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RESEARCH

Development of Entada Mannii Fiber Polypropylene Matrix Composites for Light Weight Applications

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A B S T R A C T

This study investigates the use of Entada mannii fiber as potential reinforcement of thermoplastic composites suitable for light weight applications. Composites of 5 wt.%, 10 wt.% and 15 wt.% were produced by compression moulding with 5 wt.% Maleic anhydride polypropylene (MAAP) as compatibilizers. Tensile properties, impact strength and hardness properties of the composites were evaluated. Thermogravimetric analysis (TGA), X-ray diffractograms (XRD) and Fourier transformed infrared spectroscopy (FTIR) of treated and untreated fibers were evaluated while the fractographic analysis of surface morphology of the composites was performed using Scanning electron microscopy. The result revealed that reinforcing thermoplastic with 15 %.wt treated Entada mannii fiber revealed a greater improvement in tensile strength and Young's modulus by 58 % and 61 % respectively relative to pure PP and the hardness properties of the composite also increased by 56 % as compared with pure PP . This improvement is noticeable for the 15 wt.% treated fiber reinforced composites and could be attributed to good interfacial bonding between the fiber and the matrix. However impact strength of treated fiber composite revealed an improvement with 10 wt.% treated fiber composites by 48 % relative to Pure PP. Fracture surface images of treated fiber reinforced composites revealed less fiber pullout while the TGA showed the treated fiber degrades at higher temperature as compared with untreated fiber. Thus, the cellulose percentage crystallinity index of the treated fiber increases from 47.9 % to 57 % as a result of the influence of alkaline treatment.

1. INTRODUCTION

The use of natural fiber in reinforcement of polymeric composites offered a great advantage over the counterpart synthetic fiber. Natural fibers such as bamboo, coir, jute, flax, sun hemp, ramie, kenaf, rice husk, sugar cane, pineapple among others have been investigated with

encouraging results [1- 3]. They have properties such as low cost, low density, readily available with good dimensional stability [1,4,5]. They are used in various engineering applications such as constructions and automobile industries. Natural fibers are flexible during composites production process such as extrusion and injection moulding [6,7]. These made them less fracture and also maintain high aspect ratio [7]. While fiber reinforced composites have already proven their worth as weight-saving materials, the current challenge is to improved their mechanical, physical properties and make them cost effective [8]. Although natural fiber reinforced composites depends on the properties of the fiber constituents as well as the region surrounding the fiber, known as the interphase [9]. To a very reasonable extent, it is important to understand these relationships between the fiber- matrix interactions and improve the compatibility of the hydrophobic fiber and hydrophilic matrix using chemical treatment as reported in literatures [6,9,10]. The extent of this improvement has been observed to be marginal for some fibers and significant for others as the fiber response to chemical treatment is largely dependent on the fiber constituents and the chemical used as reported by researchers [11].

Beckermann and Pickering [7] worked on the Engineering and evaluation of hemp fiber reinforced polypropylene composites: fiber treatment and modification. Hemp fiber surface was improved using alkaline treatment suitable for composites reinforcement. Improvement in tensile strength Young's modulus, crystallinity index and thermal stability of the composites was observed. Balogun et al. [12] analysed the structural characteristics thermal degradation behaviour and tensile properties of hand extracted *Entada mannii* fiber and observed that, tensile strength and crystallinity index of the fiber increased after alkaline treatment. This however enhance the fiber- matrix interfacial adhesion.

Asumani et al. [13] investigated the effects of alkali-saline treatment on tensile properties and flexural properties of short fiber non-woven kenaf reinforced polypropylene composites. Three- aminopropyltriethoxysilane treatment improved the kenaf fiber and also improved tensile strength of the short fibre non-woven kenaf composites. This enhanced the fiber-matrix interfacial adhesion. The results obtained is better than those obtained from alkali or silane treatment alone.

Thus in attempt to have economic growth, the untapped area of converting agro waste to wealth which are readily available in abundant at a cheap cost, and provide alternative to expensive synthetic will continuous to be investigated [14].

Therefore the aim of this paper is to study, the thermal, physical and mechanical properties of *Entada mannii* fiber as potential reinforcement of thermoplastic composites suitable for light weight panels in automotive applications. Balogun et al. [15] reported that *Entada mannii* belongs to the family (*Oliv) Tisser.* Leguminous mermosaesae, liana plant. The plant is about 5 to 10m high semi-climber which grows in the tropical forest of Nigeria, Gabon and Madagascar. The plant stems have considerable strength and stiffness with extreme variations in mechanical properties during development from young to adult growth [12,14]. They were traditionally used many decades past in most native Nigerian communes to make ropes due to their high stiffness [12,15]. Presently, they find very limited use and have not yet been well investigated as reinforcement in PMCs. Availability, eco-friendliness, renewability and high specific strength and stiffness are some of the attractions of *Entada mannii* bast fibers, which has motivated our quest to assess its viability as reinforcement in polymer matrix composites (PMC).

