

Mechanical And Morphological Properties Of Raw And Alkali Treated Borassus Fruit Fiber Reinforced Epoxy Composites

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Abstract

The use of natural fibers as reinforcement in Polymer Matrix Composites results in light weight, high specific strength and eco friendly Biopolymer materials. The raw and 5% alkali treated Borassus fruit fiber reinforced epoxy composites were prepared using five different fiber lengths 1 mm, 3 mm, 5 mm, 7 mm and 10 mm respectively. The effects of fiber length as well as alkali treatment on tensile and impact properties were evaluated as per ASTM standards. It was found that the alkali treatment of the fibers improved the tensile and impact properties. The influence of fiber length is an important factor in the reinforcement of composites and it was observed that the 5mm length alkali treated fiber reinforced composites contribute better mechanical properties when compared to other length fiber composites. The Fourier Transform Infrared Spectrometry (FTIR) and Scanning Electron Microscopy (SEM) analysis on tensile fractured specimens were also carried out to find out the chemical compounds present in the composites and fiber matrix adhesion characteristics.

KEY WORDS: Biopolymers, Scanning Electron microscopy, Natural fibers, Alkali treatment, FTIR, Mechanical properties.

1. Introduction

The interest among researchers is focused on employing the natural fibers as reinforcement in Polymer Matrix Composites due to their attractive characteristics

like renewability, light weight, low density and carbon neutrality. Moreover, the attempt will contribute towards safer and cleaner environment. The flexibility of natural fibers, when comparing to brittle synthetic fibers, made it potential reinforcement to polymers.

Boopathi et al. [1] studied the physical, chemical and mechanical properties of the Borassus fruit fibers. In their study they have found that 5% alkali treatment improved the mechanical properties of Borassus fruit fiber and suggested the application of the same in the reinforcement of composites for structural applications. Gomes et al. [2] developed a green composite with curaua/cornstarch-based biodegradable resin. A comparison of the mechanical properties of raw and alkali treated fiber reinforced composites was made. The tensile test result proved that alkali treatment improved the fracture strain three times more than that of the raw fiber composites. This result emphasized the significance of alkali treatment to the cellulose based fiber composites. The chemically modified coconut fibers / polyester resin composites were investigated for their mechanical properties by Mulinari et al. [3]. The natural fibers have some disadvantages like high moisture absorption and poor wettability. These effects on mechanical properties of Jute fiber reinforced with polyester matrix were studied by Akil et al. [4]. The flexural properties decreased due to these effects.

The influence of alkali treatment on the flexural and impact properties of rice husk with polyethylene matrix was investigated by Favoro et al. [5]. The chemical modification of the fiber surface, improved the fiber matrix adhesion characteristics. Sgriccia et al. [6] performed the experimentation on raw and treated fiber composite surfaces. They evidenced the removal of hemicelluloses and lignin from the natural fiber surfaces by alkali treatment. Mysamy and Rajendran [7] modified the agave fibers by alkali treatment and compared the mechanical properties like tensile, compressive, flexural and impact results with raw fiber reinforced composites. They confirmed that due to the alkali treatment the fiber-matrix adhesion and hence the mechanical properties of composites increased. Asasutjarit et al. [8] studied the chemical composition modification and surface modification of coir fibers with different pretreatment categories and observed that the mechanical properties, modulus of rupture and internal bond increased due to the same.

The effect of addition of coupling agent in the palm fibers /polypropylene composites was studied by Oliveira et al. [9]. The flexural strength and modulus were found to be improved. Haque et al. [10] compared the mechanical properties of palm and coir fiber reinforced polypropylene bio-composites. It was observed that the treated fiber reinforced specimens produced better mechanical properties when compared to the raw composites. The coir fiber composites had better mechanical properties than palm fiber composites. Franco and Gonzalez [11] studied the mechanical properties of henequen fiber/ polyethylene composites. It was found that the mechanical properties improved from 3 % to 43% after the alkali treatment of fibers.

Jacob et al. [12] investigated the fiber matrix adhesion of sisal fabric with natural rubber. The chemical treatments like modifications, mercerization, silanation and its effect on the mechanical properties were also analyzed. Bos et al. [13]

evaluated the mechanical properties of flax/polypropylene compounds. It was found that the flax fibers were effective in strength and stiffness improvement of compounds. Oksman et al. [14] reviewed the mechanical properties of composites reinforced with different natural fibers like jute, flax, sisal and banana with polypropylene matrix. He observed that sisal fibers recorded the best impact properties and flax fibers would improve in terms of flexural properties with the increase of fiber content. Also the highest stiffness was achieved by the addition of jute fibers. Monteiro et al. [15] observed properties of coir fiber/polyester composites with fiber loading below and above 50%. The results showed that composites with less than 50% fibers were rigid and the increase of fiber loading above 50% would make them flexible agglomerates. Harish et al. [16] carried out the mechanical property evaluation of natural fiber coir composites. The tensile, flexural and impact properties of coir fiber composites were compared with the glass fiber reinforced composites and the results indicated that the coir could be used as an alternative reinforcing material in low load bearing thermoplastic applications.

Sun et al. [17] compared the mechanical properties of sisal fiber and jute fiber. The critical fiber length of the sisal fiber was found to be 2.27 mm. They also highlighted that the fiber matrix adhesion would be low for the reinforcing fibers which were much longer than the critical fiber length. Jarukumjorn and Suppakarn [18] hybridized the glass fiber with the sisal/ polypropylene composites and studied their tensile, flexural and impact strength for them. They confirmed that the glass fiber hybridization improved the mechanical and thermal properties. Anuar et al. [19] presented the mechanical properties of kenaf fiber and glass fiber hybridization with thermoplastic natural rubber. The increase of kenaf fiber content decreased the tensile properties of the composite.

