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THE EFFECT OF TREATMENT ON KENAF FIBER USING GREEN APPROACH AND THEIR REINFORCED EPOXY COMPOSITES

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Abstract

In this study we show the novel treatment (surface modification) of kenaf fiber using amino acids and the studies of kenaf fiber reinforced epoxy composites. The kenaf fiber was treated at room temperature for 24h, using two amino acids namely glutamic acid (acid) and lysine (base). Thermogravimetric analysis (TGA) of the treated kenaf fiber (glutamic acid treated and lysine treated) showed more weight loss than compared to untreated fiber. The tensile studies of the composites suggested improved mechanical properties in both the cases, glutamic acid treated and lysine treated kenaf fiber than compared to untreated samples. Dynamic mechanical analysis (DMA) of the composites suggested that the storage modulus, loss modulus and tanδ were most influenced by the chemical treatment. The morphological studies of the kenaf fibers before and after treatment were examined using scanning electron microscopy (SEM) and revealed that the chemical treatment for 24h, removed any impurity from thefibers surface. In addition, morphological studies of fractured mechanical testing composite samples using SEM were performed to understand the de-bonding of fiber/matrix adhesion. The results, confirmed that the lysine treatment is more effective than the glutamic acid treatment on kenaf fibers.

Key words: Kenaf fibers, Thermal properties, Polymer matrix, Composites and Mechanical properties.

Introduction

Recently increasing attention has been given towards the development of green products in the field of composites. However, it has been confined to the usage space of synthetic materials inseveral engineering applications. The development of green composites or partially biodegradable composites is only defined based on the nature of their constituents. A huge changeover was reported on the usage of natural fibers all over the world for the production of green composites [1,2,3]. Moreover, natural fibers can be a suitable comparator to synthetic fibers, such as glass, in many ecological characteristics but not in respect of mechanical strength. The applications of natural fibers are growing in many sectors such as furniture, construction, automobiles and packing due to their low cost, low weight and less damage compared to synthetic fibers [4,5]. However, significant improvements in the strength of polymer composites can be achieved while reinforcing natural fibers under different conditions, i.e., by using various chemical treatments on the fibers and hybridization with synthetic fibers [6]. Still, the need for these treatments was identified as a lack of strength in these composites compared to other synthetic composites, whereas, weaving of natural fibers in different orientations makes the composites stronger and comparable to those of synthetic fibers [7].

Several studies have reported that the mechanical properties of natural fiber reinforced composites depend highly on the interface adhesion property between the fibers and the polymer matrix [8,9,10,11,12]. Natural fibers contain cellulose, hemicelluloses, pectins and lignin and are rich in hydroxy1 groups; natural fibers tend to be strong polar and hydrophilic materials whilst polymer materials are a polar and exhibit significant hydrophobicity [13]. In other words, there are significant problems of compatibility between the fiber and the matrix due to weakness in the interfacial adhesion of the natural fibres with the synthetic matrices. Therefore, surface modification of natural fibers by means of treatment is one of the largest areas of recent research to improve compatibility and interfacial bond strength [14,15].

In order to develop composites with better mechanical strength, it is necessary to impart hydrophobicity to the fibers through surface treatment. Surface treatments are often used to improve the performances of natural fiber reinforced composites by bridging the gap in compatibility between hydrophilic fibers and hydrophobic matrices [16]. Natural fibers can be modified either by physical or chemical means. Physical treatments change structural and surface properties of fibers and thereby influence their mechanical bonding to polymers [17]. Chemical methods of altering natural fibers involve introducing amaterial compatible with both fibers and matrix [18,19,20].

Chemical treatments such as bleaching, alkali and acetylation was found to be a technique to enhance the matrix–fibre adhesion by increasing roughness through of clean the fibre surface from impurities and by disrupting the moisture absorption process through of coat of OH groups in fibre [21,22]. Many studies have been carried out to improve the properties of natural fiber-reinforced composites (NFRCs) using various chemical treatments [23,24,25,26]. Vilay et al. investigated the effect of fiber surface treatment (NaOH) and fiber loading (0–20 vol.%) on the flexural properties of bagasse fiber reinforced unsaturated polyester composites (BFRUSP). They found that the NaOH treated fiber composites showed better flexural strength and modulus (increase by about 11% and 20% respectively) compared to untreated fibre composites [27].

