

Production of Reinforced Polyester Composite from Okra Fibre and Sawdust

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ABSTRACT: This research presents properties of okra and sawdust reinforced polyester composite. The compatibility of the simple woven okra and sawdust with polyester was enhanced with stearic acid treatment. FTIR analysis confirmed decrease in hydrophilicity of the fibre and dust. Six composite samples; pure polyester, sawdust reinforced polyester composite, okra reinforced polyester composite, 10% sawdust in okra fibre reinforced composite, 20% sawdust in okra fibre reinforced composite and 30% sawdust in okra fibre reinforced composites were fabricated and characterized. The morphological analysis showed that the homogeneity of polyester in the samples reduces with increase in sawdust filler (10-30 wt%). Water absorption was highest (1.6%) in 30% sawdust in okra. The densities of all the composites were between 3.5 – 4.5 kg/m³. The sawdust reinforced composite recorded low impact energy of 0.25 J while the woven okra fibre reinforced polyester recorded the highest impact energy of 9.9 J. Hardness property reduced as the biomass content increased. Unreinforced polyester recorded the highest average elongation of 25% (1400 µm) and reduced elongation as filler increased. The storage modulus was highest for unreinforced composite at 40 °C but as the temperature reached 81 °C the storage modulus of unreinforced polyester dropped lower than the sawdust composite. The damping factor (1.41) was highest for 20 wt% sawdust/okra polyester composite. This research concludes that sawdust and okra are suitable for lightweight and energy damping materials in automobile applications.

KEYWORDS: Weight loss, activation parameters, adsorption, optimization, stainless steel, castor leaf.

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I. INTRODUCTION

Natural/green fibre reinforced composites have been receiving tremendous attention in recent times. This is especially in areas of material science, engineering researches and applications. Natural fibres such as wool, wood, okra, sisal, jute etc. are light weight, low cost, abundant in nature and renewable when compared to synthetic materials (Gurunathan *et al.*, 2015; Horta *et al.*, 2017). The biodegradability nature of these materials favours sustainable environment and is responsible for the growing interests. (Ahmad *et al.*, 2018; Thyavihalli *et al.*, 2019). Their properties have attracted scientists and engineers to consider them for automobile, construction, upholstery applications (Mohammed *et al.*, 2015; Li *et al.*, 2020; Merzoug *et al.*, 2020). Natural fibres have drawbacks as, they are not readily compatible with synthetic matrices (Gurunathan *et al.*, 2015; Hamidon *et al.*, 2019). Therefore, the need for fibre modification to improve the compatibility (Asim *et al.*, 2020).

Okra which is a vegetable crop that belongs to the *genus Abelmoschus*, family Malvaceae has two main species: *Abelmoschus esculentus* (L.) Moench and *Abelmoschus caillei* (A. Chev.) Stevels (Letebrhan *et al.*, 2020). *Abelmoschus esculentus* (L.) Moench is mostly cultivated in western Nigeria (Ijoyah and Dzer, 2012) where the stem is rarely consumed.

This can be extracted as a natural fibre for reinforced composite application. Effective utilization of okra is necessary to curb wastage being experienced during peak season (Ngbede *et al.*, 2017). Wood dust or sawdust on the other hand is obtained from the wood industry as waste. Wood like mahogany (*Azalia africana*) is significantly utilized in woodwork industry in Nigeria causing a substantial sawdust build-up (Okonkwo *et al.*, 2016). These build-ups when improperly disposed find their ways in open areas, drainage systems and water bodies thereby causing floods, land, water and air pollution. Nigeria's 1.8 million tons annual sawdust production if not properly utilized will have negative impact on the terrestrial and aquatic ecosystems (Owoyemi *et al.*, 2016). Sawdust utilization in fibre reinforcement reduce environment pollution caused by incineration, increased local economic growth and create employment (Oluoti *et al.*, 2014; Ogunwusi *et al.*, 2014)

