Experimental Investigation on the Flexural and Dynamic Mechanical Properties of Jute Fiber/Cork-reinforced Polyester Sandwich Composites

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The flexural and dynamic mechanical behavior were evaluated for a new jute woven fabric/cork-reinforced polyester sandwich composite. To improve the fiber/matrix adhesion, jute fibers were treated with sodium hydroxide (NaOH) and silane prior to composite preparation. The results indicated that the flexural strength and modulus of the composites increased after the alkali and alkali + silane treatments. Similarly, dynamic mechanical parameters, such as storage and loss modulus of the sandwiches, were enhanced as a result of alkali and silane treatments due to a better fiber/matrix adhesion compared with the untreated composites. It was also shown that the damping parameter decreased after the interfacial treatments, which indicated that the energy damping efficiency decreased as the interface quality was improved.

Keywords: Jute fiber; Cork; Sandwich composite; Polyester; Dynamic mechanical analysis

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INTRODUCTION

Over the last three decades, natural fibers and their composites have gained immense popularity in the business and research communities due to increasing environmental awareness and the depletion of petroleum reserves. Such interest has resulted in an intensive search for renewable and sustainable engineering materials (Wambua *et al*. 2003; Joshi *et al*. 2004; Summerscales *et al.* 2010a,b; Bordoloi *et al.* 2017a,b). Natural fibers are biodegradable, renewable, cheap, and readily available (Mohanty *et al*. 2002; Faruk *et al*. 2012). The most popular natural fibers for composite applications include jute, hemp, flax, and kenaf because of their high stiffness and strength accompanied by light weight (Bledzki and Gassan 1999; Mohanty *et al*. 2000). Jute has a special place among other bast fibers due to the fact that it is cheaper and produced in greater amounts (Pal 1984; Semsarzadeh 1985; Karmaker and Hinrichsen 1991; Karmaker and Shneider 1996). The major problems with the application of natural fibers are: a) the low compatibility between natural fibers and polymer resins, which reduce the quality of fiber/matrix interface bonding, and b) the high moisture absorption of natural fibers due to their hydrophilic structure. Fiber surface modifications, such as alkali and silane treatment, and the use of various coupling agents have proven successful at improving the fiber/matrix interfacial adhesion and thus overcoming interface-related problems (Liu and Dai 2007).

To date, various studies have been reported on the effects of interfacial treatments on the mechanical properties of the natural fiber composites. Rana *et al*. (1999) reported that the storage modulus (E') of the jute/PP composites increases as the jute fiber content is increased. They also reported an increase in E' value with the use of maleic anhydride grafted polypropylene (MAH-PP) as a coupling agent. In a similar work, it was shown that the dynamic properties of jute/PP composites improved when MAH-PP was used as a compatibilizer (Doan *et al*. 2007). Ray *et al*. (2002) reported that alkali treatment significantly improves the E' value in jute/vinylester composites compared with untreated fiber composites. Karaduman and Onal (2013) used dynamic mechanical analysis (DMA) to determine the effect of NaOH and enzymatic treatments on the dynamic mechanical properties of jute/polyester composites. They showed that E' and loss modulus (E'') both improved as a result of the treatments. It was also noted that the damping parameter (tanδ) of the composites decreased when fiber content was increased, which suggests a reduction in damping capacity (Karaduman and Onal 2013). Shanmugam and Thiruchitrambalam (2013) investigated the dynamic response of hybrid composites from untreated and alkali-treated unidirectional palm fiber and jute, in which the alkali treatment improved the E' and E'' of the composites. Jabbar *et al*. (2015) showed that enzyme, CO_2 pulsed infrared laser, and ozone treatments improved the flexural and impact properties of woven jute/epoxy composites. In addition, treated composites showed higher E', E'', and tanδ values (Jabbar *et al*. 2015). Gupta and Srivastava (2016) reported that alkali treatment significantly improves the value of E' and E'' for jute/sisal hybrid composites. Zafar *et al*. (2016) examined the effects of NaOH and NaOH + silane surface treatments on the mechanical behavior of jute/poly (lactic acid) composites. Scanning electron microscopy (SEM) showed better fiber/matrix interface bonding in the case of surface treated specimens. Composites reinforced with surface-treated jute fibers exhibited higher E' values compared with untreated fiber composites (Zafar *et al*. 2016a). Sudha and Thilagavathi (2016) reported that alkali treatment improves the mechanical properties of woven jute fabric/vinyl ester composites due to better adhesion between fibers and the resin because of the removal of lignin and hemicellulose (Sudha and Thilagavathi 2016). Lakshmanan *et al.* (2018) showed that 1% NaOH treatment of jute fibers lead to better mechanical properties of their composites.