2. MATERIALS AND METHOD

21. Materials

Entada mannii fiber of density 1.35 g/cm^3 was obtained from Ondo State, Nigeria; Polypropylene was supplied by Safron in South Africa with Melt flow rate, 230 ^{o}C/2.16 kg and density of 0.903 g/cm^3. Teflon sheet was used as the releasing agent; while 5 % Maleic anhydride polypropylene (MAAP) serves as the coupling agent to improve the fiber-matrix interfacial bonding.

2.2 Methods

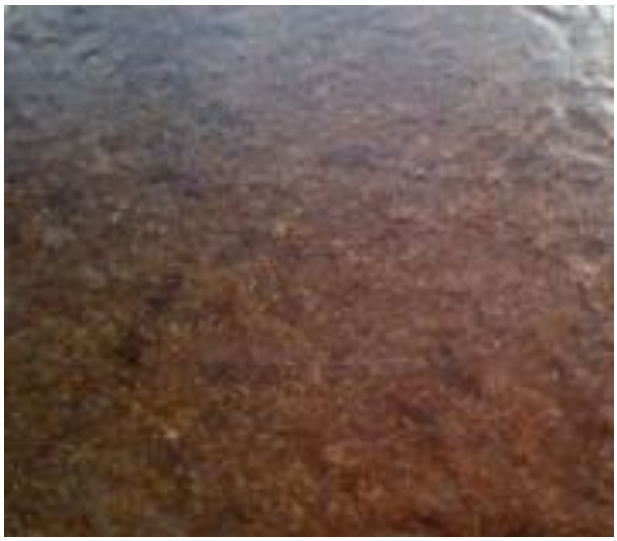
2.2.1 Fiber surface treatment

15 g of *Entada mannii* fiber was treated with 0.1 M KOH solution (500 ml) in a shaker water bath at 50 °C for 4 h. The insoluble residue was delignified at pH 3, and finally, distilled water was used to wash the fiber in order to remove mineral traces and dried in oven at 65 °C for 2 days.

2.2.2 Compounding of the composites

The *Entada mannii* fiber both treated and untreated of 5, 10, 15 wt.% were mixed with polypropylene matrix which served as the matrix. The mixture were compounded with 5 % Maleic anhydride polypropylene (MAPP). The blending was made using a twin-screw extruder with a rotor speed of 60 rpm and the barrel temperature was in the range of 130-190 °C. The extrudate was removed from the mixing chambers, cooled and granulated in an industrial granulator into pellets dimensioning 3 to 5 mm and randomly oriented and distributed in a stainless steel mould. The composites were compounded for 10 minutes at a temperature of 190 °C under a constant pressure allowing thorough penetration and dispersion of the treated fiber and untreated fiber into the matrix. Afterwards, the mold was transferred to another compression moulding machine and cold-pressed at 100 MPa for 12 min. The composites sheets produced were approximately 150 mm by 150 mm by 3 mm in thickness for both untreated and treated fiber composites Fig. 1.

a)



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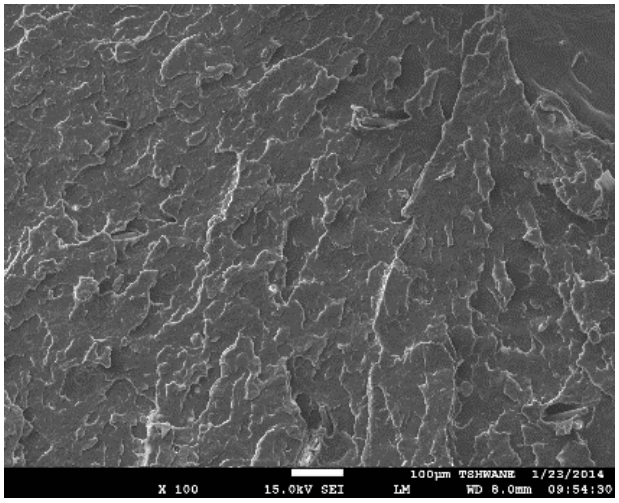


c)

Fig. 1. Production of the composites, a) Fiber separation, b) Composites production, c) Composites characterization.

2.2.3 X-ray diffraction analysis of fibers

Untreated and treated *Entada mannii* fiber were chopped into fine particles and compressed into disks using a cylindrical steel mould of (Ø = 15 mm) with an applied pressure of 20 MPa. A Phillips X'Pert diffreactometer fitted with a ceramic X-ray diffraction tube was used to determine the effect of alkaline treatment and untreated fiber crystallinity. The diffracted intensity of Cu Kα radiation (wavelength of 0.1542 nm) was recorded between 5 ° and 40 ° (2θ angle range) at 40 kV and 40 mA.

2.2.4 Tensile strength

Tensile test were performed on the composites produced using a universal tensile testing

machine operated at a strain rate of 10 mm/min with 10 KN load cell. The sample preparation, testing procedure and determination of the tensile strength and tensile modulus were in accordance with ASTM D638 [16]. Six samples were tested to guarantee the reliability of the tensile test results obtained.