Morphological properties of fibres greatly influence the performance of composites (Nayak *et al.*, 2017). Natural fibres are hydrophilic while synthetic matrices are hydrophobic. Water repellency, lamination adhesiveness, wettability and biocompatible processes are greatly affected by their surface morphology of the reinforcing fibres (Marcovich *et al.*, 1996; Popelka *et al.*, 2020). The hydrophilicity can however be modified by physical or

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chemical processes. These modifications will promote interfacial adhesion between the modified fibre and the matrix (Wassamon *et al.*, 2013). A good modification enhances stress transfer between the matrix and the fibre thereby improving the overall physical and/or mechanical properties (Valadez-Gonzalez *et al.*, 1999; Thitithanasarn *et al.*, 2012). The mechanical properties reflect the motion state inside the composites and are important to the efficient use of the fibre-reinforced polymer composites (Zhang *et al.*, 2017). Hybridized composites largely depend on fibre length, fibre strength and orientation for high mechanical strength, noise absorption, abrasion etc (Misganew *et al.*, 2020; Unterweger *et al.*, 2014). These properties are some of the capabilities that natural fibre reinforced composites are offering the current growing automobile and building industries (Ahmad *et al.*, 2014).

This work focuses on the morphology, physical and mechanical properties studies of natural fibre (of okra and sawdust) reinforced polyester composite for engineering applications.

II. MATERIALS AND METHODS

A. Materials

Okra fibre and sawdust were sourced from Dogarawa Garden and Sabon gari market, Samara Zaria in Nigeria, respectively. Stearic acid (Aldrich reagent grade), methylethylketone peroxide catalyst (Aldrich analytical standard), cobalt nephthenate accelerator (Aldrich laboratory chemical), and general purpose polyester resin (standard grade) were bought from Haddis International, Samaru Zaria.

B. Methods

1) Extraction of okra fibre and sawdust particle

The stem of the okra plant was soaked in water for 7 days to soften for fibre extraction. The water absorbed in the stem swells in inner cells and weakens the outer layers. The fibre was then extracted manually by beating the softened stems and then scraping the pulp away with a knife as described by Hulle *et al.*, (2015). The sawdust was grounded using Kere Soesterberg milling machine then sieved with a sieve of mesh size 125µm. The images of the okra fibre and sawdust are shown in Figure 1. The sample was thereafter stored in a desiccator before use.



Figure 1: (a) Extracted okra fibre; (b) Grounded sawdust.

2) Treatment of okra fiber and sawdust

40 g of the dried castor leave powder was transferred into 10 g of stearic was dissolved in 13 mls of ethanol, then added to a litres distilled water as described by La Mantia and Morreale (2011). The extracted Okra fibre of mass 300 g was then soaked into the solution and stirred for 24 hours with the aid of Hangzhou HC-100 thermo shaker. The fibre was then washed thoroughly with water and dried. The same procedure was repeated for sawdust treatment.

3) Weaving the treated okra fiber

The okra fibre was woven using the plain weave method as shown in Figure 2. The woven fibre was of 6 x 6 inches dimension based on the size of the mold.



Figure 2: (a) Weaving machine (b) Plain woven Okra fibre.

4) Preparation of the composite

The mould was thoroughly cleaned and waxed to aid removal of the composite using Isa *et al.* (2016) method. The woven fibre (6x6 inch) was placed in the cavity of the mould. For a basis of 100 g sample, 100 g of polyester resin was measured into 250 ml beaker, 2 g of catalyst (methyl ethyl ketone peroxide) was added and stirred for 1 minute. 2g of cobalt naphthenate was then added and stirred. The mixture was transferred to the mold containing the woven okra fibre and allowed to cure for 60 minutes, after which the cured composite was carefully removed and dried (60 °C) in an oven. Six samples described in Table 1 and shown in Figure 3 were produced and taken for analysis.

Table 1: Constituents and composition of the fabricated composite samples.

