



Dynamic Mechanical Properties of Kenaf, Thespesia Lampas and Okra Fiber Polyester Composites

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Abstract. Dynamic Mechanical Analysis is carried out on treated and untreated kenaf, thesipesia lampas and okra fiber polyester composites. Results indicate that composites with fibers treated by sodium hydroxide have higher storage modulus, decreased loss modulus and damping factor. Best properties are noted for treated okra fiber composite with highest storage modulus and glass transition temperature and lowest damping factor. The Scanning Electron Microscopy results indicate a superior interfacial bonding for treated kenaf fiber composite. Improved interfacial bonding upon treatment causing hindrance to the mobility of molecular chains can be seen clearly in the micrographs. The effect of chemical modification of the fibers in enhancing the dynamic mechanical properties is well demonstrated for kenaf, thesipesia lampas and okra fiber composites in this paper.

Keywords: Chemical treatment · Dynamic mechanical analysis · Storage modulus · Loss modulus · Damping ratio · Glass transition temperature

1 Introduction

Dynamic Mechanical Analysis (DMA) is a sensitive technique that characterizes the mechanical response of materials by monitoring the property changes with respect to temperature, stress and frequency of loading. In this technique, a small sinusoidal time-varying oscillating force is applied on the specimen. This technique separates the dynamic response of the materials into two distinct parts: an elastic part and the viscous or damping component. The elastic process describes the ability of the material to store energy applied to it, known as the storage modulus (E'). The viscous component describes the material tendency to dissipate energy applied to it, usually termed as the loss modulus (E''). The polymer composites being viscoelastic material, they exhibit a combination of both elastic and viscous behavior. Damping is determined by the ratio

between loss modulus and the storage modulus and is termed as $\tan \delta$. It is also known as the mechanical loss factor or the dynamic loss factor. This ratio depends on the degree of fiber and matrix adhesion. A weak fiber-matrix bonding will reflect in higher values of $\tan \delta$.

Ku and Wang [1] have noted that bleaching of hemp fibers has exhibited good results of enhancing interfacial bonding. Treatments like mercerization, permanganate, benzylation, poly methyl methacrylate and admicellar polymerization on sisal fibers have improved the interfacial bonding and enhanced the tensile, flexural and impact strengths, dynamic mechanical behaviour, electrostatic charge, thermal stability, dielectric constant and ac conductivity properties of the sisal fiber polyester composites [2–5]. Storage modulus, loss modulus and damping factor have increased due to treatment and decreased with increase in fiber content. Akil et al. [6] have studied the dynamic mechanical properties of pultruded kenaf fiber composites under varying frequencies of oscillating load over a range of temperatures, Aziz and Ansell [7] have studied alkalized kenaf–polyester and hemp–polyester composites. Thermo gravimetric analysis (TGA), differential scanning calorimetry (DSC), and DMA have produced superior properties for composites with treated fibers. Mysamy and Rajendran [8] have studied the dynamic and thermo mechanical behavior of agave americana fiber composites. Ornaghi Jr. et al. [9, 10] have studied the dynamic mechanical properties of glass/sisal fiber hybrid composites and found that adhesion has improved with glass fiber content. The glass transition temperature has increased with increase in frequency. Duc et al. [11] have studied the mechanical and damping properties of unidirectional, cross ply and 2×2 twill configurations with 40% flax fibers and 60% epoxy resin. The quality of impregnation, angle of twist of fibers, have improved matrix adhesion and enhanced the damping and stiffness properties of the composites significantly. Costa et al. [12] have presented a review on the recent publications on Dynamic Mechanical and Thermal Analysis (DMTA). Sezgin et al. [13] have studied the jute carbon polyester composites for different stacking sequence and found that for exterior carbon there is an increase in storage and loss modulus without significant effect on T_g . Monteiro et al. [14] have studied the viscoelastic and glass transition temperature behavior of fique natural fiber fabric composites that can replace Kevlar fiber composites for armor applications and noted that fiber content has increased these properties. Surya Nagendra et al. [15] have done DMA on glass/epoxy mixed with nano particles of banana fiber. 8% of banana particles by weight of resin have improved the properties by causing hindrance to mobility of molecular chains. Reduction in hysteresis and improvement in elastic properties is noted. Palanivel et al. [16] have studied treated and untreated hemp/epoxy composites filled with cellulose. Increase in cellulose filler loading and treatment by NaOH and Benzoylation have improved E' and decreased E'' and $\tan \delta$. Rashid et al. [17] have conducted DMA of phenolic sugar palm fiber powder composites. Increase in fiber content has improved the properties. NaOH treated fiber gave better results than by sea water treated fiber. A smooth semi-circular shaped Cole-Cole curves presented indicate a homogeneous mixture of fiber powder and matrix. Saba et al. [18] have presented a review of DMA on natural fiber and hybrid composites with thermoplastic, thermoset and biopolymer resins and also hybrid nano composites. They have presented clearly that E' , E'' , $\tan \delta$ and T_g are influenced by the fiber content and treatment causing decreased mobility of molecular chains. They