Jannah and co workers investigated the woven banana fiber-reinforced unsaturated polyester composites by varying percentage of fiber volumes (from 5% to 20%) and using different chemical treatments via the vacuum bagging technique. The effects of fiber-content and fiber surface modifications on the flexural, impact and water absorption properties of the composites were reported and concluded that acrylic acid treatment resulted in improved mechanical and water absorption properties of the composites compared to alkali-treated and untreated fiber composites [28].

Mwaikambo et al [29]. studied alkalization or acetylationof plant fibers resulting in changes in surface topography of the fibers and their crystallographic structure. Atiqah et al [30]. reported treatment of kenaf fiber with 6% sodium hydroxide (NaOH) solution for 3 h using the mercerization method. The highest flexural, tensile and impact strengths were obtained in a hybridcomposite of UPE reinforced with treated kenaf fibers. Lai et al [31]. studied the morphological, physical and mechanical properties of natural fibers and resulting woven composites. They observed that kenaf fibers exhibit better tensile properties than betel palm fibers because of their higher cellulose content. From morphological observations, they also found that alkaline treatment of the fibers effectively cleans their surface and increases their surface roughness.

Though, there are several studies which reported on the various chemical methods to treat the natural fiber, the method employed in this study (natural fiber treatment using amino acids) is novel, to the best of our knowledge.

2. Experimental

2.1 Materials

Kenaf fiber was sourced from Agricultural Research Council of South Africa, Glutamic acid and Lysine was purchased from Sigma Aldrich, Epoxy resin and hardner were purchased from ATM composite, Durban and was considered as matrix material in this study.

2.2 Treatment of kenaf fiber

The kenaf fiber was treated with Glutamic acid solution and Lysine solution separately. The solutions were prepared up to 10% of the total weight of the fiber to be treated. The fiber was immersed in to the prepared solutions and allowed at room temperature for 24h. After 24h the fiber was removed from the solutions and washed three times using DI H_2O and allowed at room temperature to dry, after 2 days the dried fiber was used for further characterization.

2.3 Preparation of composites

Epoxy composite with treated and untreated kenaf fiber was prepared using resin casting method. The resin casting method consists mixing of epoxy and harnner together at room temperature and pourd in to a mold cavity, which is already filled with kenaf fiber. The mixture (resin/hardener) was poured into the top opening end of Perspex mold sheets 6 mm rubber gaskets at other 3 sides. The both sides (left and right) and bottom part of the mold were clamped while the top side was unclamped and left for mold opening after resin casting. The wax was used as the mold release agent; it was applied on the inner side of the mold and the treated and untreated kenaf fiber was randomly distributed before pouring of resin/hardener mixture. The cured composite was removed from the mold after 2 days and used for further characterization after 15 days of initial casting.

2.4 Thermogravimetric analysis (TGA)

Thermogravimetric analyses (TGA) of the treated and untreated fiber carried out using SDT Q600 TA Instruments. The specimens were scanned under N_2 atmosphere; from room temperature to 600 °C at a heating rate of 10 °C/min.

2.5 Tensile properties

Tensile properties were determined according to ASTM D 3039 test standard specifications [32]. The test was performed using MTS 793 servo-hydraulic 100 KN load-cell computer-controlled screw-drive multipurpose testing machine, with the speed of test taken to be 5mm/min. Specimens of 250 x 25 x 3 mm^3 in length, width and thickness by dimensions were cut from the untreated kenaf fiber and treated kenaf fiber laminate samples using 3000 series CNC router machine. Specimens were clamped on the MTS tester and pull until they broke apart. Five specimens were tested each samples and the mean values of the tensile strength, strain at break and modulus of elasticity of all the specimens tested were calculated.

2.6 Dynamic analysis (DMA)

The Dynamic Mechanical Analysis (DMA) was carried out using instrument (TA Q800). Rectangular specimens with sizes of 60 mm × 10 mm × 3.5 mm were made and used for the dynamic mechanical tests. Temperature scan from 25 °C to 160 °C at a frequency of 1 Hz was performed. The heating rate was Ramp 2.00 °C/min to 160.00 °C. Storage modulus (‘E) and mechanical loss factor (tanδ) were measured as the function of temperature during the test.