Sample	Okra fibre (g)	Sawdust (g)	Polyester resin (g)
(a) Pure Polyester resin	-	-	200
(b) Sawdust reinforced polyester	-	20	180
(c) Okra mat reinforced polyester	40	-	160
(d) 10% Sawdust + Okra mat	34.4	3.82	161.78
(e) 20% Sawdust + Okra mat	40	10	150
(f) 30% Sawdust + Okra mat	44	20	136

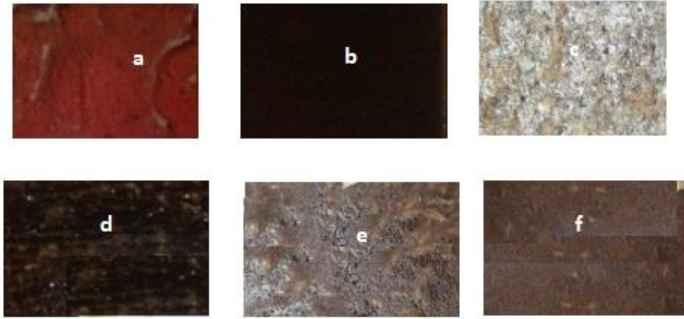


Figure 3: Composites formed from okra and sawdust reinforced in polyester resin: (a) pure polyester resin; (b) Sawdust reinforced polyester; (c) Okra mat reinforced polyester; (d) 10% Sawdust + Okra mat; (e) 20% Sawdust + Okra mat; (f) 30% Sawdust + Okra mat

5) Tests and analysis

a) Spectroscopy and Morphological testing of treated fibres: Fourier transform infrared spectrometry using (FTIR-ART machine (Happ-Genzel, Agilent Technologies, USA) and Phenom TM Prox scanning electron microscopy (SEM) were employed in carrying out functional group analysis and morphology of the composites respectively.

b) Mechanical testing: The mechanical tests performed were the impact, hardness, water absorption and tensile strength tests.

c) Impact testing: Charpy impact test machine with serial number 412-07-15269C was used with a capacity of 15 J for composites following ASTM E23 standard method.

d) Hardness test: The Rockwell hardness test of machine type 6187.5B with serial number 053158 was used. The Scale F (Soft materials) with the 1/16-inch ball was used according to the ASTM D2240.

e) Water absorption test: The 24-hour procedure according to (ASTM D570) was used and the samples were weighed before and after immersion. Water absorption is calculated in percentage using Eq. 1, where w_0 is the initial weight and w is the final weight.

$$\text{Water absorption (\%)} = \frac{w - w_0}{w_0} \times 100 \quad (1)$$

f) Tensile test: The composite was shaped into a three-dumbbell shape according to ASTM D638. The Monsanto tensometer type 'w' with serial number 9875 was used. The samples were fixed between the teeth of the machine and pulling continuously until rupture occurred. The graph was used to determine the young modulus, percentage elongation, strain and maximum tensile strength.

g) Dynamic mechanical analysis: Each of the samples of the pure polyester and composites were cut into a rectangular shape of 40 mm x 10 mm x 5 mm. Netzsch 242E analyser was operated at 3-point bending mode, set at 10 K/min and 1 Hz frequency. The storage, loss modulus, $\tan \delta$ and elongation were however determined using the Netzsch Proteous software.

III. RESULTS AND DISCUSSION

A. Fourier Transform Infrared Spectroscopy of the Fabricated Okra-Sawdust Reinforced Composite

Figure 4(a-b) shows the FTIR of the (a) untreated and treated okra fibre and (b) untreated and treated sawdust. The absorption band observed in 3280.15 cm^{-1} in the okra fibre and 3300 cm^{-1} in sawdust are characteristic bands for the hydroxyl groups from complex vibrational stretching. The free inter and intra molecular bound hydroxyl groups are responsible for the hydrophilic nature of natural fibres. It also makes them incompatible with hydrophobic matrices such as polyester. After the stearic acid treatment, the intensity of the peaks was greatly reduced implying the removal of some bonded O-H group. This hydroxyl group removal is good for fibre matrix compatibility (Wassamon *et al.*, 2013). The peak 2922.2 cm^{-1} characterized C-H stretch in methyl and methylene group was not affected by the treatment.