have also projected that DMA plays an important role on the application of composite materials used in aircraft and automobile components. Pothan et al. [19] have done DMA on banana polyester composites and stated that increased volume fraction of fibers causes more restraint at interfaces thus resulting in increased E' , decreased E'' and $\tan \delta$ and increase in T_g . Romanzini et al. [20] studied the properties of hybrid ramie/glass polyester composites by DMA for different ratios of ramie/glass fiber content. Higher activation energy was observed at 75% of glass fiber content. Jacob et al. [21] have reported the results of sisal/oil palm hybrid fiber natural rubber composites. The storage modulus has increased with fiber volume fraction and also up treatment. Abdullaha and Jamaludin [22] have studied the effect of aging of fibers on the properties of Arenga Pinnata fiber/epoxy composites by DMA. NaOH treated-accelerated aging process is adopted. The aged composites are reported to have better properties. Chaudhary et al. [23] have studied tribological properties and also by DMA for jute/hemp, hemp/flax and jute/hemp/flax combinations. Selvakumar et al. [24] have reported that higher mechanical and dynamic mechanical properties are noted for increasing human hair content in jute/human hair epoxy composites. Nimanpure et al. [25] have reported the dielectric properties and results of DMA on treated and untreated short fiber sisal/epoxy composites. 5% NaOH treated for 72 h have indicated higher values of E' for 35 g fiber with 65 g of resin with a lower value of E'' and $\tan \delta$. T_g also has increased for this fiber content.

This paper highlights the effect of NaOH treatment on the results of DMA for kenaf, thespesia lampas and okra treated and untreated fiber/polyester composites. These three plants belong to Malvaceae family. Similar cellulose fibers by other names like Nacha are available in Ethiopia. Fibers in the present work are available in India and other parts of Africa. The storage modulus, E' , loss modulus, E'' and dynamic loss factor, $\tan \delta$ and glass transition temperature, T_g are studied. The SEM also done to visualize the effect of treatment.

2 Methodology

2.1 Materials and Preparation of Composites

The fibers of kenaf, thespesia and okra are extracted from the stems of the plants and subjected to water retting process for decomposition of the green substance. They are washed in running water and then dried in sun. These fibers are treated in 2% NaOH solution for 24 h at room temperature, thoroughly washed in distilled water and then dried. The treated and untreated fibers of kenaf, Thespesia lampas and Okra are shown in Fig. 1.

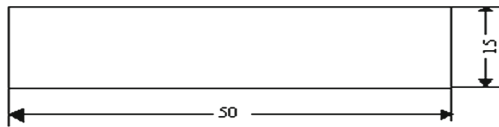
Unidirectional mats are prepared on an indigenous weaving set up and hand molded at room temperature. General purpose polyester resin, 2% catalyst, methyl ethyl ketone peroxide (MEKP) and 2% accelerator, cobalt naphthenate were used for the matrix. A fiber-matrix ratio of 1:6 by weight ratio has been arrived to give proper wetting of fibers and eliminating the entrapped air from the mix of resin-fiber and upper and lower glass plates. The two thick glass plates are coated with wax and poly vinyl alcohol (PVA) as releasing agents and a weight of 500 N is kept for 24 h.



Fig. 1. Fibers used for making laminates

2.2 Dynamical Mechanical Analysis

The specimens for DMA are prepared as per ASTM D 4065. The dimensional details of each type of specimen are presented in Fig. 2.



(Dimensions are in mm)

Fig. 2. DMA test specimen.