Shubhra et al. [20] used the SEM analysis to study the fracture behavior and fiber pullouts of Silk reinforced gelatin based composites. Rocha et al. [21] identified the chemical compounds of the raw and modified curaua fiber by the FTIR spectrum. Storozheva et al. [22] studied the possible role of acidity and basicity in surface chemistry using the FT-IR spectrum. Vibrational mode frequency calculations of chlorophyll-d were analyzed using the FTIR spectrum by Hastings and Wang [23]. Amoriello et al. [24] evolved two acid steps sol-gel phases by FTIR and compared the SEM analysis results with it. Singha and Thakur evaluated the mechanical, thermal and morphological properties of *grewia optiva* fiber/polymer matrix composites and found that the mechanical properties were improved up to 30% fiber loading [25]. Grafting of safadariffa fiber resulted in morphological transformations and improvement in physical, chemical and mechanical properties and novel regenerated copolymers were developed by chauhan and kaith [26].

The objective of this work is to prepare an eco friendly composite with natural fiber reinforcement and to replace synthetic fibers. Palm trees yielding *Borassus* fruits are abundantly available all over the world more so in Asian countries. The composite was made with raw and alkali treated *Borassus* fruit fibers as reinforcements in the polymers and the effect of fiber length, alkali treatment on the mechanical properties were investigated. The surface and fiber matrix interaction of composite materials were analyzed using FTIR and SEM.

2. Materials and methods

2.1 Fiber preparation

The Borassus fruits were collected from palm trees. The extraction process was initiated by immersing the fruits in water for a week. The retting process debonds the flushes which were sticking to the fibers. The fruits were then washed in running water. Mild pressure was applied on the fruits during washing for the maximum removal of flushes. The fruits were again immersed in water for another three days and the process was repeated till the remaining flushes were removed. The fibers were then dried in shade for 24 hours. The fibers were exposed to sunlight for half an hour for complete drying. They were smoothly rammed for removing short and particle fibers. The average fiber diameter was measured by air wedge method and was found to be 0.2412 mm. The length of the fibers varied from 50 to 110 mm. The composition of Borassus fruit fiber was found to be 68.94% cellulose, 14.93% hemicellulose, 5.37% lignin, 6.83% moisture content and 0.64% wax content [1].

2.2 Alkali treatment

The raw Borassus fruit fibers were washed in water for the removal of particulates and dried at room temperature for two days. The fibers were then treated with 5% Sodium hydroxide (NaOH) solution for half an hour at room temperature. The fibers were washed with 2.5% Hydro Chloric acid (HCl) for neutralization. The fibers were rinsed with distilled water thoroughly and then dried at room temperature for 24 hours.

2.3 Resin

Epoxy resin was used as a matrix material since they offer excellent adhesion, low shrinkage and there will not be any volatile matters during curing. They offer better performance even at elevated temperatures. The resin was prepared with a mixture of epoxy LY556 of density 1.2 g/cm³ and hardener HY951 of density 0.98 g/cm³ at a weight ratio of 10:1.

2.4 Composite Preparation

Composites were fabricated using Borassus fruit fibers and epoxy resin by resin casting method. Epoxy resin and Borassus fruit fibers were mixed with the ratio of 65:35. Both raw and 5% alkali treated Borassus fruit fibers were used for making composites. The Borassus fruit fibers were cleaned, dried and chopped into 1 mm, 3 mm, 5 mm, 7 mm and 10 mm fiber lengths. Borassus fruit fibers and epoxy resin were mixed thoroughly and spreaded uniformly in the mould of size 180 mm x 140 mm x 10 mm. Then the setup was kept in an oven at a temperature of 60 °C to avoid void contents and ensure uniform wetting. Then the top plate was covered and compressed up to 24 hours for complete curing. 5% raw and alkali treated composites were thus prepared and removed from the mould.

2.5 Tensile test

The tensile test specimens were made from the composite plates as per the ASTM D 638-03 -Type I [26] standard. Tensile test specimens were tested in Electronic

Tensometer – Model PC 2000 operated with a 20KN load cell with digital load controller and extension microprocessor based elongation measurement set up. The cross head speed was 5 mm/min and the gauge length maintained was 50 mm. The fractured tensile test specimen is shown in Figure 1. The tensile test was conducted at 28 °C and at a relative humidity of $50 \pm 2\%$. The grippers held the specimen in the longitudinal axis and the load was applied over the specimen. The loads and corresponding strains were noted. Five samples were tested for each composition.

2.6 Impact Test

The Impact test specimens were made from the composite plates as per the ASTM D 256-05 [27] standard. The size of the specimen is $64 \times 13 \times 5 \text{ mm}^3$ and the specimens were notched. The Izod digital impact tester, Frank-53568 was employed for conducting the impact test at room temperature and the corresponding impact strength were recorded. Five samples were tested for each composition and the average values were calculated.

2.7 Scanning Electron Microscope analysis

Raw and treated tensile fracture surfaces of the specimens were observed using JSM-6390 Scanning Electron Microscope. SEM micrographs provided the information about the surface morphology of the tensile fracture specimens. The specimens were scanned by high energy electron beam by raster fashion. The interaction of thin electron beam on atoms of the specimen provided three dimensional magnified appearances of the surfaces. The SEM analysis for both raw and alkali treated specimens of all compositions were compared.