The carbonyl C=O stretch ($1720-1740 \text{ cm}^{-1}$) as in unconjugated ketone, carbonyl and ester groups in the untreated fibre represents the non-cellulosic components (pectin, lignin and hemicelluloses) which reduced significantly in treated fibres. There was also reduction in absorbed water (OH) shown at peak ($1630-1650 \text{ cm}^{-1}$). A sharp drop in peak at ($1020-1050 \text{ cm}^{-1}$) corresponding to symmetric C-OH stretching of lignin was observed. The lignin reduction is vital in natural fibre-synthetic matrix compatibility. The wavenumber (1420 cm^{-1}) indicating aromatic skeleton vibrations combined with C-H in-plane

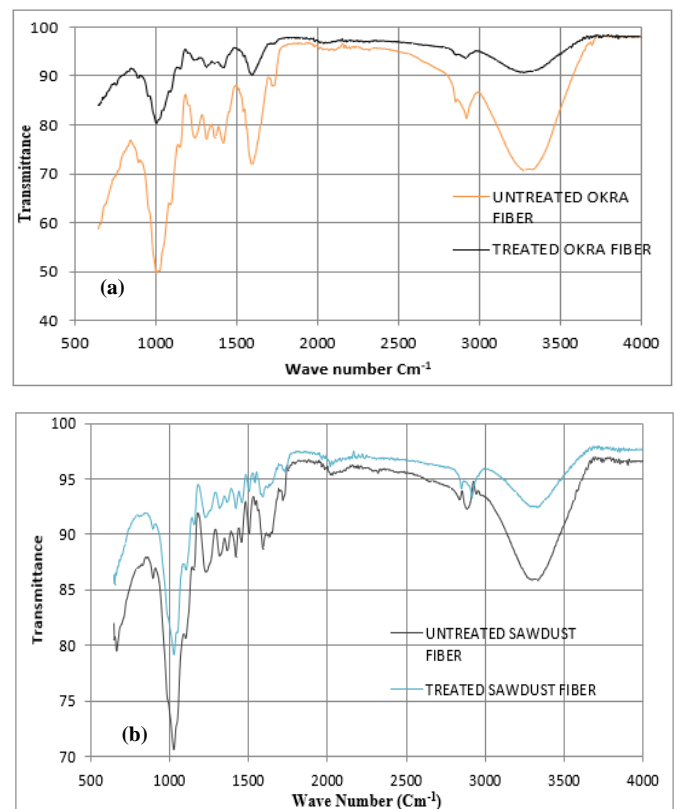


Figure 4: FTIR spectra of untreated and treated (a) okra and (b) sawdust.

deformations (CH_2 bending of cellulose) was still present in the treated fibre. The peak at 1319.5 cm^{-1} signifies the presence of C-O group of the aromatic ring in polysaccharides. At 1367.9 cm^{-1} , there is bending vibration of C-H of the aromatic ring in polysaccharides. The 1244.95 cm^{-1} band in the untreated okra fibre (Figure 4a) represents the C=O stretching vibration of the acetyl group in lignin which reduced in the treated fibre.

B. SEM Analysis for the Produced Composites

The SEM micrographs in Figure 5 show the micro fibres and sawdust particles as highlighted. The micrographs revealed the homogeneity of the pure polyester composites (a) and dispersions of sawdust particles in the composites containing sawdust (samples b, d, e and f). The sawdust gave a better dispersion compared to the okra fibre in the matrix implying sawdust will absorb more water when exposed and also have higher mechanical properties. It can also be deduced that dispersion and homogeneity of polyester in the samples reduces with increase in filler sawdust as seen in the 10%, 20%, 30% sawdust and okra fibre composites.

