization and cleaning purposes.

The alkali treatment of woven jute and kenaf fabrics was done (mercerization) using a solution of 5 wt% of NaOH in order to improve interfacial adhesion with an epoxy matrix. The fabrics were allowed to soak in the solution of alkali and stirred with intervals in order to treat them evenly. The soaking was done in 4 hours in room temperature (25 ± 2°C). The fibers of the alkali solution were then carefully rinsed with distilled water until a neutral pH (≈7) was reached to eliminate traces of NaOH and dissolved impurities. The washed clothes were then dried in an oven using hot air at 60degC to remove moisture. This was to treat out amorphous material like hemicellulose, lignin, and surface waxes, and result in an increase in surface roughness and more reactive hydroxyl groups on the fiber surface. These changes have been reported to enhance the wettability of fibers, mechanical interlocking, and interfacial adhesion with the polymer matrix [7].

The hand lay-up technique was used to produce composite laminates and compression molded to obtain homogenous thickness and consolidation. The untreated and treated woven jute and kenaf fabrics were cut into desired sizes (300 x 300 mm2) and dried before the lay-up. Epoxy resin and hardener were well blended in a ratio of 10:1 and were degassed under vacuum to get rid of air bubbles. A roller was then used to apply the resin mixture to each layer of fabric uniformly in order to make the fibers fully moist. The laminates were made either in individual (jute/epoxy and kenaf/epoxy) or hybrid (jute + kenaf/epoxy) format by the alternating arrangement of the woven mats. Wt percent fraction of fiber content was controlled at about 40 +- 2 wt% in all the specimens to allow a fair comparison of the treated and untreated composite [8].

The stacked layers were laid-up and then placed in a steel mold after which compression at a pressure of 5 Mpa was applied to the mold during 24 hours of room temperature curing. The curing process was performed in an oven at 80degC over a 3 hour period so as to ensure that the epoxy resin would crosslink fully. The demolding of the fabricated laminates was done carefully followed by trimming and conditioning of the laminates at ambient temperature during 48 hours, and then the laminates were tested. The hand lay-up technique was used to produce composite laminates and compression molded to obtain homogenous thickness and consolidation. The untreated and treated woven jute and kenaf fabrics were cut into desired sizes (300 x 300 mm2) and dried before the lay-up. Epoxy resin and hardener were well blended in a ratio of 10:1 and were degassed under vacuum to get rid of air bubbles. A roller was then used to apply the resin mixture to each layer of fabric uniformly in order to make the fibers fully moist. The laminates were made either in individual (jute/epoxy and kenaf/epoxy) or hybrid (jute + kenaf/epoxy) format by the alternating arrangement of the woven mats. The amount of fiber weight fraction was kept at around 40 +- 2 wt% of all specimens so that there was fair competition between treated and untreated composites [9].

To examine the microstructural and interfacial characteristics, Scanning Electron Microscopy (SEM) was performed on the fracture surfaces of tensile specimens. Small portions from the fractured ends were sputter-coated with a thin layer of gold to enhance surface conductivity before imaging. SEM analysis was conducted using a field-emission scanning electron microscope operating at an accelerating voltage of 15 kV. The images were captured at various magnifications to study the extent of fiber pull-out, debonding, and matrix adherence. The morphological features observed in SEM images were correlated with mechanical test results to interpret the effectiveness of alkali treatment in improving fiber–matrix adhesion. Flowchart of the present work path is shown in Figure 1.