2.8 FTIR analysis

Fourier Transform Infrared Spectra for the specimens were recorded using Thermo scientific Nicolet IS10 Spectrometer at room temperature. The resolution of the spectrometer is 4 cm^{-1} and the range is $4000\text{-}400 \text{ cm}^{-1}$. The specimens were exposed to the infrared light. When the vibrational frequency of the bond matches with the infrared light frequency, the absorption happened. The interferogram was recorded and the spectrometer performed the Fourier transform operation to obtain the spectrum. FTIR spectrum is used to analyze the chemical compounds and functional group of composites.

3. Results and Discussions

3.1 Tensile properties

The tensile properties of raw (UT) and 5% alkali treated (T) Borassus fruit fibers reinforced epoxy composites with different fiber lengths were analyzed and shown in Figure 2 - 4. It is evident that the alkali treatment to the Borassus fruit fibers removes the impurities from the fiber surface [6] and imparts better interfacial bonding between fibers and matrix [11]. The strong fiber matrix interlocking thus influenced

better tensile performance for the treated fiber reinforced composites. During tensile loading the fibers failed by breakage instead of fiber pull out for the alkali treated specimens, due to better interfacial bonding. The alkali treated fibers are flexible than the raw fibers and hence the engineering stress to strain rate, true stress to strain rate improved than that of the raw specimens.

5 mm length alkali treated fiber reinforced specimen withstood more loads (1938.72 N) with the displacement of 7.81 mm when compared to other specimens (Figure 2). The tensile strength and modulus of the Borassus fruit fiber influenced the tensile strength of the composite. Also it produced elevated engineering stress and true stress values than the other specimens (Figure 3 & Figure 4). The 5mm alkali treated specimen failed at a maximum true stress value of 24.29 MPa. Since alkali treatment to the fiber removed the surface impurities from the fiber, better mechanical interlocking between fiber and resin developed and hence the adhesion characteristics got improved [1]. It is observed that the alkali treatment improved the tensile strength and elongation properties. The critical fiber length is the minimum fiber length required to transfer the load effectively in composites. Since 1 mm fiber length is far below the critical fiber length, the bonding strength and hence the tensile strength observed was low. The 3 mm fibers were short in length hence detachment and fiber pullouts were the reason for their low performance. The 5 mm length fibers were well embedded with matrix and indicated that it is close to critical fiber length at which the fiber matrix adhesion properties were superior. The increase in fiber length after 5 mm might cause entanglement of fibers in the matrix and hence leads to the reduction in tensile properties.

3.2 Impact properties

The impact strength for the raw and alkali treated fibers of 1 mm, 3 mm, 5 mm, 7 mm and 10 mm length fiber reinforcements on the composite specimens is shown in Figure 5. The alkali treated fiber reinforced composites improved the impact strength to 25 - 30% more than the raw fiber reinforced composites. The fiber shrinkage and removal of cellulose during the alkali treatment imparted good interfacial bond strength between the fibers and matrix and hence improved the impact strength [7]. It is interesting to note that the 5 mm length reinforcement increased the impact strength of composites than the other cases. The impact strength of 5 mm raw and treated fiber reinforced specimens was 128.3 J/m and 181.76 J/m respectively. It was observed that 1 mm fiber length reinforcement produced the lowest impact strength of 85.5 J/m and the increase in the fiber length after 5 mm gradually decreased the impact strength. This is due to the fact that 1mm fiber is far below the critical fiber length and for the much longer fiber than the critical fiber length, the fiber matrix adhesion would be low [17].

3.3 SEM of tensile test specimens after fracture

The surface morphology of the tensile test fractured specimens for 1 mm and 5 mm length raw and alkali treated Borassus fruit fiber reinforced composites are shown in Figure 6 a-d. The SEM image reveals the significance of fiber-matrix adhesion. The Figure 6 a shows the SEM image of 1mm length raw fiber

reinforced tensile fractured specimens, in which the presences of voids were noticed. The voids were created due to the fiber pullouts during the tensile loading. This is an indication of poor fiber- matrix interfacial bonding and adhesion.

Figure 6 b shows the SEM image of 1mm length alkali treated fiber reinforced tensile fractured specimens. The alkali treatment improved the adhesion characteristics and rich matrix was observed. Since 1 mm fiber length is not enough to reinforce the matrix, the fiber pullouts lead to the voids and poor bonding.

Figure 6 c shows the 5mm length raw fiber reinforced tensile fractured specimens, in which the poor bonding was observed. The various surface impurities present on the fiber surface acted as a layer in between the fiber and matrix and thus lead to the poor bonding. Since 5 mm length fibers are noticed close to critical fiber lengths lesser voids are found due to fiber pullouts.

Figure 6 d shows the 5mm length alkali treated fiber reinforced tensile fractured specimens. The shearing off of the fiber evidenced the good adhesion characteristics of the fiber with the matrix. The alkali treatment increased the surface area of contact between the fiber and matrix and hence improved the bonding characteristics. The visualization of rich matrix confirmed the better flow of resin and bonding with the fiber. The SEM images of the treated specimens thus revealed the significance of alkali treatment and improvements on tensile properties of composites.