2.2.5 Impact Strength

The impact strength of the *Entada mannii* fiber composite was evaluated using an Izod impact test machine. The sample preparation and testing procedure were in accordance with ISO 180 standard [17]. All the composite specimen were notched and the test specimen supported by a cantilever beam. Hammer head of 7.5 J was released with impact velocity of 2 m/s to strike and break the notch specimens. Six specimens were tested at room temperature and the values were recorded.

2.2.6 Hardness test

The hardness test of the Entada mannii fiber reinforced composites was measure using a Brinell hardness tester according to ASTM E10.A load of 60kg was applied on the sample for 20 sec. using a 2 mm diameter ball at three different points and the average value was calculated and recorded.

2.2.7 Morphology analysis (Scanning Electron Microscope analysis)

The surface morphology and fracture morphology of composites after tensile test were examined using a JEOL JSM-7600F model scanning electron microscope. The sample were placed in vacuum chamber, air dried and coated with 100 A thick irradium in JEOL sputter ion coater at 15Kev.

3. RESULTS AND DISCUSSION

3.1 TGA analysis for both treated and untreated fibers

Figure 2 shows the thermogravimetry analysis of the treated and untreated *Entada mannii* fibers. Thermal decomposition of the fibers measured by weight loss and temperature was observed between 100-600 °C for both treated and untreated fibers. It was observed that changes in weight loss occurred due to thermal degradation of the treated fibers after alkaline treatment. A slight increase in temperature was also observed between 300-370 °C as a result of exothermic combustion. The untreated fiber degraded at lower temperature between 200-300 °C and became thermally unstable due to the presence of these fiber constituents lignin and hemicellulose [1,18]. Beckerman and Pickering, [7] reported that, untreated fiber degrades at lower temperatures due to the presence of thermally unstable fiber constituents such as hemicelluloses and pectins, whereas the alkali treated fiber is more thermally stable due to the removal of these constituents with temperatures increase. It is also reported that, thermal decomposition process mainly occurred on cellulose which in turn increase the overall degradation temperature of treated fiber [1,19].

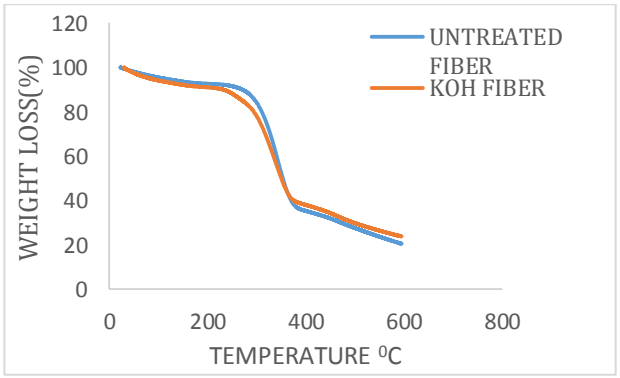


Fig. 2. Thermogravimetry analysis of the treated and untreated *Entada mannii* fiber.

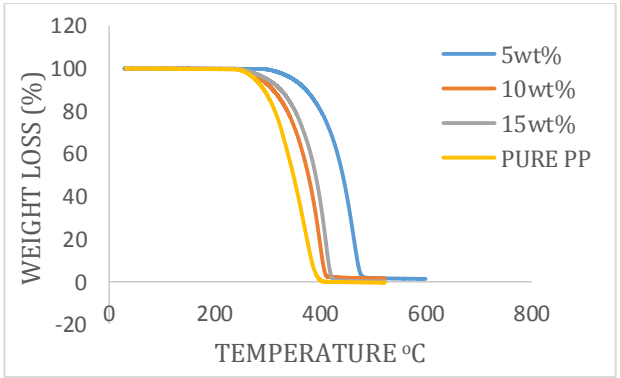


Fig. 3. Thermogravimetry analysis of the treated *Entada mannii* fiber composites.

Figure 3 revealed the thermogravimetric analysis for the *Entada mannii* fiber reinforced composites of 5 wt.%, 10 wt.%, 15 wt.% and pure PP. It was observed that the thermal degradation of the treated *Entada mannii* fiber reinforced composites decreases by reduction in

sample weights. The reduction in the composites weight is attributed to the evolution of the fiber constituents and the moisture contents from the treated fiber reinforced composites which was maintain at around 250 °C. This is remarkable for the 10 wt.% treated fiber reinforced composites. Treated and untreated composites tends to lose weight with the addition of fiber into the matrix relative to pure PP. Beckerman and Pickering [7] reported that reinforced with hemp fiber lose weight more than unreinforced composites due to higher frictional and shear forces experienced during compounding. There was a difference in the thermal degradation of the composites with the increase in temperature for all composites between 300 °C and 400 °C. This is because of the initial decomposition of the fiber constituents and thermal depolymerization traces of the hemicellulose and lignin from the fiber surface. At temperature between 400 °C to 600 °C, thermal decomposition stages was completed for all the composites and fiber degradation occupied was noticed at 350 °C and 400 °C when the composites had lost almost 95 % of the fiber constituents with increase in fiber loading. The weight loss of the reinforced composites revealed the degradation temperature of the composites with the respect to increase in fiber loading consistently higher than pure PP composites. At 15 wt.% composites fiber loading increase, indicated the heat resistance was effectively removed by the alkaline treatment.