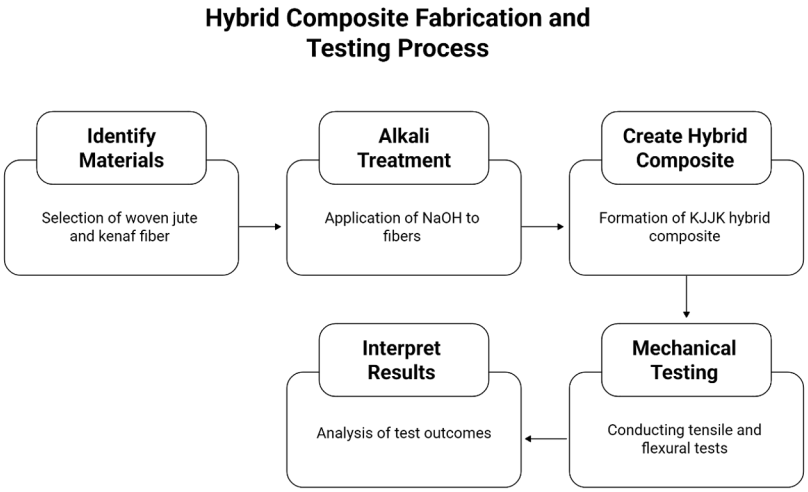

Fig. 1. Flow chart of present study.

3 Results and discussions

The woven natural fiber reinforcements used in the present study are shown in Figure 2(a). The fabrics consist of woven jute and woven kenaf, both selected for their mechanical

integrity, renewability, and availability. Its woven structure can be used to provide the ability to carry loads in a multidirectional manner and great stability of dimensions when fabricating the composites. The fabrics of jute are relatively rough and crosses in yarns can be observed, but kenaf fabrics are fine-tuned and homogenous in weaving. The variations in weave structure and yarn density affect the resin penetration and interfacial bonding behavior when composite processing is done. Figure 2(b) represents the test specimens that have been made out of the fabricated laminates, the arrangement of the composite shown will be in the KJJK order, where K represented kenaf and J represented jute. This mixed piling system was to be used to create a system that would be tough and stiff at the same time, comparable to kenaf, and jute. The other stacking also facilitates the enhancement of the loading between the layers, which further increases the fracture resistance and delamination resistance to stress. The appearance of the uniform fiber structure and imperfect laminate surface are the visually observable parameters that allow to say that the epoxy resin was properly impregnated and the composite structure was successfully consolidated. Surface morphology and interfacial properties of alkali-treated KJJK hybrid composite were investigated with the help of Scanning Electron Microscopy (SEM) demonstrated in Figure 3(a-c). The respective Energy Dispersive Spectroscopy (EDS) is also presented in Figure 3(d). These studies give information on the influence of fiber surface treatment on matrix adhesion and the overall interfacial quality.

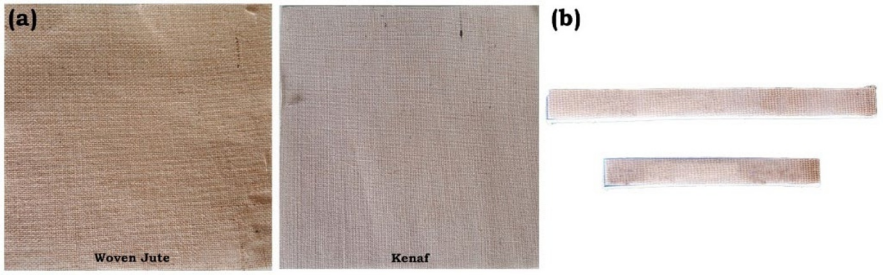

Fig. 2. (a) Woven jute and kenaf fabrics used as reinforcements, and (b) Prepared composite specimens in KJJK stacking (K = kenaf, J = jute).

Figure 3(a) shows that in the case of the untreated composite surface, the resin has not covered them uniformly and the microvoids can be seen on the surface and the interfacial wetting between the fiber and matrix is poor. The micrographs show that there is evident evidence of fiber pull-out and bonding which means that there is weak adhesion due to the appearance of surface impurities like waxes, hemicellulose, and lignin. These impurities serve to block the interlocking of the hydrophilic fibers of the hydrophobic epoxy framework to allow the transfer of loads during tensile loading [10]. Figure 3(b), on the other hand displays the morphology of the alkali-treated KJJK composite whose fiber surface is far more clean and rough. NaOH treatment removes amorphous constituents, which increases the roughness of surfaces, resulting in an increased ability of the wetting process of the fiber by the epoxy resin. Consequently, the composite surface treated is less bulky, has smaller number of voids and the fiber pull-out is minimal, which indicates enhanced fiber-matrix bonding. This increased adhesion increases the efficiency of transfer of stress between the matrix and the reinforcing fibers resulting in increased mechanical strength [11].