3.4 Fourier Transform Infrared Spectrometry analysis

The Fourier Transform Infrared Spectrometry analysis was made for the raw (UT) and 5% alkali treated 1 mm and 5 mm length Borassus fruit fiber reinforced composites and the chemical compounds were identified.

Figure 7 a - shows the spectrum for 1 mm raw fiber reinforced composites. The band at 1457.42 cm^{-1} showed the C-C stretching of the aromatic ring [21]. The peak at 1653.05 cm^{-1} indicated the presence of C=O stretch of acetyl group of hemicellulose. The peak at 2360.15 cm^{-1} is the indication of carboxylic acids with O-H stretching and also the increase of oxygen atoms but not enough to form carbonate atoms [22]. The peak at 2922.76 cm^{-1} represented the C-H modes of methyl and methylene groups. The band around 3000 cm^{-1} to 4000 cm^{-1} was observed with ups and downs. This band is assigned to symmetric and asymmetric N-H stretching [24].

Figure 7 b - shows the spectrum for 1 mm treated fiber reinforced composites. The peak at 1653.05 cm^{-1} was reduced and hence it is the sign of partial removal of hemicelluloses due to alkali treatment. The band at 1457.24 cm^{-1} showed little increase in the C-C stretching of the aromatic ring. The peak at 2360.12 cm^{-1} was found little decreased. It is the sign of carboxylic acids with O-H stretching with the decrease of oxygen atoms. The smooth band without many ups and downs between 1716.92 cm^{-1} to 2360.12 cm^{-1} was interpreted as good molecular strength of treated fiber reinforced specimens [7]. The peak at 2921.25 cm^{-1} was interpreted for C-H modes of methyl and

methylene groups. The reduction of the same was noticed in these specimens than the raw specimens [23]. The band around 3000 cm^{-1} to 4000 cm^{-1} was observed with ups and downs as like the raw specimens. This band is assigned to symmetric and asymmetric N-H stretching [24]. The peak at 3367.60 cm^{-1} improved with little distortion and showed the Nitrogen bond which increased fiber matrix adhesion in these specimens than raw specimens.

Figure 7 c - shows the spectrum for 5 mm raw fiber reinforced composites. C-C stretching of the aromatic ring was assigned to the peak at 1457.15 cm^{-1} and the peak 1653.19 cm^{-1} showed C=O stretch of acetyl group of hemicellulose. The carboxylic acids with O-H stretching were assigned to the peak at 2360.50 cm^{-1} . More ups and downs were noticed between 3000 cm^{-1} to 4000 cm^{-1} band.

Figure 7 d - shows the spectrum for the 5 mm treated fiber reinforced composites. The alkali treatment removed the hemicellulose partly and hence the peak at 1638.48 cm^{-1} was found reduced. The C-C stretching of the aromatic ring was reduced and was indicated by the peak 1456.97 cm^{-1} . The peak at 2361.25 cm^{-1} decreased and it is the sign of carboxylic acids with O-H stretching with the decrease of oxygen atoms. The stable band without much ups and downs between the peaks 1683.34 cm^{-1} to 2343.15 cm^{-1} was interpreted as for maximum molecular strength of treated fiber reinforced specimens. The peak at 2924.97 cm^{-1} represented the existence of C-H modes of methyl and methylene groups. The band around 3000 cm^{-1} to 4000 cm^{-1} was observed with minimum ups and downs for the treated fiber reinforced specimens when compared to 5 mm raw fiber reinforced specimens. The existence of strong stable Nitrogen bonds at peak 3526.16 cm^{-1} was observed. It is interesting to note that this peak 3526.16 cm^{-1} for the 5mm treated fiber specimen was superior to the peak of 3367.60 cm^{-1} for the 1mm treated specimen. The FTIR results for the both 1 mm, 5 mm treated and raw fiber reinforced specimens thus showed that the alkali treatment of the fibers facilitates strong fiber matrix interfacial bonding and adhesion in the composites. Out of two fiber types 1 mm and 5 mm, the 5 mm treated fiber reinforcement was observed to give superior properties to the composites.



FIGURE 1 Fractured Tensile test specimen.