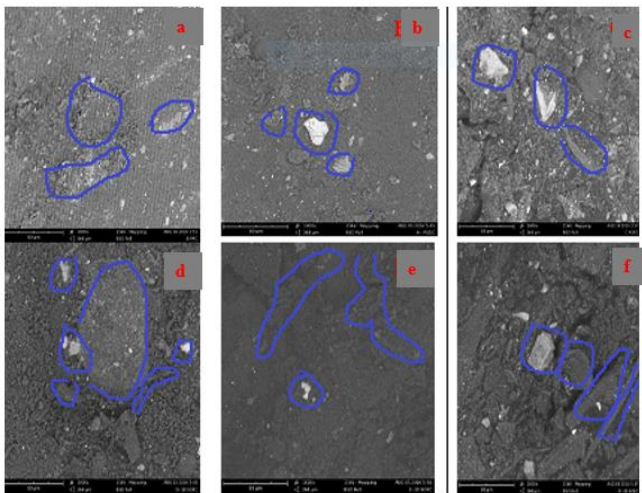


Figure 5: SEM analysis of the fabricated composite samples: (a) pure polyester resin; (b) Sawdust reinforced polyester; (c) Okra mat reinforced polyester; (d) 10% Sawdust + Okra mat; (e) 20% Sawdust + Okra mat; (f) 30% Sawdust + Okra mat.

C. Physical Properties of the Composite Samples

The ability to absorb water increased from sample A to F as shown in Figures 6-7. Sample A (unreinforced polyester) had the least water absorption of 0.09%, this is as a result of the hydrophobicity of pure polyester resin.

The hydrophobicity property of the polyester decreased with increase in natural fibre added to it, even though the fibres were treated. The treated natural fibres still possess some degree of hydrophilicity although reduced as shown in the FTIR results presented (Negawo *et al.*, 2018; Obogai *et al.*, 2020).

The composite with okra and 30% sawdust (Sample F) recorded the highest water absorption capacity of 1.6%. The

densities of all the composites were between $3.5 - 4.5\text{ kg/m}^3$. For the hybrid composites (samples D - F), D recorded the highest density of 3.82 kg/m^3 .

D. Mechanical Properties of the Composites

The mechanical properties of the composites were analysed based on the impact energy, hardness, elongation and Young's modulus as shown in Figures 8-11. The okra mat reinforced composite recorded the highest impact energy of 9.9 J while sawdust composite had the least impact energy of 0.25 J. Addition of 10% by weight sawdust to okra mat reduced its impact to 4.35 J which is about the same energy of unreinforced polyester composite. Implying that biomass even though light weight at certain composition and orientation can be tailored to have same or even better property of the unreinforced polyester (Obogai *et al.*, 2020).

Hardness and percentage elongation property reduced with filler content implying reduced ductility nature of the material as filler content increased (Sanjay *et al.*, 2018; Siakeng *et al.*, 2018) with unreinforced polyester recording the highest average elongation of 25%.

The Young's modulus is tenacity, rigidity and resistance of a material to deformation; otherwise known as modulus of elasticity (Silva-Guzmán *et al.*, 2018). It is. Increase in sawdust composition led to an increase in Young's modulus of the woven okra mat (samples C-F) until it attained a maximum Young's modulus value of 120 N/mm^2 . It then further dropped to 58 N/mm^2 at 30% sawdust on okra fibre reinforced polyester. This increase in property can be attributed to a proper adhesion, compatibility and interaction between constituent okra and sawdust in the polyester matrix (Ramadevi *et al.*, 2014; Horta *et al.*, 2017).

E. Dynamic Mechanical Analysis (DMA) of the Produced Composites

The dynamic mechanical analysis as presented in Figures (12-15) showed storage modulus decreased with temperature. This is as expected due to increased molecular mobility of polymer chains (Mandal and Alam, 2012; Sethuraman *et al.*, 2020). The Young's modulus decrease is significant at the glass transition temperature ($>60\text{ }^\circ\text{C}$) where there were several orders of magnitude drop (Paiva and Frollini, 2006; Poletto, 2017; Doddi *et al.*, 2019). The unreinforced composite recorded the highest storage modulus followed by sawdust reinforced polyester (b). This trend in storage modulus is similar to that of the Young modulus (Figure 11) i.e the storage modulus of unreinforced sample (a) is 2800 MPa while sample with 10% sawdust (b) is 2100 MPa. This difference however reduces as the temperature increased until the temperature reaches $80\text{ }^\circ\text{C}$ where the storage modulus of the two composite coincides. At $81\text{ }^\circ\text{C}$, the sawdust reinforced composite storage modulus exceeds that of the unreinforced polyester.