3.2 Wide angle X-ray diffraction of the treated and untreated Entada mannii fiber

The X-ray diffractograms of the treated and untreated *Entada mannii* fiber are presented in Fig. 4. The results show the influence surface treatment on the *Entada mannii* untreated fiber between the regions of the crystalline and amorphous of the fiber as denoted by major crystalline peaks at around 2θ = 23.4 ° which represented the crystallographic plane (0 0 2). It is also observed that X-ray diffractograms (0 0 2) revealed the alkaline treated fiber peaks which is more intense than untreated fiber peaks as a results of chemical treatment and removal the fiber constituents (lignin, hemicellulose and pectins) from the fiber surface thus exposing the cellulose contents. It is also observed that the cellulose contents increases with the removal of fiber constituents and the peaks became narrow whereas the untreated fibers containing amorphous/ materials gave a broad peak bands. However, crystallinity index is useful only on a comparison basis as it is used to indicate the order of crystallinity of the crystalline regions [7,18].

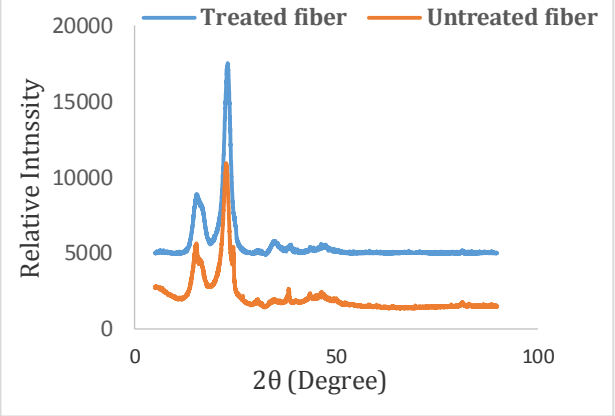


Fig.4. X-ray diffraction for KOH treated and untreated *Entada mannii* fiber.

The fiber crystallinity index (*Ic*) of the treated and untreated *Entada mannii* fiber was calculated using the formula (1) [7,19]:

$$I_c = \frac{I_{002} - I_{am}}{I_{002}} X\ 100 \qquad (1)$$

where I_{002} is the maximum intensity of diffraction of the (0 0 2) lattice peak at a 2θ angle between 23 ° and 24 ° and I_{am} is the intensity of diffraction of the amorphous materials which is taken at a 2θ between 15.50 ° and 16 ° where the intensity is at minimum [7].

Table 1. Crystallinity index values for both treated and untreated fibers.

% of Fiber	I_{am} (2θ =15.5)	I_{002} (2θ =23.1)	Crystallinity index %
KOH Treated	3337	7900	57.8
Untreated fiber	379	7280	47.9

Table 1 show the crystallinity index of the *Entada mannii* fiber for both treated and untreated fiber. Cellulose crystallinity of the fiber increases as a result of the influence of alkaline treatment and thereby increases the percentage of the crystallinity index for the untreated fiber from 47.9 % to 57 % treated fiber. During the chemical reaction, fibers constituents were removed from the fiber surface and hence increases the cellulose contents and the crystallinity index of the fiber. Similar observation was reported by Emanuel et

al. [20] and Calado et al. [21] that alkaline treatment removed the lignin and hemicellulose from the fiber surface and hence increases the crystallinity fraction of the cellulose which loses part of the amorphous with respect to the peak characteristic of the cellulose system. Decrease in tensile strength of the untreated fibers may be due to the cellulose degradation and removal of the excessive amount of the lignin and hemicellulose (amorphous) which is responsible for the binding of the microfibrils together in fibers [7,22].

3.3 Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra of treated and untreated *Entada mannii* fiber is presented in Fig. 5.It is evident that alkaline treatment of the fiber removes the lignin and hemicellulose covering the fiber surface and increases the cellulose contents. The spectra region of the untreated fiber and the alkaline treated fiber revealed that changes occurred between 3120 cm^{-1} and 3435 cm^{-1} which is attributed to O-H stretching of hydrogen bond network. This became less intense with a partial dissolution of the lignin and hemicellulose in the KOH treated fiber with breaking of the hydrogen bond between the cellulose and hemicellulose compounds. Similar work was reported by Arrakhiz et al. [22]. Brigida et al. [23] that FTIR analyses also reveal a reduction in hemicellulose content in the treated fibers. The absorbance peak characteristics at 2849 cm^{-1} shows the C-H stretching vibration of methyl and methylene groups in the cellulose and hemicellulose molecules.