The specimens of kenaf fiber composite-untreated fiber (kfc-ut), thesipesia lampas fiber composite (tfc-ut) and okra fiber composite (ofc-ut) are shown in Fig. 3. The Pyris Diamond DMA equipment by Perkin-Elmer Instruments is used for the testing. The specimen is placed in its holder and subjected to a small, time-varying sinusoidally oscillating force. The phase shift between the applied stress and the corresponding strain is measured.

2.3 Differential Scanning Calorimetry

Composites exhibit changes in material properties such as volume, enthalpy, heat capacity, thermal expansion and tensile modulus as the material is heated through glass transition temperature, T_g and it goes from glass to rubbery state. DSC is performed with the help of Mettler using Star SW 8.1 analyzer to measure T_g . The temperature is programmed in the range of 25°–300 °C with a heating rate of 10 °C/min in nitrogen atmosphere with a flow rate of 30 ml/min.

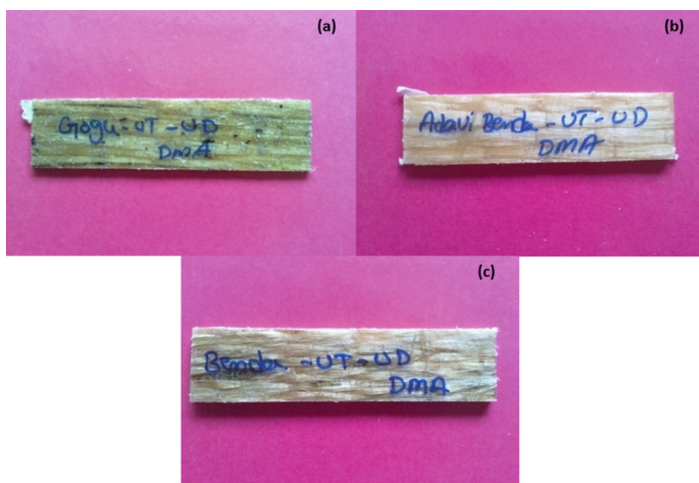


Fig. 3. Specimens for DMA(a) kfc (b) tfc (c) ofc.

2.4 Scanning Electron Microscopy

The morphology of the fractured surfaces of the composites is studied by Scanning Electron Microscopy (SEM) using EVOMA15 Smart SEM. SEM reveals the quality of fiber-matrix bond bonding that mainly affects the properties of the composite. A small electron beam spot (usually circa $1\ \mu\text{m}$) scans the fractured surfaces repeatedly. The importance of SEM is that it produces images that are similar to those of a large scale piece and even the irregularities on the surface of the material can be observed.

Before performing SEM, the surfaces of the fractured specimens are coated with platinum. They are placed on the stub and then inserted into the scanning barrel. The atmosphere in the scanning barrel is vacuumed to prevent interference effects on then scanned picture due to presence of air. Magnification, focus, contrast and brightness of the pictures are adjusted to produce the best micrographs.

3 Results and Discussion

3.1 Dynamic Mechanical Properties

The storage modulus is useful in assessing the molecular basis of the mechanical properties of the material. This property is very sensitive to the structural changes occurring at the fiber matrix interface due to variations in the degree of bonding. In order to analyze the DMA data, the storage modulus, loss modulus and $\tan \delta$ are plotted against the temperature. Figure 4(a) shows the storage modulus of the untreated kfc, tfc and ofc. At room temperature, the initial storage modulus of kfc-ut, ofc-ut and tfc-ut are 7.85 GPa, 6.4 GPa and 6.3 GPa respectively. Nevertheless, it is important to emphasize the composite behavior at high temperatures. However, the storage modulus E' gradually decreases with increase in temperature as the matrix softens and the drop is maximum in the temperature range between $50\ ^\circ\text{C}$ – $120\ ^\circ\text{C}$. At, the values of storage modulus are

equal and are again matching from onwards for the three composites. The tfc (ut) has higher storage modulus between 50 °C–100 °C. The tfc fibers are basically stiffer than kenaf and okra fiber and could have resulted in the variation of the storage modulus. Onset of drop of modulus corresponds to the molecular mobility of the matrix.