2.7 Scanning electron microscopy (SEM)

The fibers (treated and untreated) and composites; samples were gold-coated by an electro deposition technique to impart electrical conduction. The morphology of the fibers and composites was studied using JEOL JEM-7500F (Tokyo, Japan) operated at an accelerating voltage of 2 kV.

3. Results and Discussions

3.1 Kenaf Fibers

It is well known that natural fibers can be treated using various methods such as acetylation, merisization, grafting and silane treatment, in order to improve their mechanical properties. We focused on the development of biodegradable composites for mass transit applications in order to reach our approach to the natural fiber, kenaf fiber used in this study.. The fiber was treated using amino acids such as glutamic acid (acid; pH=3), lysine (base; pH=9) as shown in schem 1. Briefly, the glutamic acid and lysine solutions were individually prepared as per calculated 10 wt% of the fiber to be treated. The fiber was immersed in the prepared solution and left at room temperature for 24h. After 24h the fiber was removed from the solution and washed 3 times using DI water and allowed in oven for 12h at 55 °C for drying. The dried treated and untreated kenaf fiber was used for further characterization.

3.1.1 Thermal properties of kenaf fibers

Thermogravimetric analysis of the kenaf fiber before and after treatment showed three steps of weight loss, one is at about 50-150 °C due to the physorbed water, which is of very less percentage. The weight loss from 225-300 °C and 325-425 °C is ascribed to the removal of organic functional groups of kenaf fiber (cellulose, hemicelluloses, pectins and lignin) and amino acids, which used for surface modification. In all fibers the maximum weight loss is observed nearly at 350 °C. At 600 °C the treated fiber behavior is similar to untreated fiber as shown in Fig. 1a. The thermal stability of the treated and untreated fiber is shown in the Table 1. The second derivative of the untreated and treated fiber is suggested that the clear three steps of weight loss as shown in Fig.1b, 1c and Fig.1d.

3.1.2 Morphology of Kenaffibers

The morphological changes on the fiber surface before and after chemical treatment are shown in figure 2. The amino acid treatment leads to significant differences in the fibre surface morphology. The untreated fibers clearly showed impurities considerably covered with waxy substances on the surface as shown in Fig. 2a, which can influence the adhesion properties with the resin in the composites manufacturing. However the fibers, treated with glutamic acid (Fig. 2b) and treated with lysine (Fig.2c) showed the removel of the waxy layer and impurities from surface and the treated surface of fibre becomes rather rougher and fibrillation as compared tothat of untreated fibre. Moreover, it can be seen that the fibres have been spitted into finer fibres. This could lead to high interlock and adhesion between the fibres and the matrix.

3.2 Composites

All the composites prepared in this study are presented in Table 2. It is worth noting that the amino acid treatment of the fibers does not influence the thickness, the fibers weight fraction P_f. The specimens were cut from the prepared composites using the standards as shown in Fig. 3.

3.2.1 Tensile Properties

Fig. 4, shows the stress-strain curve of tensile properties of the untreated, glutamic acid treated and lysine treated kenaf fiber composites.The untreated and lysine treated composites showed the same failure modeand glutamic acid treated composite showed lower failure mode. However, in the case of neat epoxy resin longer failure modewas observed.

Fig. 5. Shows the tensile properties of composites. All of the composites show higher tensile moduli than the neat epoxy resin. Inparticular, the tensile modulus strongly increases using lysine treated kenaf fibers as reinforcement. The neat epoxy resin and untreated shows tensile moduli 2.11 GPa and 2.33 GPa respectively. Moreover, glutamic acid treated and lysine treated composites show tensile moduli 2.46 GPa and 2.66 GPa respectively.

However, it is worth noting that the treatment of kenaf fibers in amino acid solution influences the tensile properties of composites. In particular, the lysine treated composites show tensile strength and modulus higher than untreatedand glutamic acid treated composites as shown in Fig. 5a and 5b. These results confirm that the treatment of kenaf fibers in amino acid solution for 24h improve their compatibility with the hydrophobic polymer used as matrix. Therefore, the slightly increase of mechanical properties of thetreated fibers than the untreated ones, observed previously, is widely compensated by the cleaner surface of the treated fiber that promotes a better adhesion between reinforcement and matrix with a consequent improvement of the mechanical properties of the composite.