Cork is a cellulose-based material that is obtained from the bark of cork oak. It is mainly used for bottle stoppers and insulation materials, floor and wall coverings, gasket sealers, joint fillers, *etc*. Cork has many desirable properties for an industrial material since as it is lightweight, flexible, fire resistant, and impermeable. It also has outstanding sound and thermal insulation properties as well as good vibration damping, but cork plates have a low out-of-plane mechanical strength and are fragile, especially under bending loads (Gibson *et al*. 1981).

In this study, sandwich composites were fabricated using cork plates as the core material and jute woven fabric-reinforced polyester composite plates as facing materials. The main aim is to support the fragile cork material from two sides to improve its mechanical properties and develop lightweight and strong biocomposites. The effect of alkali and silane treatments on the flexural and dynamic mechanical properties of the resulting jute/cork-based composites are investigated for their potential usage in automotive and housing applications.

EXPERIMENTAL

Materials

Plain woven jute fabrics with an areal density of 300 g/m^2 (Cuvsan Ltd., Gaziantep, Turkey) were used for the fabrication of jute/polyester composite facings. The warp and weft densities of the woven fabric were 5 yarns/cm and the yarn count was 300 tex for both yarns. Unsaturated polyester resin (Polipol-3401; Poliya Inc., Istanbul, Turkey) was used as matrix material together with 2 wt% methyl ethyl ketone-peroxide (MEK-P) as initiator and 0.2 wt% cobalt as catalyzer. Cork plates with a thickness of 5 mm (Duplas Inc., Istanbul, Turkey) were used as the core material.

Chemical treatments

Jute fabrics were treated with NaOH to improve the fiber/matrix interface adhesion. The NaOH concentration and other treatment parameters were chosen based on the authors' previous studies (Karaduman *et al.* 2013; Karaduman and Onal 2013). The jute fabrics were treated with 10 wt% NaOH solution at a temperature of 25 °C for 30 min. After the treatment, the fabrics were neutralized using 2 wt% acetic acid solution to obtain a neutral pH value. Then, the fabrics were rinsed and oven-dried at 70 °C for 8 h before being used as reinforcement.

After the alkali treatment, the jute fabrics were treated with silane using the procedure described by Zafar *et al.* (2016b). Jute fabrics were treated with 5 wt% γ-aminopropyltriethoxy silane (APS) dissolved in a water–ethanol (40:60, w/w) mixture. Acetic acid was added to the solution to maintain a pH of 4. Then, the jute fabrics were immersed in the solution for 3 h. Fabrics were then rinsed and oven-dried at 70 °C for 8 h.

Composite preparation

After the surface treatments, two layers of jute woven fabrics were placed on each face of the 5 mm-thick cork material, and the polyester resin mixture was applied with the aid of a roller. The amount of polyester resin mixture applied to the fabrics was kept constant for each sample to obtain the same percentage of the ingredients. After the materials were satisfactorily wetted by the resin, they were placed into a compression molding machine and consolidated in a one-shot process under a pressure of 5 MPa at room temperature for 1 h. After consolidation, the samples were cured for 12 h at room temperature. Cork, jute, and polyester weight ratios were obtained by weighing each component prior to composite production and dividing by the final weight of the sandwich sample. In the final sandwich materials, cork, jute, and polyester weight ratios were 15%, 18%, and 67%, respectively. The final thickness of the sandwiches was 8.6 mm (cork thickness: 5 mm; each facing thickness: 1.8 mm).

Methods

Composite density measurements

Density measurements of the produced sandwich composite samples were performed according to ASTM D792 (2000) using a Precisa XB 220A density measurement balance (Precisa Gravimetrics AG, Dietikon, Switzerland).