The variation of storage modulus at different temperatures for treated kfc, tfc and ofc are presented in Fig. 4(b). At room temperature, the initial storage modulus of treated kfc, ofc and tfc are 8 GPa, 8.28 GPa and 4.9 GPa. The storage modulus and the thermal transition temperatures of the composites have shifted to higher values when the treated fibers are used. NaOH treatment has resulted in increase of storage modulus of the ofc-t and kfc-t by 31.63% and 2% compared to the ofc-ut and kfc-ut composites. It suggests that the surface treatment of fibers has increased the interfacial adhesion. In case of tfc, the treatment has resulted in 21.9% decrease of storage modulus. The loss modulus, E'' of the untreated kfc, tfc and ofc has reached a maximum value as the storage modulus, E' has decreased which can be seen in Fig. 4(c) and (d). This behavior is produced by the free movement of the polymeric chains at higher temperatures. The initial values of E'' for the composites studied are listed in Table 1. The change in slope of the loss modulus, E'' spectrum represents β -transition and it is clearly visible in loss modulus curves of the treated and untreated composites.

Unlike the curves shown in Fig. 4(e) for the kfc-ut, tfc-ut and ofc-ut, the loss modulus curves for the kfc-t, tfc-t and ofc-t shown in Fig. 4(f) are very much dispersed. It can be noted that the surface treatment of the fibers has increased the $\tan \delta$ value for kfc-t from 0.22 at 76.2 °C to 0.24 at 81.6 °C, for tfc-t from 0.22 at 95.6 °C to 0.25 at 84.5 °C. But the $\tan \delta$ has reduced for ofc-t from 0.21 at 81.7 °C to 0.19 at 104.5 °C upon surface treatment.

Increase in the value of T_g of kfc-t and ofc-t is taken as a measure of the increased interfacial interaction upon treatment. The presence of more fiber content in the composite reduces the magnitude of the peak value of $\tan \delta$. Decrease in $\tan \delta$ for untreated tfc is probably due to the participation of more number of fibers in bending. The increase in width at the peak of the $\tan \delta$ curve for the treated ofc is due to molecular relaxations that took place due to rise of temperature of the composite during loading process. The molecular motions generally contribute to the damping of the material apart from those of the constituents. Increase in peak height in kfc is due to the decrease in stress transfer from fiber to matrix because of fiber agglomeration and increase in fiber to fiber contact. Slight reduction in $\tan \delta$ for the treated ofc composites compared to that of the untreated ofc. This indicates the restricted mobility of the molecules due to the stronger interfacial addition between fiber and the resin. Further, decrease in $\tan \delta$ indicates an improvement of the fatigue property. The shift of the $\tan \delta$ curves towards right into the higher temperature range is an indication of the presence of certain process that has restricted the mobility of the chains in the crystalline phase so that more energy is required for the transition. Therefore, it seems that natural fibers restricted the matrix polymer chains and increased the transition temperature.

Low values of $\tan \delta$ associated with glass transition temperature of ofc-t reflects improved load bearing properties of the system. Reduction in $\tan \delta$ represents an increase of storage modulus compared to loss modulus measured at the same temperature. An

inherently high elasticity of the cellulose fibers might have contributed for the higher value of storage modulus and decrease in $\tan \delta$.

Cellulose content of Kenaf: 55%; Thespesia lampas: 60.6%; okra: 60–70% and tensile strength of Kenaf: 504.8 MPa; Thespesia lampas: 573 MPa; okra; 234–380 MPa; Young’s modulus of Kenaf: 51.8 GPa; Thespesia lampas: 61.2 GPa; okra-5–13 GPa; It shows that okra fiber is a low strength fiber with high cellulose content among the three. High cellulose content has made it superior with high storage modulus compared to other two types of fibers.