This implies that if the material is required at higher temperature environment and at modulus not exceeding 500 MPa, the sawdust reinforced composites will perform better. The loss modulus which is the maximum energy given out

during deformation exhibited similar trend to the storage modulus. The unreinforced material recorded the highest loss modulus followed by the sawdust reinforced composite. This behavior of storage and loss will be better appreciated in the damping factor which is a ratio of loss modulus to storage modulus. It is observed that the loss modulus initially increased with temperature to about 60 -75 °C before dropping (Asim *et al.*, 2019; Krishnasamy *et al.*, 2019).

The peak of the damping property is usually linked to the glass transition property of the material (Sethuraman *et al.*, 2020). While all the peak of Tan δ occurred around 100 °C,

the glass transition temperature (T_g) can be said to be at 100 °C. There is no significant difference in T_g as the reinforced material increased. The damping factor (1.41) was highest for 20 wt% sawdust /okra polyester which implies that sawdust and okra can help to increase damping property of a material and are candidate material for material damping and automobile industry (Ahmad *et al.*, 2014, Poletto, 2017; Chaudhary *et al.*, 2018). The elongation measures the quantity and quality of the ductility nature of a material. A decrease in elongation was observed for filled composite indicating that the fillers reduced the elasticity property of the material.

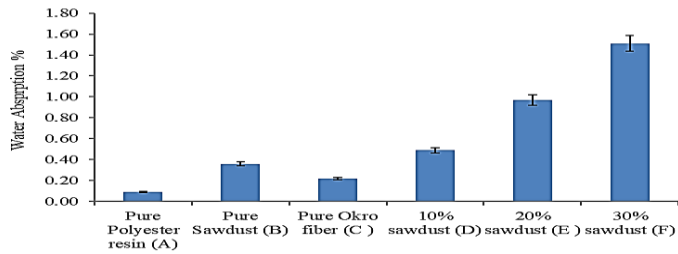


Figure 6: Water absorption property of the composites.

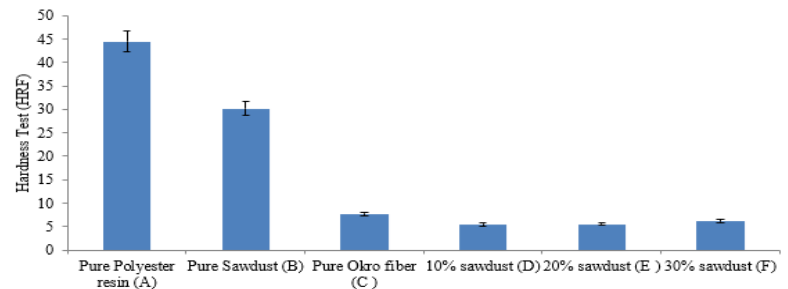


Figure 9: Hardness test on the composites.

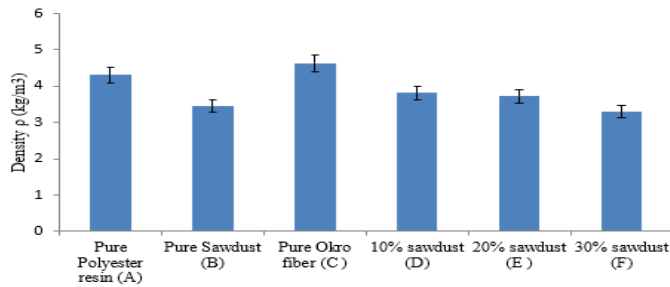


Figure 7: Densities of the composite samples.