Three-point flexural test

Three point flexural tests of the sandwich composites were conducted according to ASTM C393 (2000) in a 5 kN-capacity Shimadzu AG-XD (JP) testing machine (Shimadzu Co., Kyoto, Japan) with a crosshead speed of 2.8 mm/min. The span length was 75 mm. Three rectangular plate specimens with dimensions of 125 mm × 20 mm × 8.6 mm were tested for each sample group and the average values were reported along with standard deviations. The core shear strength (τ) and facing flexural strength (σ) were calculated according to Eq. 1 and Eq. 2, respectively,

$$\tau(MPa) = \frac{P}{(d + c)b} \tag{1}$$

$$\sigma(MPa) = \frac{PL}{2t(d + c)b} \tag{2}$$

where *P* is the maximum load (N), *d* is the sandwich thickness (mm), *c* is the core thickness (mm), *b* is the sandwich width (mm), *L* is the span length (mm), and *t* is the facing thickness (mm).

Dynamic mechanical analysis

The DMA was conducted using a Perkin-Elmer DMA 8000 device (PerkinElmer Inc., Waltham, MA, USA) in accordance with the ASTM D4065 (2012) standard. Single-cantilever bending mode was chosen for the analysis. Rectangular specimens with dimensions 30 mm × 12 mm × 8.6 mm were used for the tests. All of the tests were conducted in a temperature range of 20 °C to 200 °C with a heating rate of 2 °C/min, under nitrogen flow and the oscillation frequency was 1 Hz. Three specimens were tested for each sample type and the average values were reported together with standard deviations.

Scanning electron microscopy

SEM analysis was performed using a Leo 440 scanning electron microscope (Oxford Instruments PLC, Oxfordshire, UK) to determine the quality of adhesive contact at the fiber/matrix interface and the effect of interfacial treatments. The samples were sputtered with gold-palladium (AEM Ltd., Hunan, China) before the analysis. An accelerating voltage of 10 kV was used for the analysis.

RESULTS AND DISCUSSION

The measured density values of the prepared sandwich composite samples were in the range of 0.65 to 0.67 g/cm^3 with an average of approximately 0.66 g/cm^3. The density differences of the samples were thus insignificant. Table 1 lists the results of the three-point flexural tests of the composite samples. The results are depicted in Fig. 1. It can be clearly seen that the flexural strength and modulus of the alkali treated and alkali + silane-treated composite samples considerably increased when compared to the untreated samples. Chemical treatments also reduced the maximum deflection values of the composites. These results were attributed to the positive effect of surface treatments on the fiber/matrix interface of the composites.

Table 1. Flexural Properties of Sandwich Composites

Sample	Flexural Strength (MPa)	Flexural Modulus (MPa)	Max. Deflection (mm)	Core Shear Strength (MPa)	Facing Flexural Strength (MPa)
Untreated	26.59 (0.3903)	1501.78 (36.11)	8.06 (0.3135)	1.33 (0.0099)	27.78 (0.4125)
NaOH	28.86 (0.4122)	1703.92 (45.51)	5.50 (0.2884)	1.39 (0.0112)	29.07 (0.4241)
NaOH + silane	31.25 (0.4226)	1801.63 (40.15)	4.53 (0.2751)	1.41 (0.0117)	31.54 (0.4255)

*Standard deviations are given in parentheses.