Figure 3(c) gives further a high-magnification SEM image of an interface with a well-adhered fiber-matrix after alkali treatment. The close interaction of the fiber surface with the epoxy resin is a confirmation of the removal of surface contaminants and factor of its successful chemical modification. The presence of cracks observed around ends of the fibres seems to indicate that there is a propagation of the same through the matrix as opposed to the interface which is indicative of a stronger interacial bond. Figure 3(d) was needed to establish

the presence of essential elements like carbon (C), oxygen (O), sodium (Na) and silicon (Si). High carbon and oxygen content is a symbol of the organic cellulose support of the fiber and epoxy resin, and the trace of sodium represents the residual sodium hydroxide as a by-product of the alkali treatment procedure. The fact that the impurities were detected with the lowest level also confirms that the alkali treatment indeed did clean the fiber surface hence adding to the improved bonding properties [12].

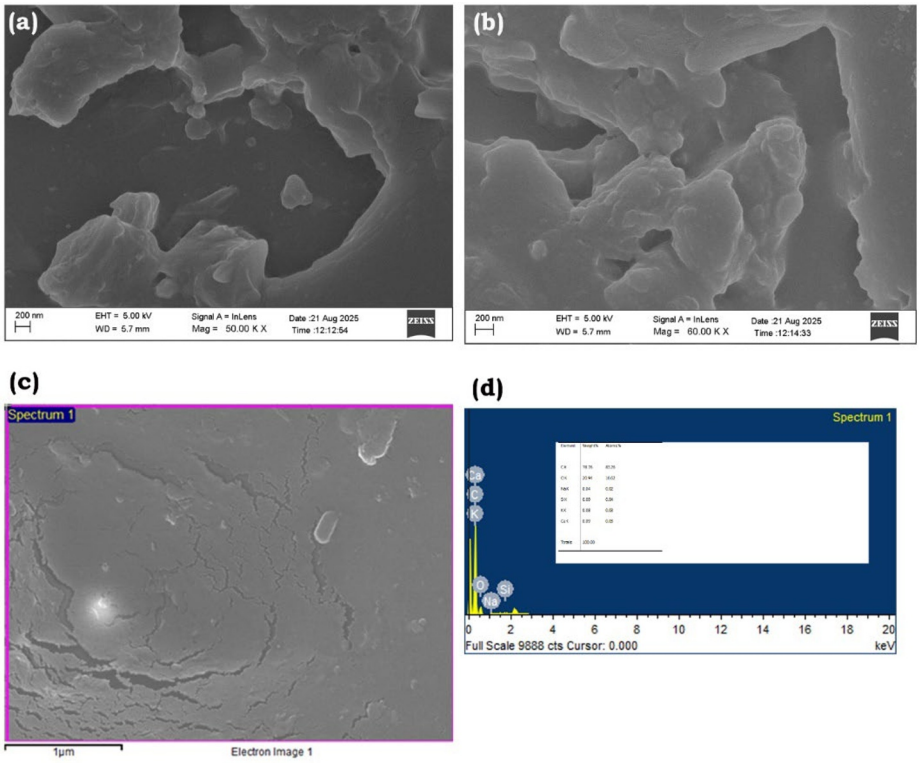

Fig. 3. (a) SEM of untreated composite showing fiber pull-out and voids, (b) SEM of alkali-treated composite showing improved adhesion, (c) High-magnification SEM of strong fiber–matrix interface, and (d) EDS spectrum confirming C, O, and minor Na after treatment.

The mechanical behavior of pure and alkali treated KJJK hybrid composite was tested among tensile strength, flexural strength, impact strength, and the elastic modulus. These findings are shown in Figures 4 (a-c) and Figure 5, which are related to various mechanical parameters. In Figure 4(a), tensile strength in untreated and treated is compared. The untreated composite had tensile strength of 92 Mpa and the alkali treated specimen had tensile strength of 128 Mpa which is significantly greater by about 39 per cent than the untreated composite. This is improved in adhesion between the different interfaces following the treatment with NaOH making it easier to transmit the stress developed by the epoxy material onto the reinforcing fibers. The alkali treatment causes an increase in roughness of the surface to form mechanical interlocking, as well as an increase in the effective surface area of stress distribution, which consequently results in tensile load-bearing capacity [13].