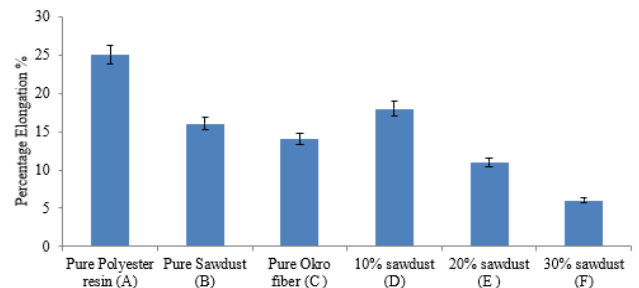


Figure 10: Percentage elongation of the composites.

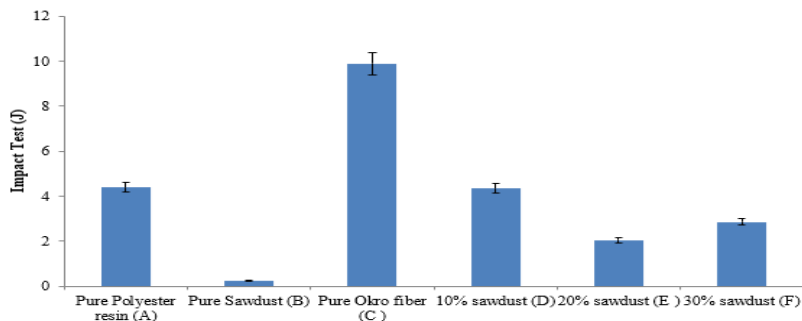


Figure 8: Impact energy of the composites.

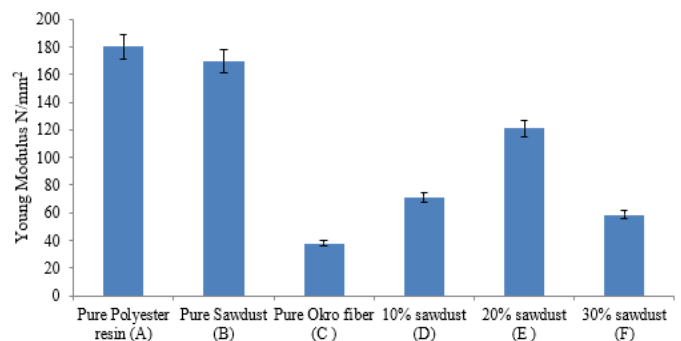


Figure 11: Young Modulus of the composites.

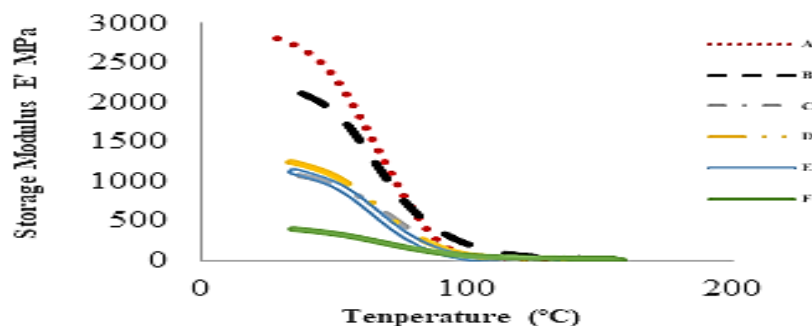


Figure 12: Effect of temperature on the storage modulus of pure polyester and the composites.

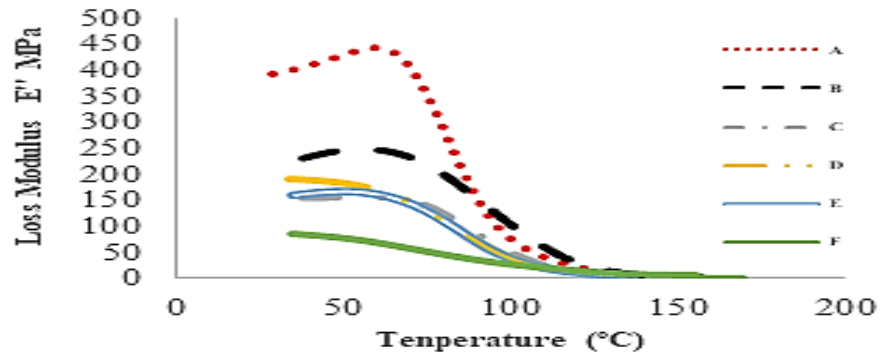


Figure 13: Effect of temperature on the loss modulus of pure polyester and the composites.