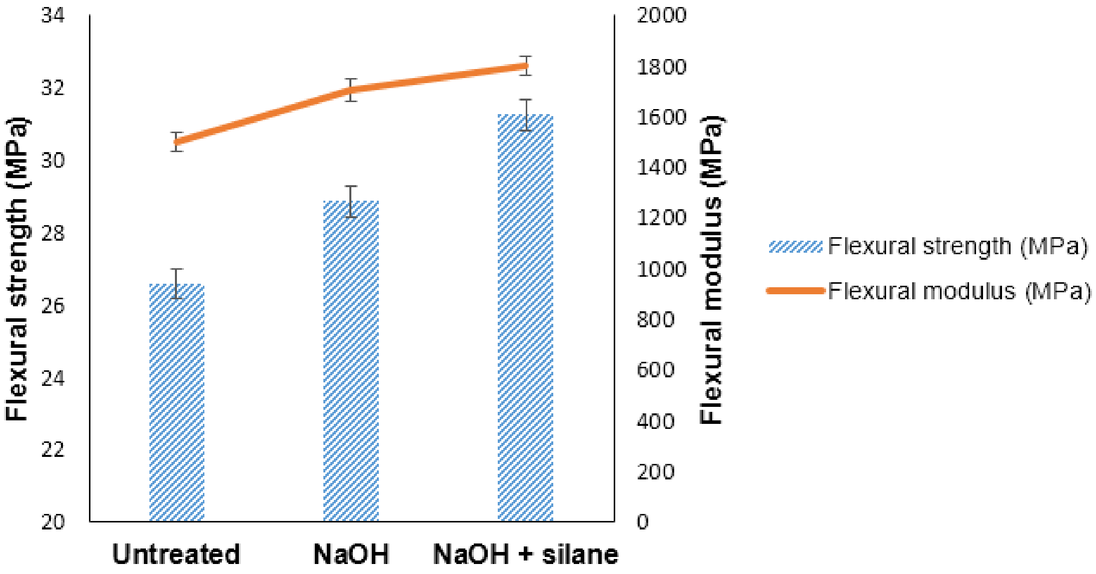


Fig. 1. Flexural test results of jute/cork sandwich composites

Force-deflection graphs for different sandwich composite samples (Fig. 2) also suggest that the composites subjected to alkali and alkali + silane pretreatments showed more brittle behavior when compared to untreated samples. This is also an indication of improved fiber/matrix bonding after these treatments.

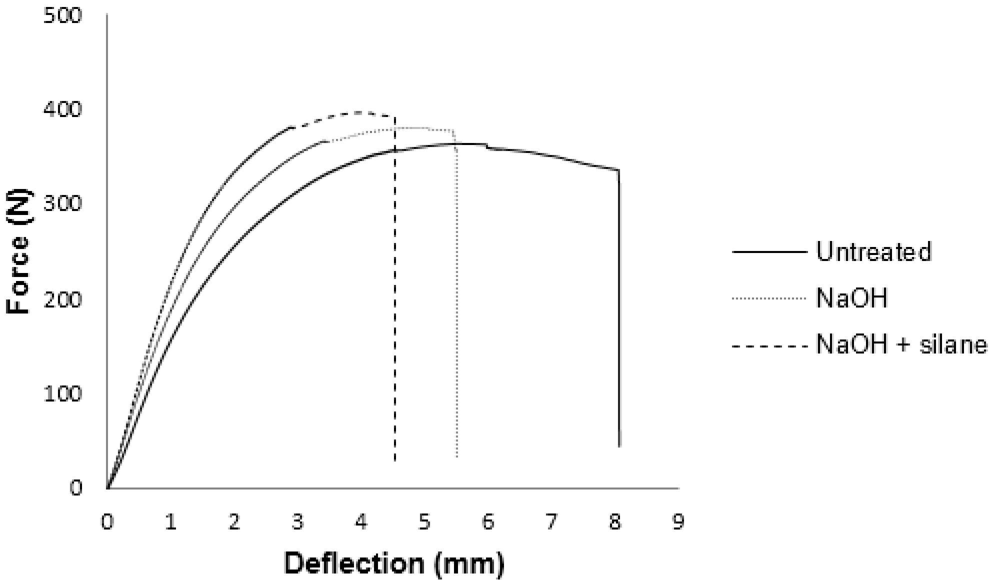


Fig. 2. Force-deflection graphs for different sandwich composite samples

Figure 3 shows the crack surfaces of the composite samples after the flexural tests. In general, untreated, NaOH-treated, and NaOH + silane-treated samples showed similar failure patterns. The failure took place on the tension (bottom) side of the samples as a combination of fiber breakage and brittle matrix cracking. There was no sign of delamination between the core and facing materials, which indicates a good bonding.