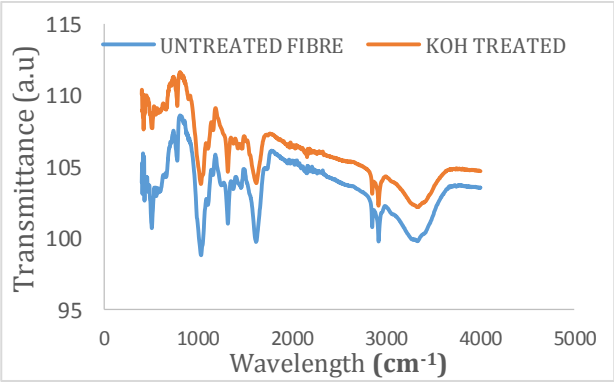


Fig. 5. FTIR spectra of treated and untreated *Entada mannii* fiber.

At 1629 cm^{-1} peak shows the removal of hemicellulose by alkaline treatment molecules which may be attributed to C=O stretching vibrations. The asymmetric C-H_3 and C-H symmetric represented the absorbance band at 1471 cm^{-1} and 1318 cm^{-1} which revealed the deformation of the lignin molecules due to alkaline treatment. The large peak observed for the untreated fiber can be associated to the prescence of lignin and confirmed by the peaks [23,24]. The treated alkaline fiber shows the peaks at 1400 cm^{-1} and 1300 cm^{-1} revealed C-O stretching vibrations for the untreated fiber disappears after treatment. The disappearance shows that the fiber constituents such lignin and hemicellulose are removed during the alkaline treatment. As reported by Hongyu et al. [25] the disappearance of the peak after alkalization indicated that the removal of hemicellulose than the lignin. However, it is also evident that C-O stretching vibrations at the peak of 1036 cm^{-1} revealed that acetyl groups is reduced with the removal of the hemicellulose and waxes by the alkaline treatment which disappears with the alkaline treatment. Broadband is related to the vibration C-O of esters, ethers and phenol groups attributed mainly to a presence of waxes in the epidermal tissue [23], and the disappearance of this band in the treated fibers results from the removal of waxes.

3.4 Tensile strength

From Fig. 6, it is evident that all composites showed an increase in tensile strength with the addition of treated, untreated *Entada mannii* fiber and 5% MAPP in the matrix.

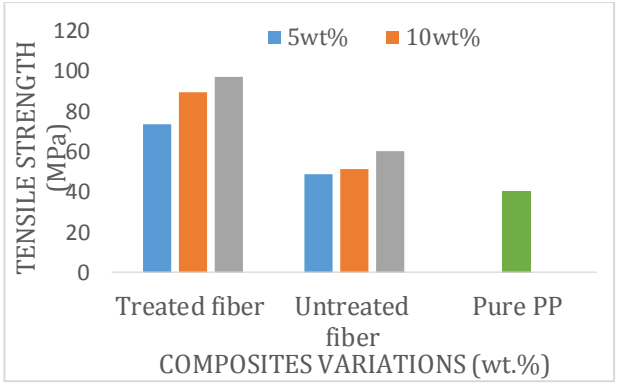


Fig. 6. Tensile strength of the *Entada mannii* fiber reinforced composites.

Addition of MAPP compatibilizers improved the interfacial adhesion between the fiber and the matrix. This improvement is largely due to the chemical bonding of the OH groups on the fiber surface which enhanced the molecular chain

adhering of the fiber to the matrix [7].The presence of the OH group available on the treated fiber groups coupled with 5 % MAPP enhanced the fiber to matrix interfacial adhesion is attributed to improve surface area and removal of fiber constituents such as lignin and hemicellulose from fiber surface. From Fig. 8, it is also evident that 5 % MAPP greatly improved the adhesion between the fiber and the matrix with less fiber pull out and debonding on the fracture surface morphology of the alkaline treated composites than untreated composites as compared with results obtained in Fig. 11. Tensile strength increases with increase in fiber loading. At 15 wt.%, tensile strength was found to increase by 37 % and 58 % respectively relative to untreated composites and pure PP. This could be could be largely due to improvement of the fiber – matrix interfacial adhesion which increase the fiber flexibility to withstand stress when load was applied. On the other hand, the tensile strength of the untreated fiber decreases than treated fiber reinforced composites due to the prescence of fiber constituents such as lignin and hemicellulose promoting a poor fiber- matrix interfacial adhesion. Similar work was done by Balogun et al. [14] and reported that tensile properties of hand extracted untreated *Entada Mannii* fiber decreases due to prescence of fiber constituents. This poor interfacial adhesion between the untreated fiber reinforced composites and the matrix resulted in stress concentration and fracture the composite at lower value.

3.5 Young's Modulus

The results of the Young's modulus of the *Entada mannii* fiber reinforced composites are presented in Fig. 7.