Nevertheless, the untreated and treated composites show lower tensile strength than neat resin as shown in Fig. 5b, even if fiber chemical treatment was performed. This means that, if the fibers are randomlydispersed in a polymer matrix, they act as stress concentrators leading to premature failures of composites. Similar results have been obtained for epoxy composites reinforced with lignocellulosic particles [33].

3.2.2 Dynamic Mechanical Analysis (DMA)

Fig. 6a, shows the variation of storage modulus E’ as a function of temperature of neat resin, untreated and treated composites at frequency of 1 Hz. The value of E’ was found to be higher for kenaf fiber treated with lysine composite (i.e. 2937 MPa) than treated with glutamic acid composite (i.e. 2791 MPa), untreated (i.e. 2809MPa) and neat resin (i.e. 2650 MPa) in the plastic region (i.e. at low temperature).

At higher temperatures, due to loss in stiffness of both the fiber and the matrix, the storage modulus drops. It is worth notice that composites reinforced with amino acid treated fibers had a lower fall in the value of E’ when temperature is increased compared both tocomposites reinforced with untreated fibers and to neat resin. Inparticular, the storage modulus at 100 °C of the treated with lysine compositeswas found to be about 65.7 MPa, notably higher than those of the treated with glutamic acid composites (49.4 MPa), untreated (54.8 MPa) and neat resin (9.2 MPa), respectively.

Overall, the lysine treatment of natural fibers slightly influences E’ of the composites in the plastic region whereas, if the mercerization of the fiber was performed, the storage modulus greatly increases in the rubbery region. This trend confirms that the treatment of the hydrophilic kenaf fibers in amino acid solution for 24h improve their compatibility with the hydrophobic polymer used as matrix.

Loss modulus E’’ represents the viscous response of the material. Fig. 6b, shows the variations of E’’ of neat resin, untreated, treated with glutamic acid and treated with lysine composites, as function of temperature. It can be noted that in all the samples loss modulus increased in the plastic region and then decreased with increasing temperature in the rubbery region. The effect of chemical treatment is found to influence on the value of E’’ below and above the glass transition temperatures (temperature at peak values of E’’). In both the plastic region and in the rubbery region, E’’of neat resin was found to be lower than the untreated and treated composites. In particular, the amino acid treatment of kenaf fiber leads to improvement of the loss modulus of composites at high temperature.

According to the published reports; Tanδ, also named damping, is determined by the ratio of loss modulus E’’ and storage modulus E’. Incorporation of fibers in a composite system affects the damping behaviour of the composites, which is due to shear stress concentrations in the fibers along with viscoelastic energy dissipation in the matrix [34]. Tanδ depends on the filler–matrix adhesion: i.e. a weak filler–matrix adhesion leads to higher values of tanδ [35] while a good filler–matrix adhesion limits the mobility of the polymer chains thus reducing the damping. In particular, low damping means that the particular composite has good load bearing capacity.

Fig. 6c, shows that the presence of the lysine treated and glutamic acid treated kenaf fibers dramatically reduce tanδ, thus indicating the presence of good adhesion, resulting in low damping. These results confirm the good effect of the amino acid performed on the fiber/matrix compatibility, resulting in improved stress transfer and good interfacial adhesion.

The glass transition temperature Tg can be calculated as the temperature at which the damping or the loss modulus [36] attain their maximum values. As shown in Fig. 6d, the glass transition temperature is slightly influenced by the presence of treated kenaf fibers in the epoxy matrix, when compared to untreated and neat resin composites,. In particular, the T*g* varies from 63.49 °C for neat resin to the range between 62 °C, 62.15 °C and 62.35 °C for untreated, treated with lysine and treated with glutamic acid composites respectively, thus no shift of T*g* to higher temperatures can be attributed to the presence of untreated kenaf fibers. The presence of untreated kenaf fibers does not reduce the mobility of the matrix chains whereas the amino acid treatment of kenaf fibers improves the fiber–matrix interfacial adhesion thus reducing the mobility of the matrix chains.