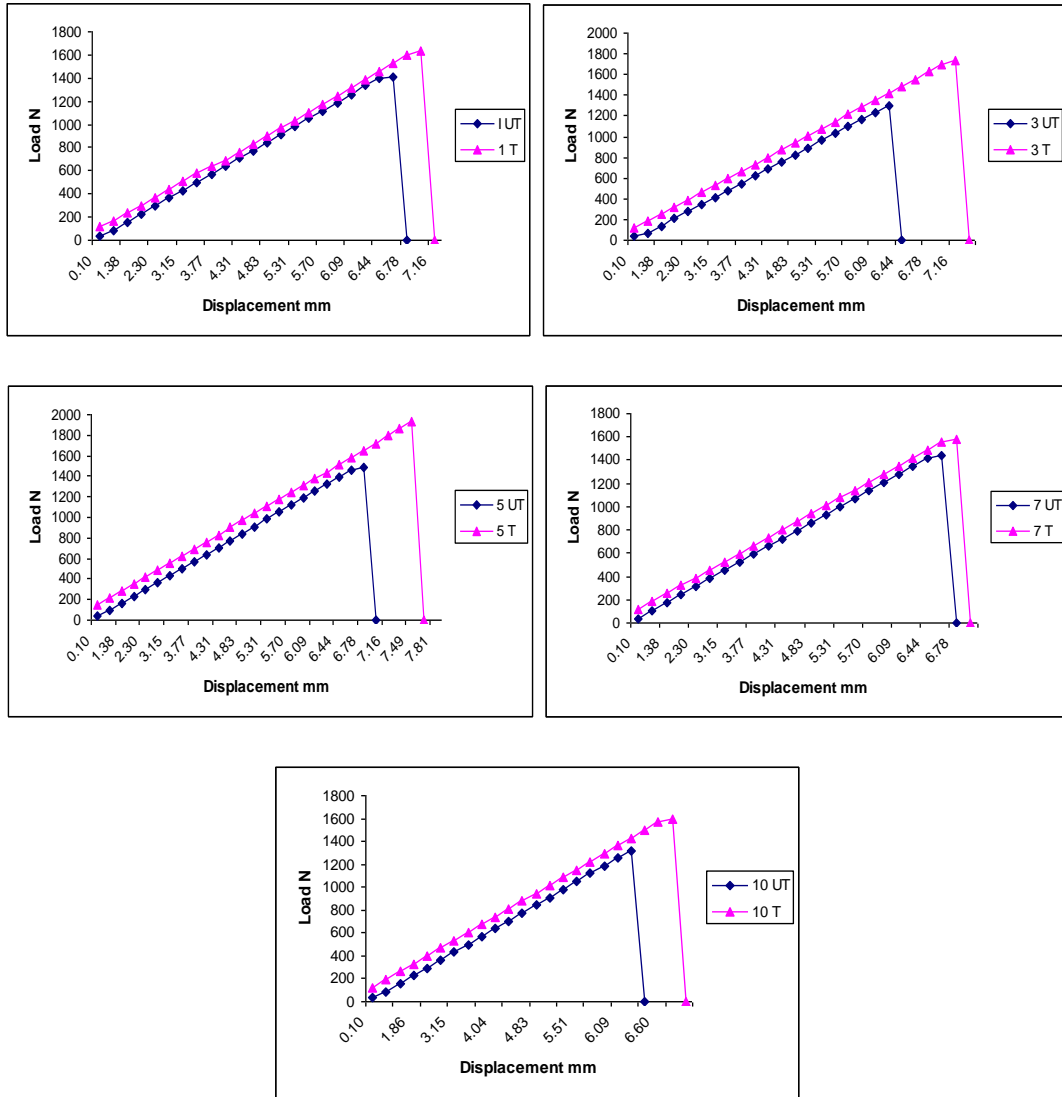
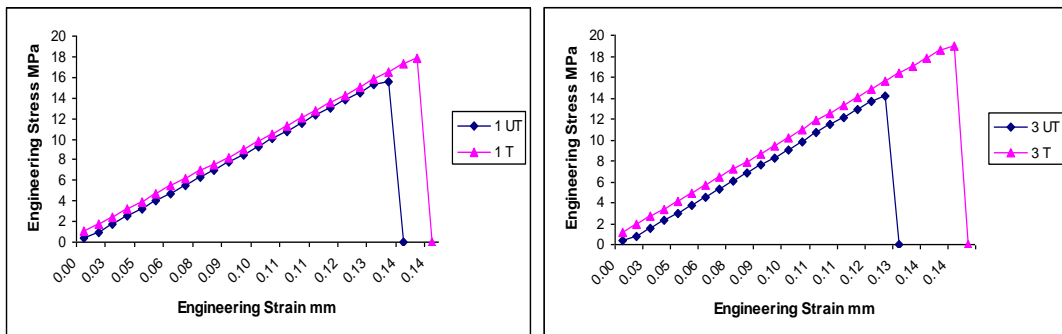