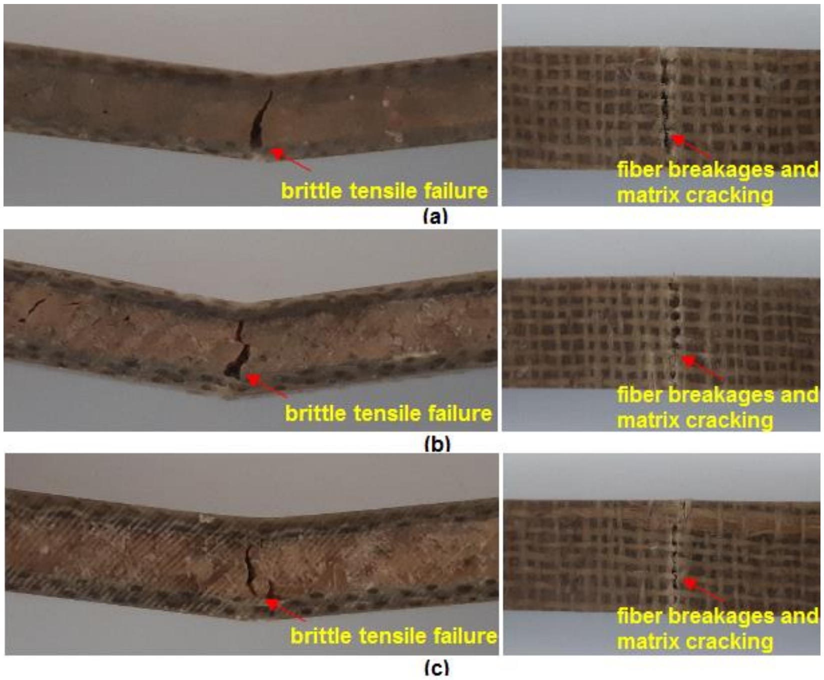


Fig. 3. Crack surfaces of the samples after the flexural tests (magnification: 3x). (a) untreated (b) NaOH-treated (c) NaOH + silane-treated

Table 2 and Fig. 4 present the results of the DMA of the composite samples. In general, the surface treatments resulted in a noticeable increase in the storage modulus (*E'*) and loss modulus (*E''*), whereas the damping parameter (tan*δ*) values of the composites decreased after the treatments. It was also noted that the α-transition temperature shifted to higher temperature regions after the alkali and alkali + silane treatments. In the composite structure, jute fibers, and cork provided strength and stiffness to the polymer matrix and noticeably restricted the mobility of the polymer matrix at low and elevated temperatures. The fiber/matrix interface especially limited the movement of the main chain of the polymer macromolecule as well as the side groups due to fiber/matrix bonding at the interface. Both the alkali and alkali + silane treatments improved the interface and in turn increased the *E'*, and *E''* values of the composites. The decrease in tan*δ* was attributed to the increased energy absorption capability of untreated fiber composites due to the friction losses in the process of fiber sliding in the matrix. The shift in α-transition temperature (T_α) to higher temperature regions after the chemical treatments can also be attributed to the increased restricting effect and increased thermal stability after the surface treatments (Gill *et al.* 1984).

Table 2. Dynamic Mechanical Properties of Sandwich Composites

Sample	Max *E'* (MPa)	Max *E''* (MPa)	Max *tanδ*	T_α (max *E''*) (ºC)
Untreated	618.32 (25.65)	80.68 (5.512)	0.3304 (0.0059)	54.9 (1.343)
NaOH	1166.39 (28.65)	130.46 (6.251)	0.2974 (0.0061)	66.1 (1.451)
NaOH + silane	1387.13 (29.62)	148.06 (7.652)	0.2665 (0.0062)	67.5 (1.556)

E': storage modulus; *E''*: loss modulus; *tanδ*: damping parameter; and T_α: α-transition temperature. Standard deviations are given in parenthesis.