The flexural strength results are represented in Figure 4(b). The flexural strength of the untreated composite was 142 Mpa and 190 Mpa in the case of alkali treated composite indicating an increment of around 34. The enhancement of the flexural performance reflects the augmentation of forging shear currents caused by bending, which are normally generated at the interface between the fibers and the matrix. The increased surface activity and

wettability of the alkali-treated fibers enhance the capacity of the matrix to hold compressive and tensile stress on the opposite laminate interfaces to increase the bending resistance. The results of impact strength given in Figure 3(c) have shown that there was an incredible growth in impact strength of 18 kJ/m2 of the untreated composite to an alkali treated composite of 27 kJ/m2, a significant increase of almost half. This implies that the treated composite can take in higher energy previously fractured, since of high fiber-matrix bonding and enhanced distribution of the load across the interface. The increased energy absorption characteristic is especially valuable in applications where impact resistance is of importance like automobile interior panel application or protective casing application [14].

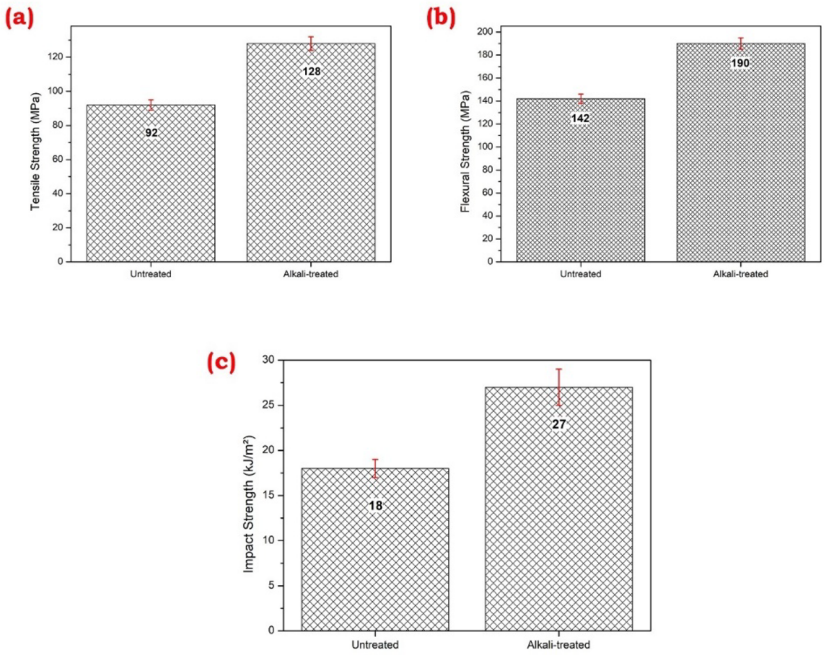

Fig. 4. (a) Tensile strength of untreated and alkali-treated composites, and (b) Flexural strength comparison, and (c) Impact strength comparison showing improved energy absorption.

Consistent mechanical tendencies are also reflected by the tensile and flexural moduli of the composites described in Figure 5. Un-treated composite had tensile modulus of 4.6 Gpa and flexural modulus of 5.4 Gpa and treated with alkali had higher tensile and flexural modulus of 5.5 Gpa and 6.3 Gpa respectively. The high stiffness observed with the alkali treatment is due to the enhancement of the efficiency of load-sharing between the fibers and the matrix, as well as elimination of microvoids. The stress transfer at the interface becomes good resulting in smaller localized deformation, which results in superior values of elastic modulus. These findings also agree with other earlier experiments, who have shown similar enhancements of 30-50 percent of mechanical properties in jute and kenaf fibers after alkali treatment. The positive changes have validated that the hybrid configuration alkali treated (KJJK) does not only improve tensile and flexural performance but also provides an acceptable stiffness to toughness balance. The mechanical test results are supported by the SEM observational results. Clearly, the untreated composites displayed fiber pull-out and cracking of the matrix, which showed weak interfacial bonding, in relation to their lower tensile and flexural strength. Meanwhile, the composites that were alkali treated showed more adhesive quality and cleaner texture of their fibers, which are associated with increased