FIGURE 2 Load Vs Displacement of raw and treated composites



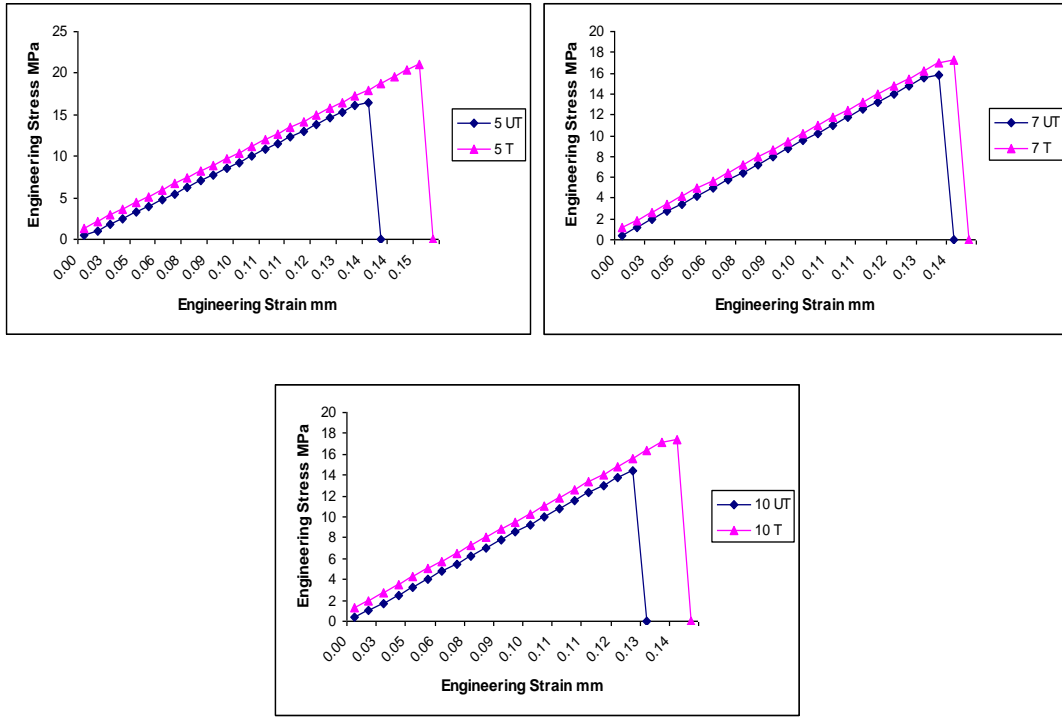
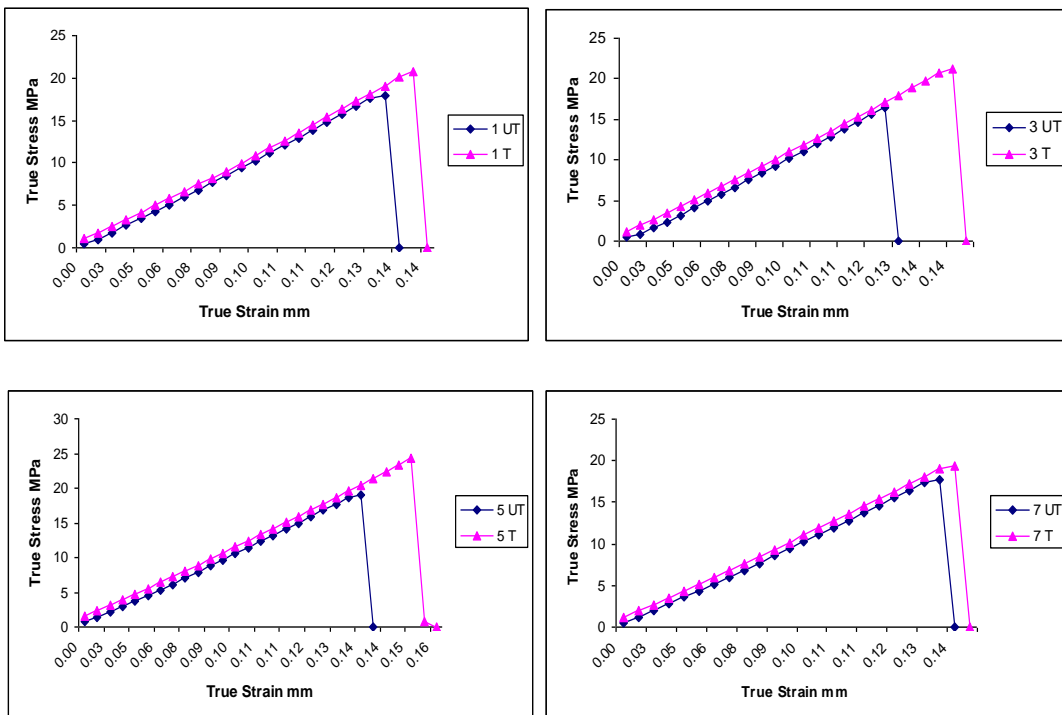


FIGURE 3 Engineering stress Vs Engineering strain of raw and treated composites



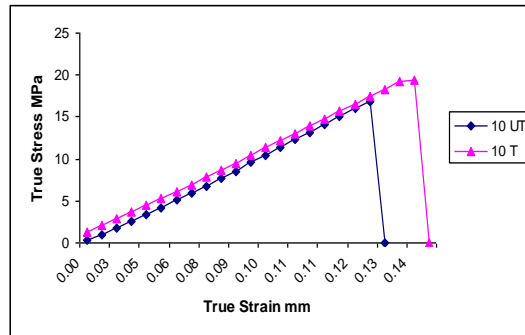


FIGURE 4 True stress Vs True strain of raw and treated composites

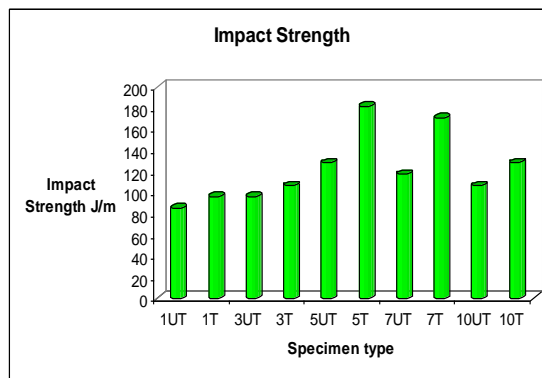


FIGURE 5 Impact strength of raw and treated specimens.