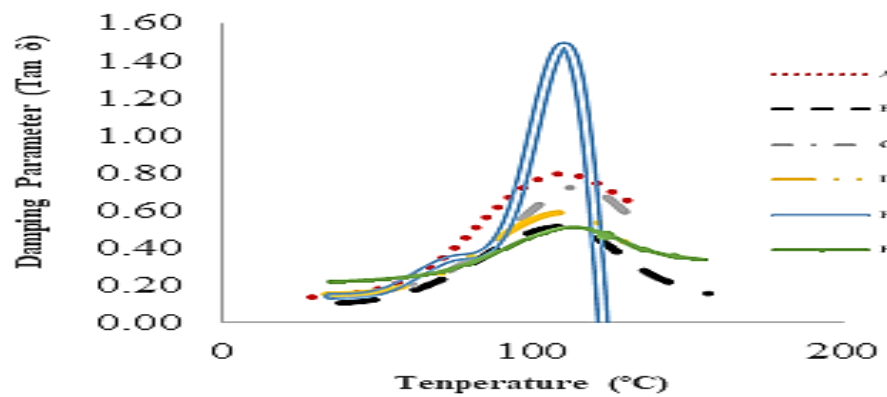


Figure 14: Effect of temperature on the Tan δ of pure polyester and the composites.

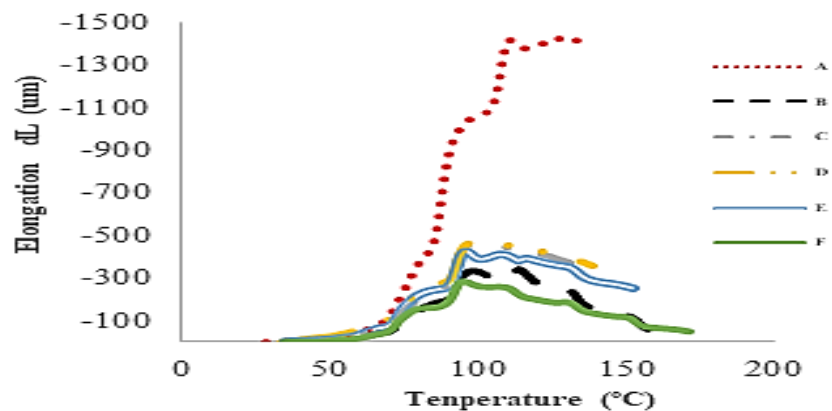


Figure 15: Effect of temperature on the elongation parameter of pure polyester and the composites.

IV. CONCLUSION

The use of stearic acid in fibre treatment reduced the hydrophilicity of the material thereby improving the fibre/matrix interaction. The physical properties of the composites showed that the composite with okra and 30%

sawdust (Sample F) recorded the highest water absorption capacity of 1.6%, also the densities of all the composites ranges between 3.5 – 4.5 kg/m³. The okra mat reinforced composite recorded the highest impact energy of 9.9 J than the unreinforced (control) material. The hardness of the reinforced material showed a decline as the biomass content increased in the composite. The unreinforced polyester recorded highest average elongation of 25%. This property

dropped with increase in okra and sawdust in the composite. In the hybrid composite, sawdust content improved the Young's modulus of the woven okra mat (samples C-F). Even though the unreinforced composite recorded the highest storage modulus at 40 °C, the sawdust reinforced composite recorded the highest at 81 °C and beyond prompting the use of reinforced composites at higher temperatures.

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