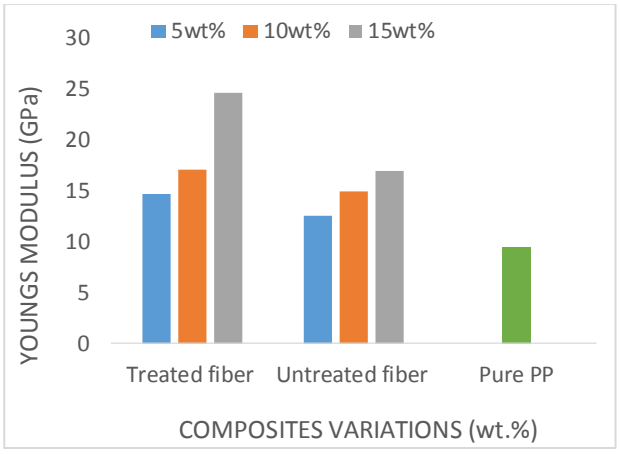


Fig.7. Young's modulus of the *Entada mannii* fiber reinforced composites.

On the average, its observed that the Young's modulus of the composites increases with increase in fiber loading which is remarkable for the 15 wt.% composites than pure PP composites. At 15 wt.% composites gave the optimum Young modulus of 31 % stiffer than untreated and 61 % than pure PP composites respectively. This is because of effective transfer of load between the matrix and the fiber which increases the fiber to matrix interfacial adhesion and the stiffness of the composites. Cho et al. [26] reported the improvement of the treated fiber–matrix interfacial adhesion was observed as related to the stiffness of composites.

Similar work was done by Asumani et al. [13] reported that an increase in modulus is associated with the better fiber-matrix bonding with sufficient load transfer between the fiber and the matrix. Consequently, a drop in Young's modulus of the 5 %.wt composite could be attributed to poor interfacial adhesion between the fiber and the matrix. This result of the Young's modulus could also be related with the results obtained from the tensile strength in Fig. 6.

3.6 Impact strength

The variations of the impact strength of *Entada mannii* fiber reinforced composites for treated, untreated and pure PP are presented in Fig. 8, it is evident that impact strength of the treated composites increases with increase in fiber loading.

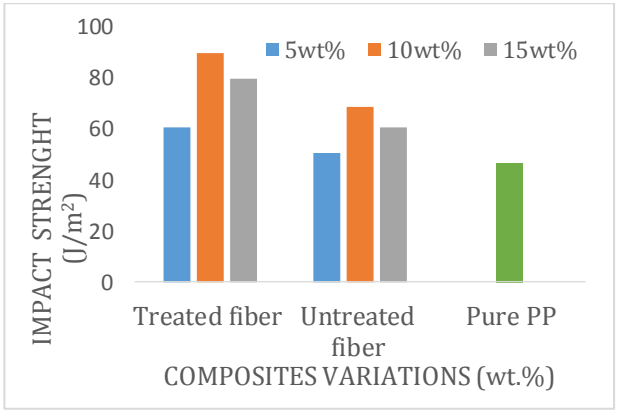


Fig. 8. Impact strength of the *Entada mannii* fiber reinforced composites.

This could be largely due to evenly distribution of the fiber in the matrix which enhanced better fiber – matrix interfacial adhesion. This improvement is noticeable for the 10 wt.% and

on the average, 10 wt.% fiber exhibited the higher impact strength of all the composite by 23 % relative to the untreated composites and 48 % pure PP respectively. Similar work was reported by Venkateshwaran et al. [6] on the average, the impact strength of the treated composites improved significantly relative to untreated composites. A slight drop in impact strength of the 15 wt.% composites as compared with 10 wt.% was attributed to increase in fiber density and poor interfacial adhesion between the fiber and the matrix. Apparently, as the fiber loading increases, agglomeration of the fiber occurred and hence created a stress concentration which imping the crack propagation and fracture the composites at lower value [30].

3.7 Hardness properties of the composites

Figure 9 revealed the hardness values of *Entada mannii* fiber reinforced composites. It is observed that the hardness value increased as the fiber loading increases.

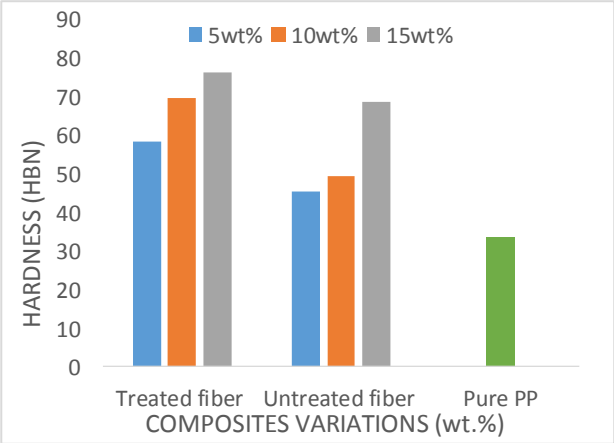
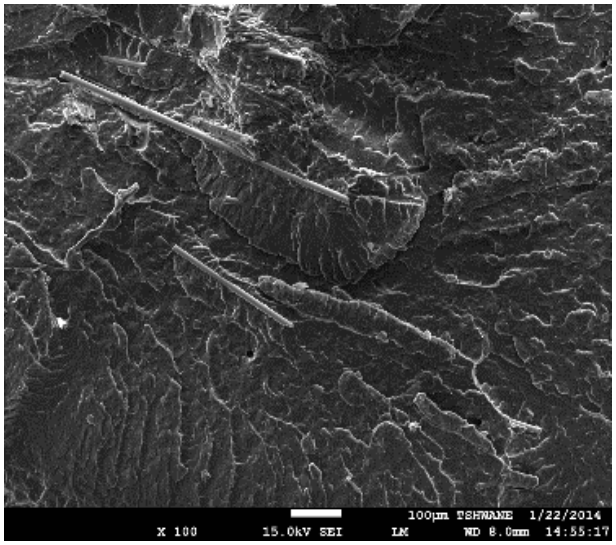