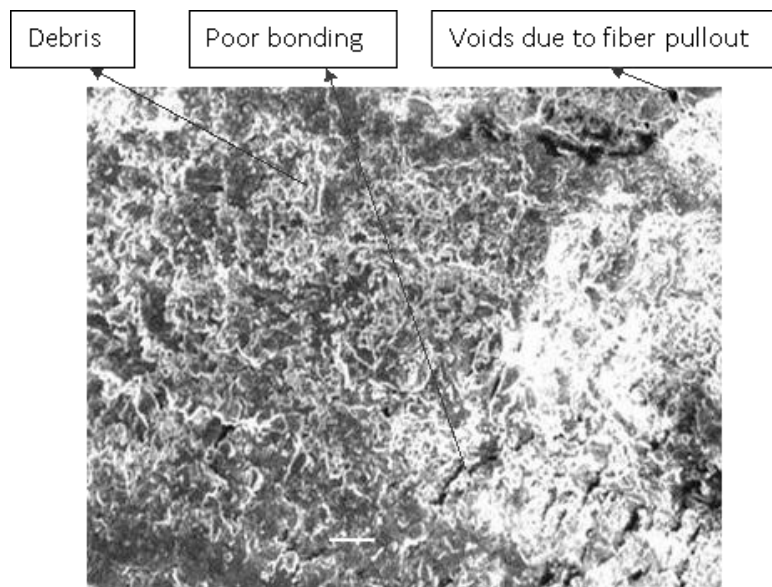


FIGURE 6 a SEM image of 1mm raw fiber reinforced tensile fractured specimen

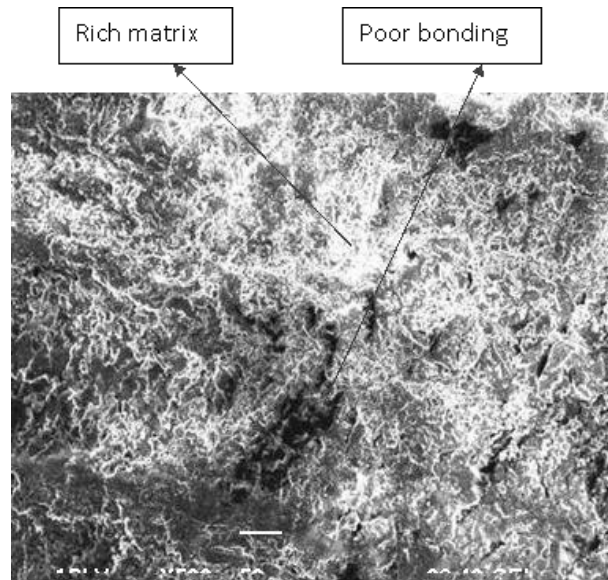


FIGURE 6 b SEM image of 1mm alkali treated fiber reinforced tensile fractured specimen

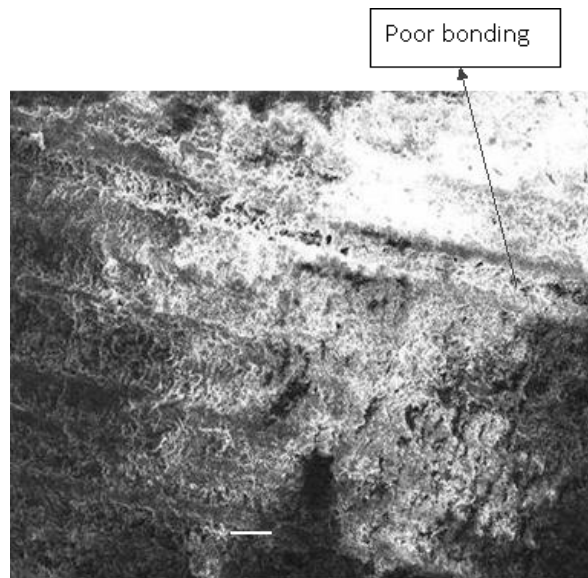


FIGURE 6 c SEM image of 5mm raw fiber reinforced tensile fractured specimens

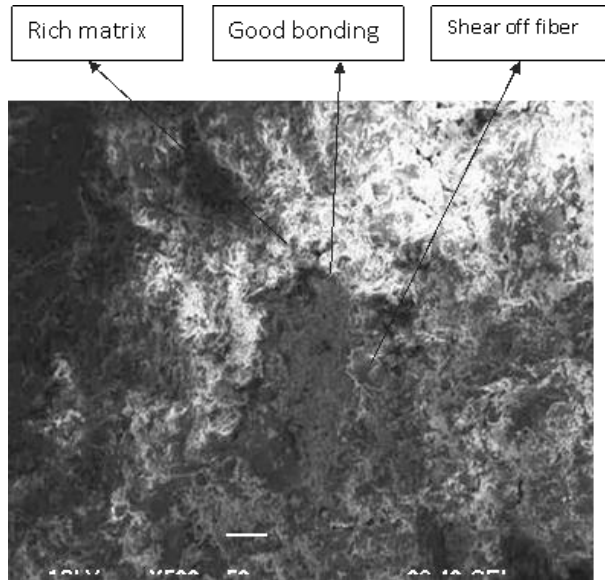


FIGURE 6 d SEM image of 5mm alkali treated fiber reinforced tensile fractured specimens