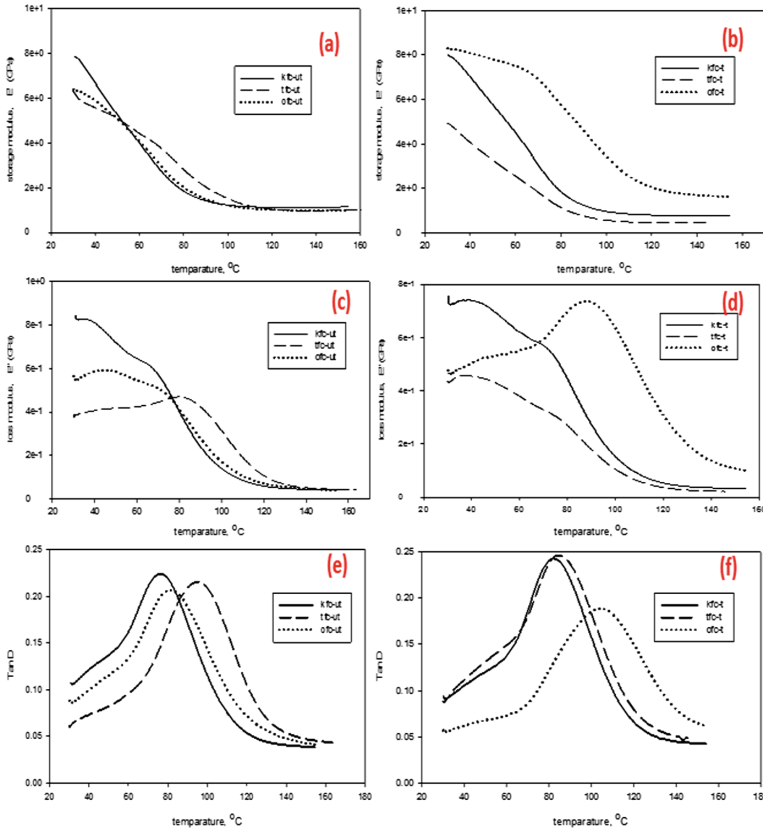


Fig. 4. (a) and (b). Storage modulus of untreated & treated fiber composites; (c) and (d): Loss modulus of untreated & treated fiber composites; (e) and (f): Dynamic Loss factor of untreated and treated fiber composites.

The storage modulus, loss modulus and the damping factor and the temperature at which test is conducted are given in Table 1. Generally, for hard plastics, the damping factor $\tan \delta$ equal to 0.01–0.1 is considered as low damping, 0.1–1 as medium and > 1 as high damping.

Table 1. Dynamic mechanical analysis data

Composite	Temp °C	E' GPa	E'' GPa	Tan δ
kfc-ut	30.83	7.85	0.84	0.107
kfc-t	30.25	8.01	0.76	0.094
tfc-ut	30.09	7.31	0.39	0.061
tfc-t	30.06	4.93	0.44	0.088
ofc-ut	30.06	7.40	0.57	0.088
ofc-t	30.13	8.29	0.48	0.057

Table 2. Comparison of glass transition temperature.

Composite	T_g (°C) from			
	Tan δ peak	E'	E''	DSC
kfc-ut	77.2	66.4	67.2	72.8
kfc-t	81.9	61.5	71.9	71.7
tfc-ut	95.6	54.9	79.6	73.7
tfc-t	84.5	51.0	75.5	72.2
ofc-ut	81.7	49.3	70.8	74.4
ofc-t	104.6	67.2	88.5	73.9

3.2 Glass Transition Temperature

The transition of a polymeric material from glassy to rubbery state has been recognized as an important material property. From the curves of storage modulus, E' , loss modulus E'' and $\tan \delta$, the glass transition temperature, T_g can be found either from the first inflection point of the storage modulus curve or from the peak of loss modulus curve or from the peak of $\tan \delta$ curve. ASTM D 4065 suggests that T_g to be taken as the peak of the loss modulus. The glass transition temperature of the material using DMA generally varies up to 25 °C due to the difference in methods adopted. The T_g from a DMA curve is slightly higher than the T_g measured using DSC as indicated in Table 2. Increase in glass transition temperature (T_g) of kfc-t and ofc-t is taken as a measure of the increased interfacial interaction upon treatment.

3.3 Scanning Electron Microscopy

The surface morphology of kfc-ut can be clearly seen in Fig. 5(a), wherein the individual fibers are protruding at the fracture surface. This indicates the poor wetting characteristic of the untreated Kenaf fiber (kfc-ut). The fracture surface of the kfc-t shown in Fig. 5(b) exhibits a relatively better bonding between fiber and the matrix. Contrary to the pull out of fibers in case of kfc-ut, there is a sharp breakage of fibers in case of kfc-t.