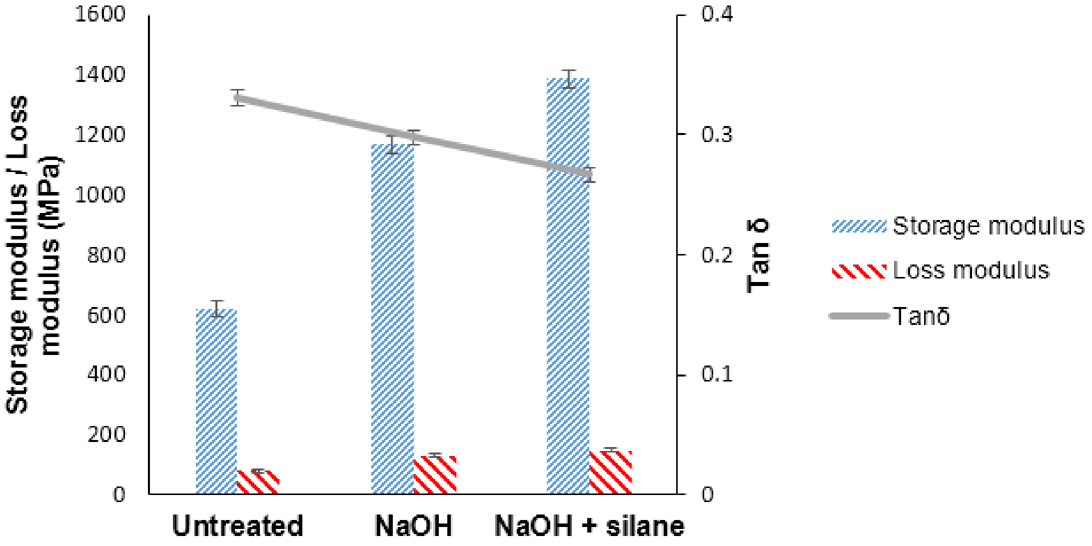


Fig. 4. Dynamic mechanical parameters of jute/cork sandwich composites

Figure 5 shows the *E'* values of the untreated, alkali-treated, and alkali + silane-treated jute/cork sandwich composites as a function of temperature. The *E'* values of the composites were at their maximum value at room temperature and gradually decreased as the temperature increased during the dynamic loading due to the degradation of fibers and polymer matrix at higher temperatures. Jute fibers and cork, as well as the fiber/matrix interface, provided a certain stability to the polymer matrix and delayed the failure of the material under load.

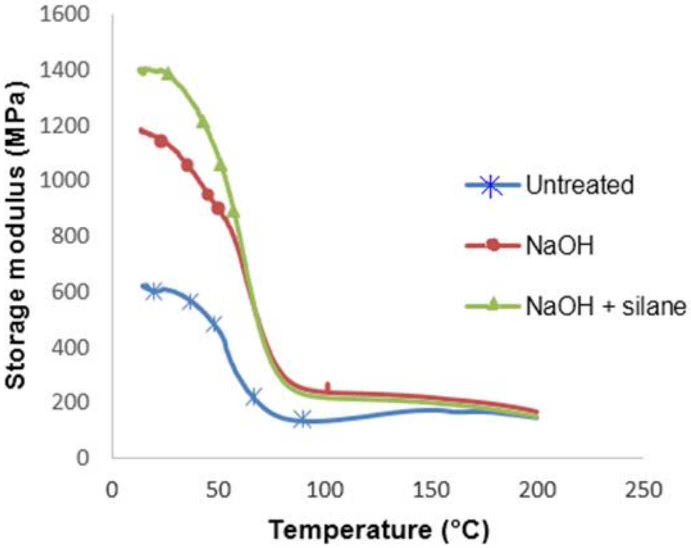


Fig. 5. Storage modulus (E') *versus* temperature graphs of different jute/cork sandwich composites

The fiber/matrix interface also played an important role in transferring the load from weak matrix material to stronger jute fibers. Therefore, a better fiber/matrix adhesion promoted better load distribution capability of the composite and enhanced the mechanical properties. Figure 5 shows that the untreated jute fiber composites had the lowest *E’* when compared to alkali- and alkali + silane-treated composite samples. Alkali-treated samples showed higher *E’* compared with the untreated samples. This was attributed to the fact that alkali treatment improves the fiber/matrix adhesion by removing polymers with a low degree of polymerization such hemicellulose, pectin, and some components of lignin, as well as other impurities from the fiber surface and creates a rougher surface morphology (Karaduman and Onal 2013). This promoted the mechanical locking between the matrix and fibers and enhanced the adhesion quality at the interface and improved the load distribution capability of the material. A further improvement in *E’* was recorded after the silane treatment, which indicated that the silane treatment further improved the fiber/matrix adhesion at the interface.

Figure 6 shows the *E''* values of the untreated, alkali-treated, and alkali + silane-treated jute/cork composites as a function of temperature. It was shown that the *E''* values of the composites gradually increased as the temperature increased, reaching a maximum value in the α-transition region (54.9 ºC to 65.3 ºC) and then decreased with further increment in temperature. An increase in *E''* with temperature was indicative of an increased resin mobility at higher temperatures, which resulted in enhanced energy absorption capability and toughness of the material. The temperature corresponding to the maximum *E''* is considered as the α-transition temperature (T_α) of the material (Akay 1993; Rana *et al*. 1999). The T_α values of the composite samples were recorded in the range of 54.9 ºC to 65.3 ºC. Similar to the storage modulus, the loss modulus values of the composites increased with surface treatments. The alkali + silane treatment resulted in the highest loss modulus, followed by the alkali treatment, and the lowest value was obtained with the untreated samples.