Fig. 9. Hardness properties of the *Entada mannii* fiber reinforced composites

The increase in the hardness could be attributed to the increase in the hard surface of the composites with even dispersion of fiber with the matrix. This is remarkable for the 15 wt.% treated fiber loading with an increase of 10 % and 56 % respectively as compared with untreated composites and pure PP. Prescence of the evenly distributed fiber in the matrix trends to slow down the nucleation of the crack in the fiber- matrix interphase and hence increases hardness of the composites. Subramonian et al. [27] reported that, hardness of the fiber reinforced composites increases due to resistance of the composites to deformation increases .This composites surface gave a better resistance to plastic deformation in the transverse direction of the fiber. The increase in hardness is a result of increase in the hard and brittle phase of fiber [28]. The hardness result obtained is similar to what we obtained from the tensile strength in Fig. 6.

3.8 SEM micrograph

Figure 10 show the SEM micrographs of the of untreated *Entada mannii* fiber reinforced composites. It is evident that due to the presence of the fiber constituents such as lignin and hemicellulose deposit on the fiber surface contributed to the poor fiber – matrix interfacial adhesion and which confirmed the failure of the untreated composites.



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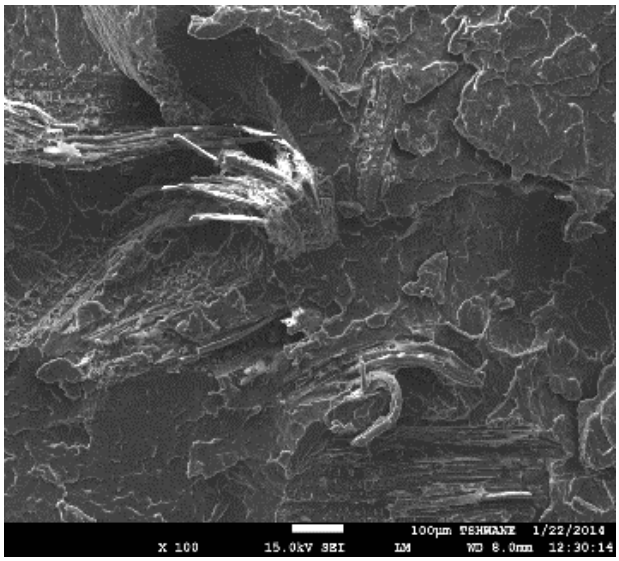

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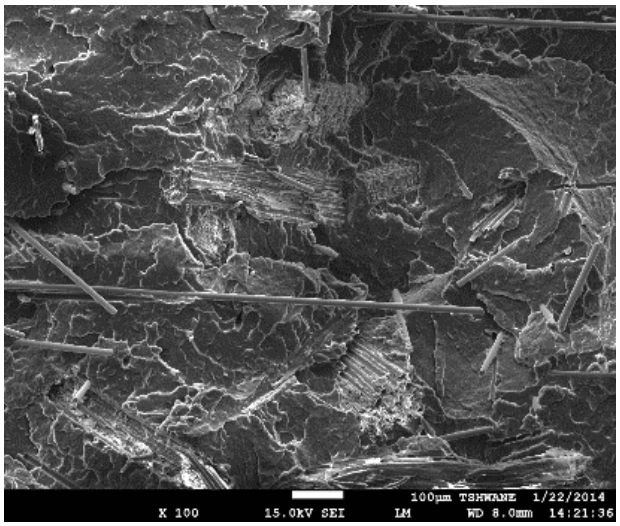
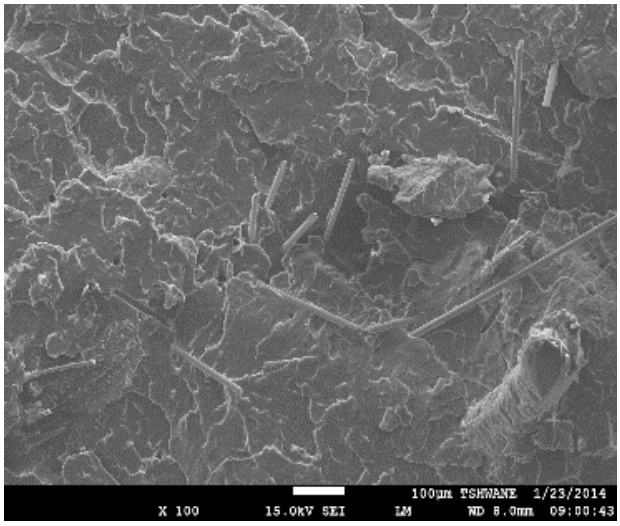


Fig. 10. SEM images of: a) 5 wt.% Matrix yielding, b) 10 wt.% Fiber debonding, c) Fiber pullout at 15 wt.% of untreated fiber reinforced composites.