mechanical performance of a substance. The removal of decontaminants of the surface as is done in the treatment with NaOH enables greater wetting and chemical bonding between the hydroxyl groups of the cellulose and the epoxy resin. This interfacial augmentation is directly converted to augmented mechanical conduct because the tensile, flexural, and impact strength has been extraordinarily improved. When the experimental results are combined, it can be observed that the alkali treatment has a great influence on enhancing the mechanical performances of the woven jute-kenaf epoxy composite through surface modification of the fibers and adhesive bonding of the fibers and the matrix. The KJJK hybrid structure is an effective exploitation of the jute stiffness and kenaf toughness, making the composite to have a balanced mechanical response in order to find limitless uses in lightweight structural applications. The hybridization and synergy of fiber treatment provides an effective way of coming up with sustainable composite materials with superior strength, stiffness, and impact resistance [15].

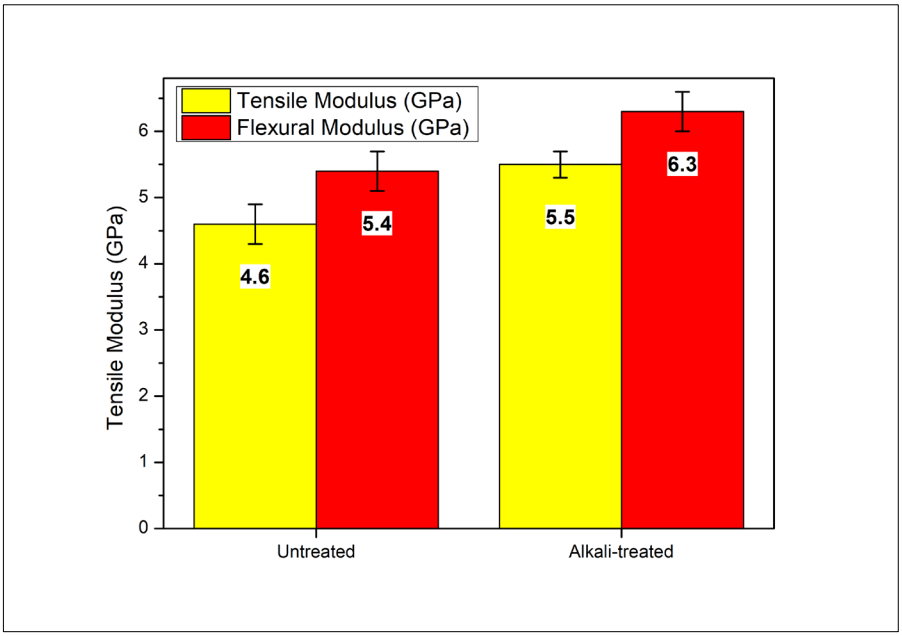

Fig. 5. Tensile and flexural modulus of untreated and alkali-treated KJJK composites showing higher stiffness after NaOH treatment.

4 Conclusions

Alkali treatment (NaOH) was also proven to combat the surface impurities like hemicellulose, lignin and waxes in woven jute and kenaf fibers to produce elevated surface roughness and enhanced wettability with the epoxy matrix. An ideal balance of rigidity and toughness was achieved in the hybrid KJJK stacking sequence (kenaf-jute-jute-kenaf) sequence that increased the overall laminate performance. SEM analysis was used to validate better fiber-matrix interfaces bonding and a reduced number of voids and fiber pull-out in the curing composites treated with fewer voids compared to the untreated ones. The samples of EDS results indicated the presence of the largest carbon and oxygen with traces of sodium, which supported the successful surface modification and low content of alkali substances. The tensile strength went up to 128 MPa (almost 39% higher) after being treated with alkali, as the tensile strength showed that stress transfer at the fiber-matrix interface was improved.

The flexural strength increased by 142 MPa to 190 Mpa (nearly 34% increase) which shows that it has better bending strength which was caused by increased adhesion between the interfaces. The strength of impact rose drastically between 18 and 27 kJ/m2 (almost by half) signifying enhanced ability to accept energy and toughness of the composite.

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