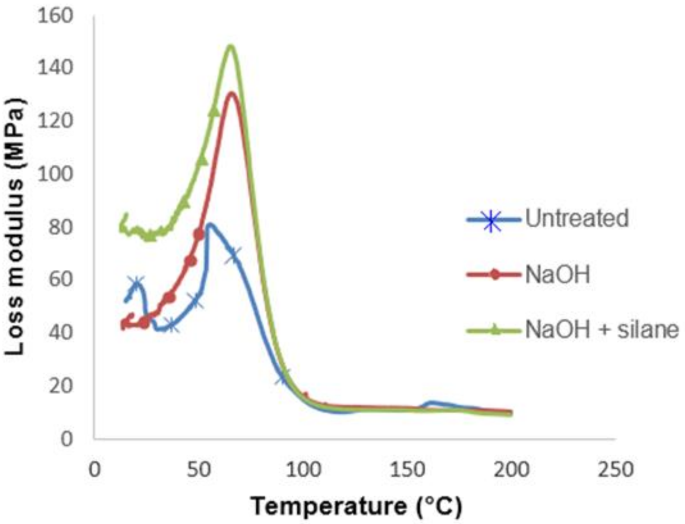


Fig. 6. Loss modulus (*E''*) *versus* temperature graphs of different jute/cork sandwich composites

The damping parameter (tan *δ*) shows the energy dissipation capability of a material. Figure 7 shows the tan *δ* of jute/cork sandwich composites as a function of temperature. It was shown that tan *δ* increased with increased temperature due to the increased mobility of polymer molecules at elevated temperatures. Further increase in temperature led to decrease in tan *δ* values due to the degradation of jute fibers and the polymer. It was also shown that the interfacial modifications resulted in lower tan *δ* values, which indicated lower energy damping capability. This result was attributed to the fact that composites with poor fiber-matrix adhesion generally show higher energy damping because the fibers in the matrix have a certain degree of mobility in the matrix phase due to the low adhesion. In such composites, poor fiber-matrix adhesion permits fibers to slide in the matrix under load and the fiber-matrix friction caused by this sliding absorbs a great deal of energy, which results in a higher tanδ. Similar results were reported in previous literature where tan δ decreased as a result of improved fiber-matrix adhesion (Chua 1987; Correa *et al*. 2007).