3.2.3 Morphology of the Composites

The fractured surface of lower-strength composites due to tensile loading was selected as a representative sample for the interpretation of the degree of interfacial adhesion. SEM images of the randomly oriented kenf fiber composite after fractureunder tensile loading are shown in Fig. 7. The untreated kenaf fiber composite resulted a broken fibers, crack propagations through the matrix and de-bonding of fibers at the interface as shown in Fig. 7a. The glutamic acid treated kenaf fiber composite shows the kind of debonding between the fiber and the matrix as shown in Fig. 7b, but still not appear to be to the maximum extent. The lysine treated kenaf fiber composite showed a closely packed interfacial bonding between fiber and matrix is evident from Fig. 7c. This also shows a hollow cross-section of a single kenaf fiber along with the crack propagation direction. The appearance of transverse cross-sections of the fiber end reflects the absence of fiber pullout and ensures enhanced interfacial adhesion.

4. Conclusion

The kenaf fiber was successfully treated using amino acids (glutamic acid and lysine). The effect of amino acid treatment of kenaf fibers and their epoxy composites on thermal and mechanical properties was evaluated as follows:

- The chemical treatment for 24h allowed cleaning the fibers surfaceremoving each impurity;
- The thermal stability of the lysine treated kenaf fiber more compare to glutamic acid treated kenaf fiber;
- All the composites showed higher tensile moduli than the neat epoxy resin;
- The chemical treatment for 24h improved the mechanical propertiesof the composites in both glutamic and lysine treated fiber used as reinforcement;
- The storage modulus was most influenced by the chemical treatment in the rubbery region rather than in the plastic region;
- The amino acid treatment led to a notable reduction of tanδ peaks inaddition to significant shifts of tanδpeaks to higher temperatures;
- The trends of storage modulus, loss modulus and damping of the composites were not influenced by the stacking sequence.

These results confirm that the amino acid treatment improves fiber–matrix interfacial adhesion thus reducing the mobility of the polymerchains and enhancing stress transfer. Moreover lysine treatment is more effecting than the glutamic acid treatment.

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Tables and Captions

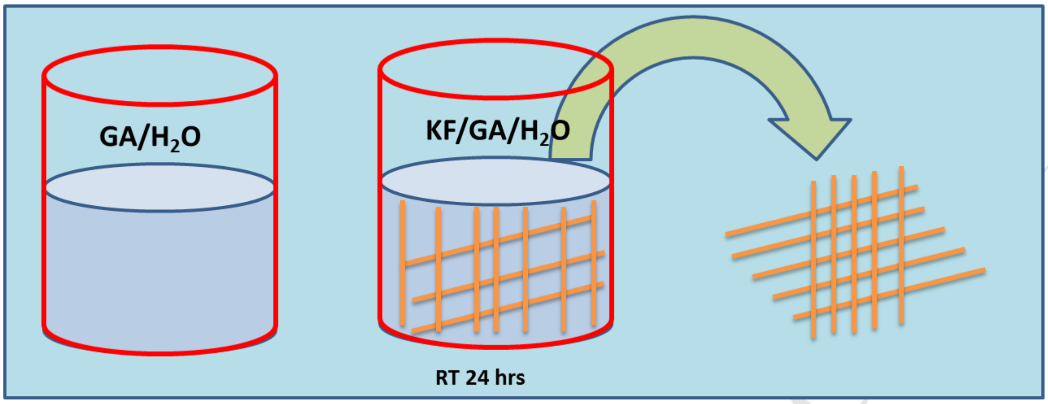
Table 1. Thermal stability of the treated and untreated kenaf fiber.

Sample Configuration	**Maximum degradation temperature °C (T_{max})**	**Residue at 600°C (Wt %)**
Untreated Kenaf fiber	339.60	27.34
Treated with lysine	347.96	31.78
Treated with glutamic acid	350.50	36.58

Table 2. Kenaf fiber/ Epoxy Laminates investigated

Sample	Untreated KF	Glutamic acid Treated KF	Lysine Treated KF
Fiber length	60-30mm	60-30mm	60-30mm
Thickness (mm)	4 ± 0.10	4 ± 0.05	4 ± 0.13
P_f (%)	~ 41%	~ 41%	~ 41%

Figures and Captions



Scheme 1. Schematic illustration of fiber treatment.