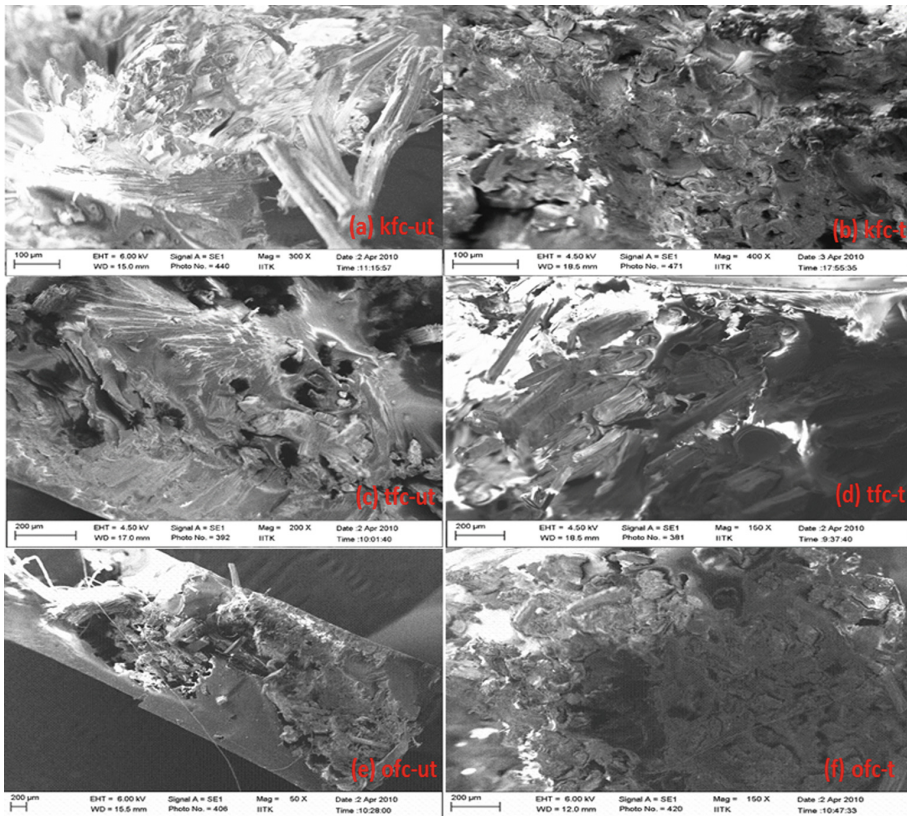


Fig. 5. SEM micrograph for fractured surface of (a) kfc-ut, (b) kfc-t. (c) tfc-ut, (d) tfc-t, (e) ofc-ut, (f) ofc-t.

The SEM image of the fractured tfc-ut shown in Fig. 5(c) the phenomenon of fiber pull out is clearly seen with many left out holes in the matrix. This indicates a weak fiber-matrix interface due to poor interfacial bonding. The fibers are seen to be protruding from the fracture surface indicating considerable elongation of the fibers before fracture. This elongation of the fibers can be considered as the absence of brittleness in the untreated *Thespesia lampas* fibers.

In the fractograph shown in Fig. 5(d) for tfc-t, clearly indicates a better interfacial adhesion and good wetting demonstrating a strong bonding with a paucity of traces of fiber pull out in the matrix. In addition, a sharp breakage of the fibers without any trace of elongation is observed near the fractured surface of the composite. This can be understood as the phenomenon of increase in the stiffness of the fibers due to alkali treatment.

The fracture surface of the ofc-ut shown in Fig. 5(e) clearly indicates a poor compatibility of the matrix and the fiber depicting poor adhesion between matrix and the fibers. It also indicates a poor penetration of the matrix with the absence of matrix around the fibers. This is due to lack of proper wetting of the fibers in an ofc-ut. However, there is

an improved bonding with less fiber pullout and better fiber dispersion of the matrix is observed in ofc-t composites as shown in Fig. 5(f). We can clearly see that no voids are visible between the matrix and the fiber as it is observed in case of the ofc-ut.

Thus the data obtained in DMA is highly indicative of the complex behavior of the material for the dynamic mechanical loading as well as temperature applications. Changes during the transition of a polymeric material from glassy to rubbery state can be observed by dynamic mechanical analysis. Similar to other properties, the dynamic mechanical properties depend on the type of fiber, length of fiber, fiber orientation, fiber dispersion and fiber-matrix adhesion.

4 Conclusions

- Higher storage modulus and glass transition temperature and lowest damping factor is noted for treated okra fiber composite (ofc-t). Good interfacial bonding as a result of NaOH treatment is observed in the SEM micrographs which has resulted in the superior properties.
- Loss modulus is highest for kfc-ut and hence the damping ratio is also high. Relatively low stiffness of kenaf fibers could have resulted in higher energy absorption.
- Highest glass transition temperature can be noted based on damping factor compared to other parameters. It is comparable with the value obtained by Differential scanning calorimetry.

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