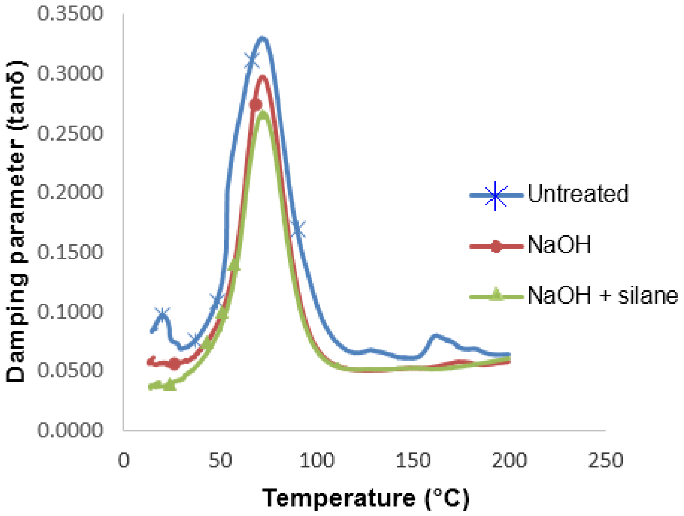
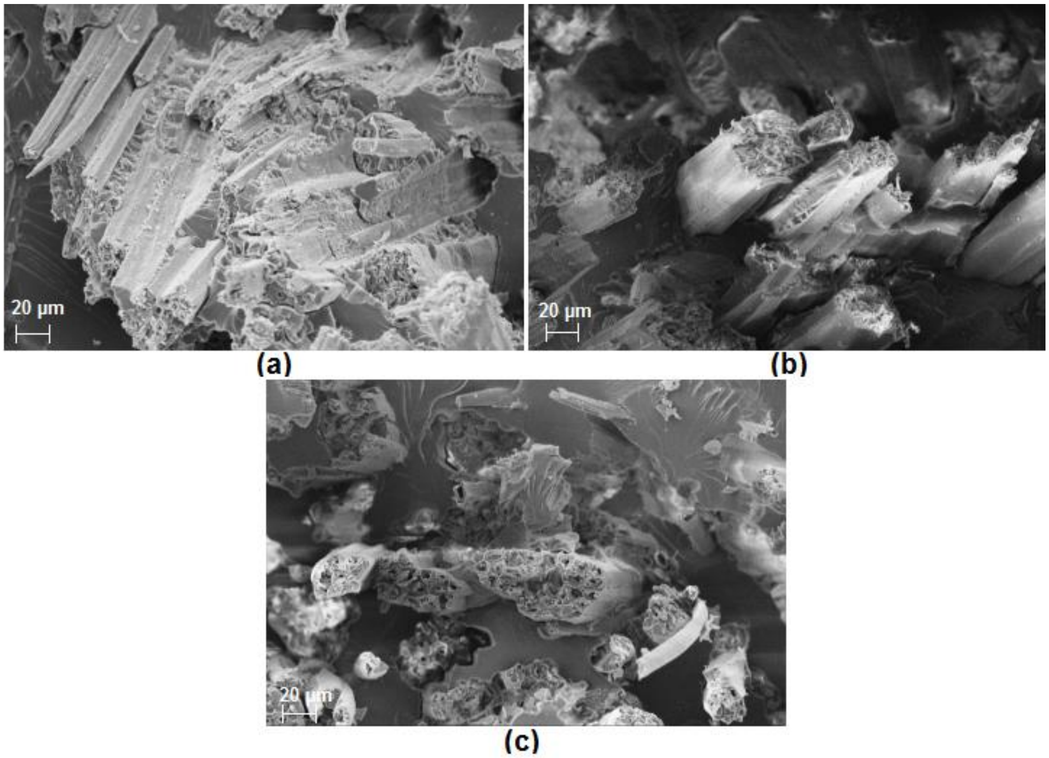


Fig. 7. Damping parameter (tanδ) *versus* temperature graphs of different jute/cork sandwich composites



(a) (b) (c)

Fig. 8. SEM micrographs of (a) untreated, (b) alkali-treated, and (c) alkali + silane-treated composite samples at a magnification of 1000×

The SEM micrographs of the fiber/matrix interfaces of the untreated, alkali-treated, and alkali + silane-treated composite samples are shown in Fig. 8. It can be clearly seen that the untreated fiber/matrix composites had a poor matrix/adhesion, as inferred from the long fiber pull-outs and insufficient fiber surface covering. The voids that were remains of fiber pull-outs were clearly visible as well as long protruding fibers. This fiber pull-out mechanism was responsible for the higher damping capability of the untreated samples. In contrast, alkali-treated and alkali + silane-treated composites showed shorter fiber protruding and less fiber pull-out, which indicated a stiffer and more brittle behavior that explained the higher storage modulus values of these composites. It was also observed that fibers had been sufficiently wetted by the matrix polymer in the case of the treated samples and fibers were completely covered by polyester resin.

CONCLUSIONS

1. The alkali and alkali + silane treatments improved the flexural properties of the composites, such as the flexural strength and modulus, which was attributed to the increased fiber/surface adhesion after these treatments.
2. The storage and loss moduli of the jute woven fabric/cork-reinforced composite samples increased with the alkali and alkali + silane treatments, which again was attributed to improved fiber/matrix adhesion after these treatments. The surface-treated samples showed more brittle behavior compared to the untreated samples.
3. The untreated samples showed higher damping parameter values, suggesting better energy damping capability. This was attributed to the poor fiber/matrix adhesion in these samples and increased friction between the fibers and matrix under dynamic loading, which acts as an energy absorbing mechanism.
4. The SEM images clearly showed that fiber surface pretreatments improved the fiber/matrix adhesion and led to a more brittle failure behavior. The untreated samples exhibited poor fiber/matrix interface and a ductile failure behavior characterized by long fiber fringes and pull-outs, explaining their high energy damping character.

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