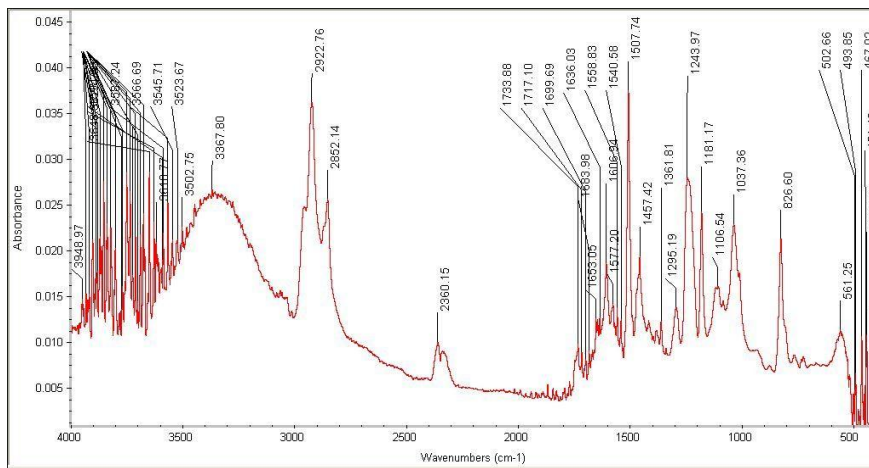


FIGURE 7 a FTIR Spectra of 1UT specimen

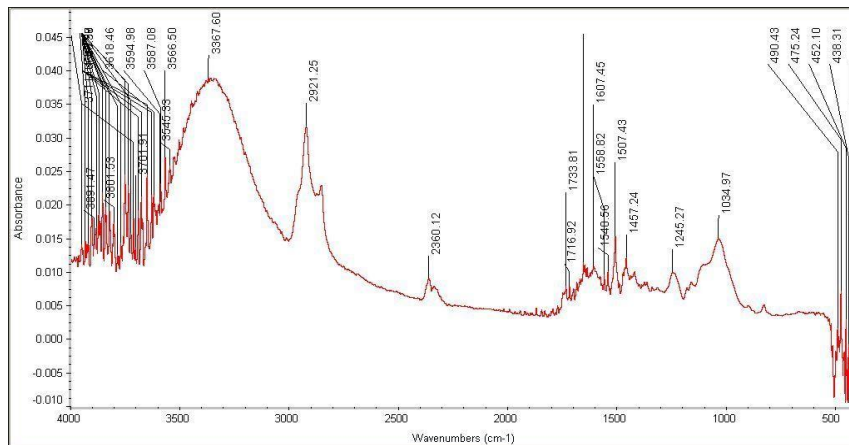


FIGURE 7 b FTIR Spectra of 1T specimen

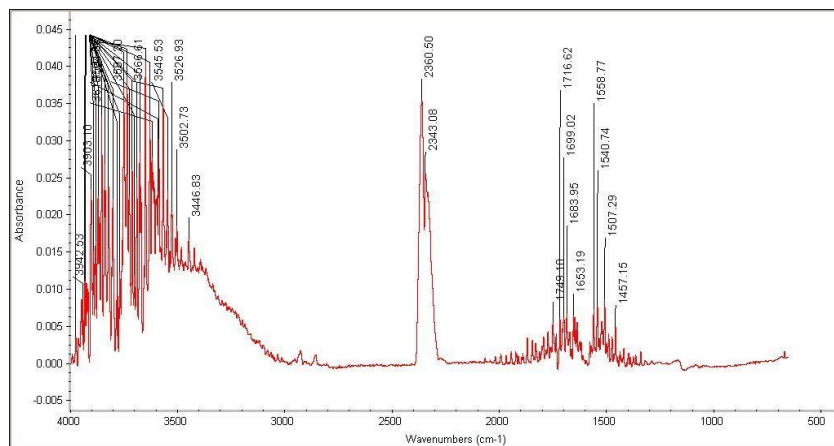


FIGURE. 7 c FTIR Spectra of 5UT specimen

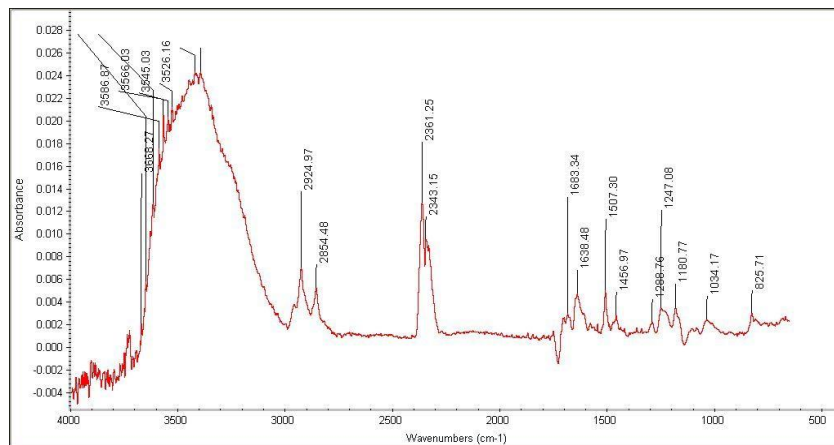


FIGURE 7 d FTIR Spectra of 5T specimen

4. Conclusions

The influence of fiber length on mechanical properties was investigated and the following conclusions were arrived at.

- Out of the five different fiber lengths chosen, 5 mm length treated fibers improved the tensile and impact properties of the composites more than the others. The improvement of tensile and impact properties for treated fibers reinforcement when compared to the raw fiber reinforcement was estimated from 10% to 30%.
- The fibers below the critical fiber lengths are considered as fillers or particles and hence their reinforcement yielded low tensile and impact properties to the composites. It is also interesting to note that for the fibers beyond 5 mm length, the impact and tensile properties of the composites were found to gradually decrease.
- The FTIR analysis evidenced the removal of hemicellulose from the fiber surface which leads to better fiber matrix interlocking and presence of strong hydrogen bond substantiated for better mechanical properties.
- The SEM analysis revealed that the fiber matrix adhesion and bonding were improved due to alkali treatment. The SEM results proved that the 5mm length alkali treated fibers provided better tensile and impact properties to the composites.
- Based on the experimentation, this paper suggests the use of 5mm alkali treated Borassus fruit fibers as reinforcements in light weight, high strength structural applications.

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