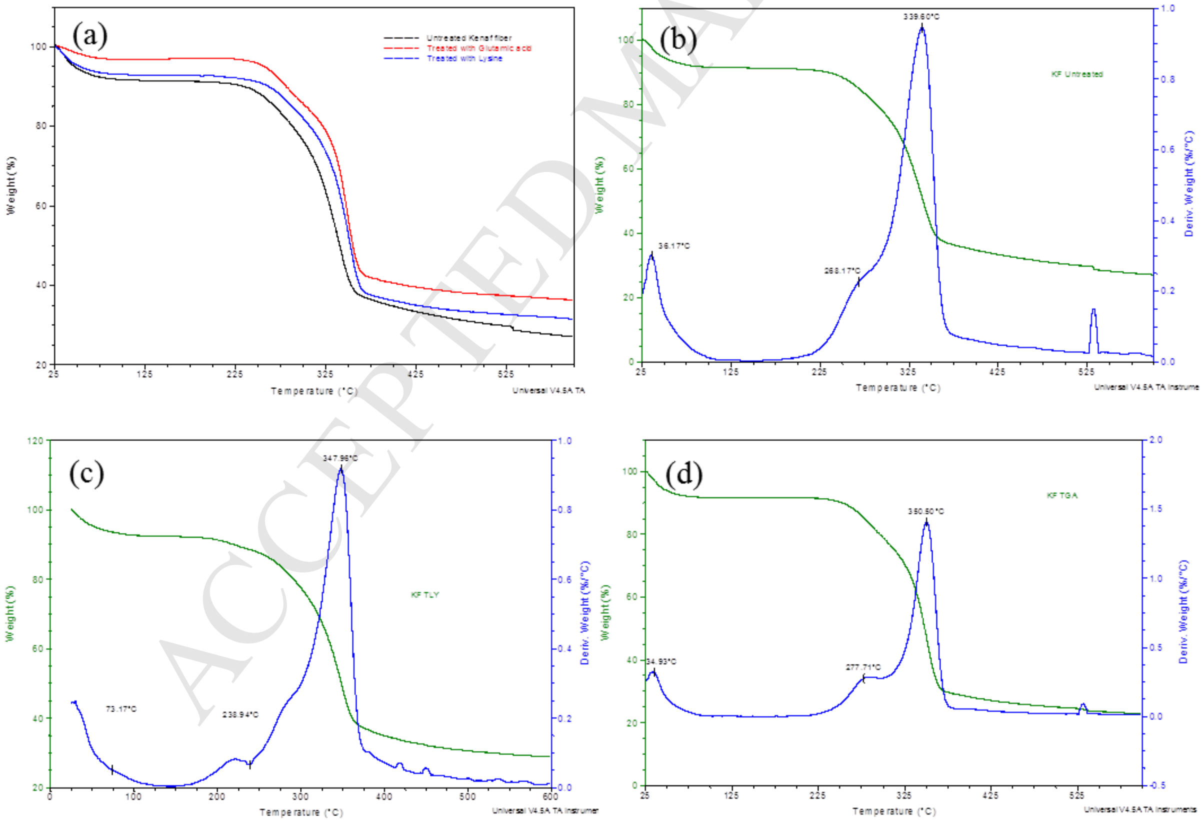


Fig. 1. Thermogravimetric curves of the samples; (a). Overlay of the treated and untreated kenaf fibers, (b). Second derivative of untreated kenaf fiber, (c). Second derivative of treated kenaf fiber with lysine(d). Second derivative of treated kenaf fiber with glutamic acid.

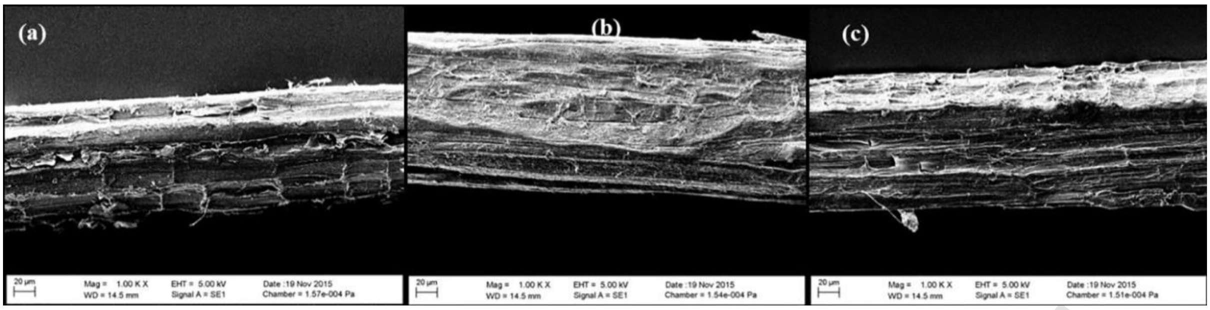


Fig. 2. SEM images on the surface of kenaf fibers; (a). Untreated, (b). Glutamic acid treated and (c). Lysine treated.