Untreated fiber reinforced is characterized with fiber pullouts, debonding and matrix yielding after fracture. This failure revealed an ineffective load transfer between matrix and the fiber [13]. This behaviour is confirmed by the results obtained in the tensile strength of untreated composites are lower than treated composites. At 15 wt.% untreated composites, poor agglomeration of the fiber in the matrix is observed with increase in fiber density and this lowers the tensile strength of the untreated composites than treated composites.

Figure 11 shows the SEM micrographs of the treated fiber reinforced composites. Less fiber pullout was observed for the composites. This confirmed a good interfacial adhesion between the fiber and the matrix.



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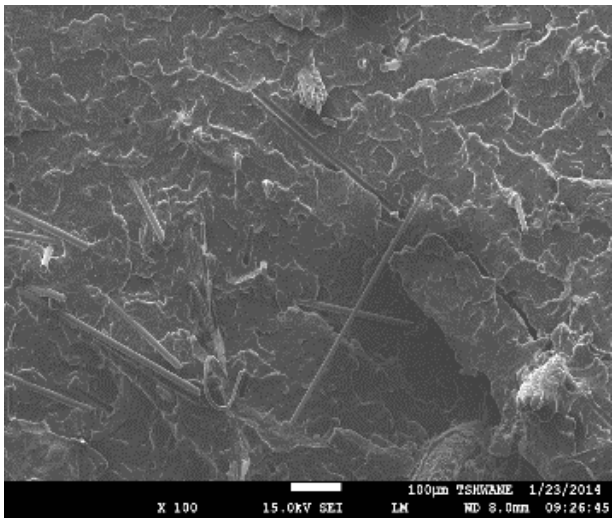

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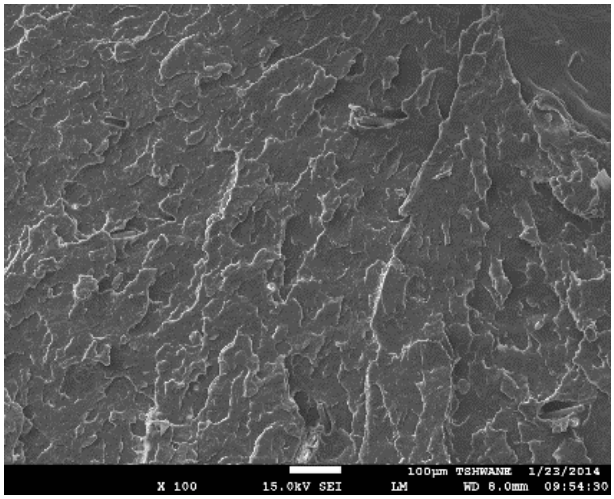

c)

Fig. 11.SEM images of: a) Fiber debonding at 5 wt.%, b) Fiber pullout at 10 wt.%, c) Less fiber pullout 15 wt.% of treated fiber reinforced composites.

The results conforms to the tensile strength result obtain from Fig. 5 causing an increase in the tensile strength of the composites. At 15 wt.% fiber loading, revealed the fiber is bonded to the matrix which also confirmed improvements of mechanical properties relative to a good interfacial adhesion between fiber and the matrix. Similar work was reported by Srinivasa et al. [29] on the fiber – matrix adhesion was improved by incorporating Areca fiber into the matrix.

4. CONCLUSION

This study investigates development of polypropylene matrix composites materials using *Entada mannii* fiber as a potential reinforcement of thermoplastic composites. Reinforcing

polypropylene with *Entada mannii* fiber revealed a greater improvement in the mechanical properties of the composites. Moreover, tensile strength and Young's modulus of the treated fibers reinforced composites was increased by 58 % and 61 % respectively relative to other composites. The impact strength of the treated composites was also improved by 48 % and the hardness of the composites by 56 % relative to other composites. SEM images show that less fiber pullout was observed for the treated fiber reinforced composites indicating a good interfacial adhesion between the fiber and matrix. Thermal stability of the composites revealed untreated fiber was thermally unstable than treated fiber reinforced composites.

Consequently, fiber pullout, debonding and matrix yielding were observed for the untreated fiber reinforced composites. Thermal degradations of the treated fiber were higher than untreated fiber due to removal of the fiber constituents such as lignin and hemicellulose by KOH treatment. However, the XRD show that treated fiber peaks were more intensed than untreated fiber due to alkaline treatment. FTIR revealed the nature of bonds and organic compounds that are present in the fibers. The work has established the potential use of *Entada mannii* fiber in reinforcement of thermoplastic composites suitable for automobile light weights applications.

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