Fig. 3. Specimens used for testing; (a) Epoxy Resin, (b) Untreated Kenaf fiber, (c) Treated fiber with Glutamic acid, (d) Treated fiber with Lysine

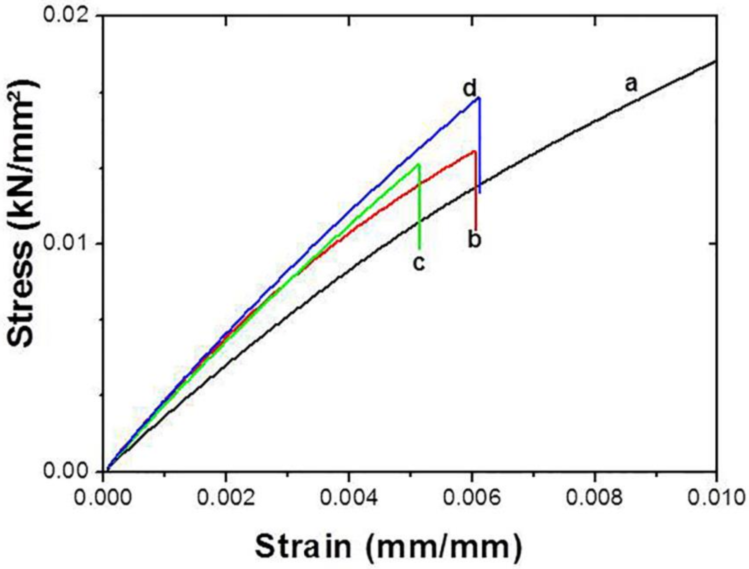


Fig. 4. Typical stress/strain curves of kenaf fiber composites obtained from tensile tests; (a). Neat resin, (b). Untreated, (c). Glutamic acid treated, (d). Lysine treated.

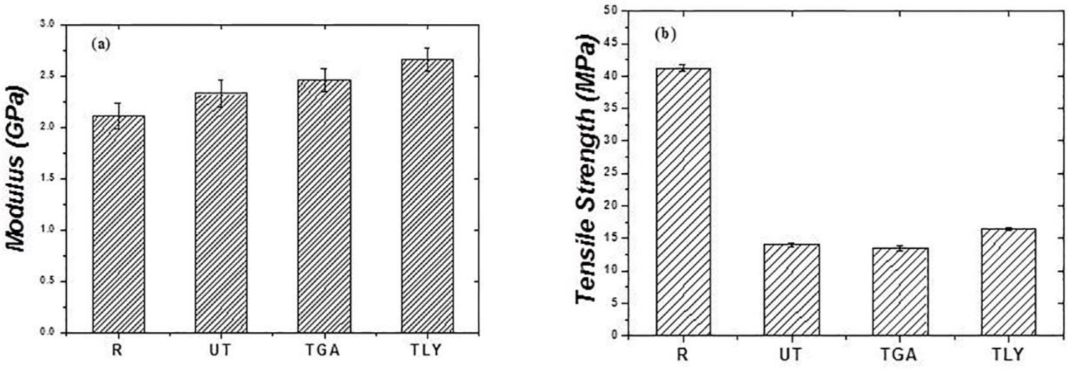


Fig. 5. Tensile properties of composites: (a) Tensile Modulus and (b) Tensile Strength.

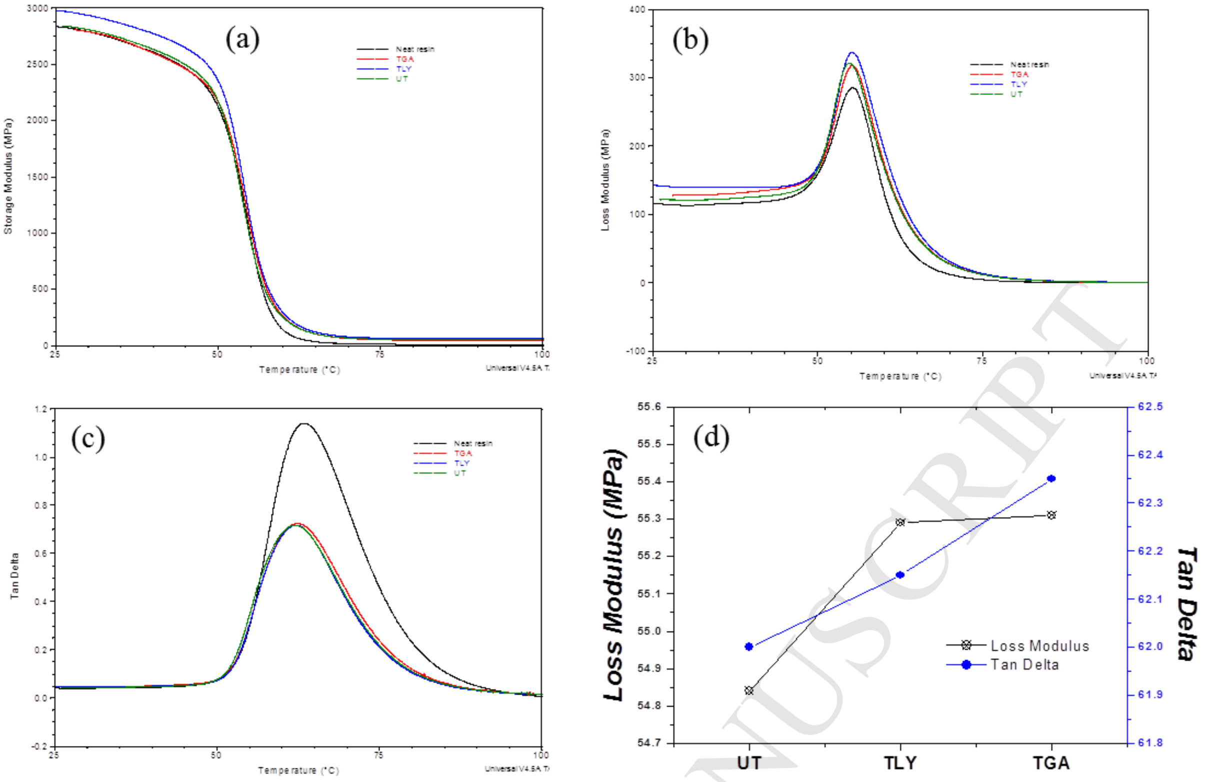


Fig. 6. DMA results of the composites; (a). Storage modulus, (b). Loss modulus, (c). Tan delta, (d). Graph of peak maximum values of Loss modulus and Tan*δ*.

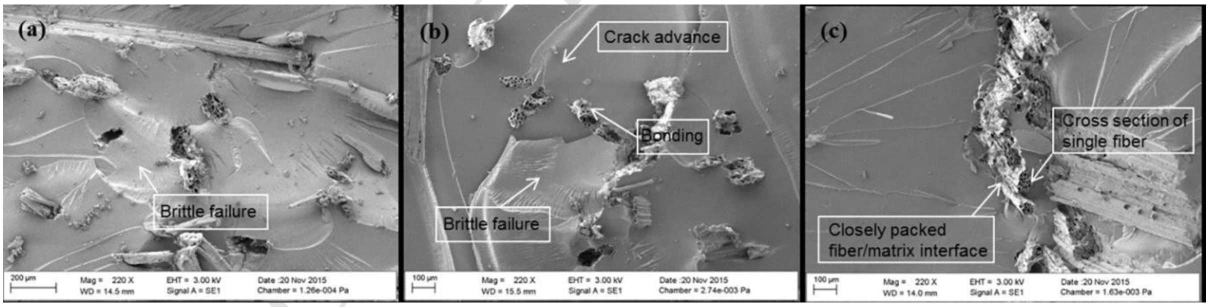


Figure 7. SEM images of tensile fractured specimen of randomly oriented kenaf fiber epoxy cpomposites: (a). Untreated, (b). Glutamic acid treated, (c). Lysine treated.