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Impact of Fibre Reinforced Polyester Composites on Tensile Strength of Baobab (*Adansonia digitata*) Stem

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Baobab plant also known as *Adansonia digitata* was used to extract the natural fibre, bast fibre was separated from the core mechanically. The bast fibre was retted by water, washed, dried and combed. A portion of the fine baobab fibre was separately treated with caustic soda (NaOH) and benzoyl peroxide prior to composites fabrication. Untreated baobab fibre was also used to prepare the composites to serve as a control. Both treated and untreated baobab fibre were subjected to Fourier Transform Infrared Spectroscopy (FTIR) analysis to confirm the modification effect. Composites of treated and untreated baobab fibre reinforced polyester were prepared by hand lay-up technique using a dumb bell-shaped mould, cobalt octanoate as a catalyst and Methyl Ethyl Ketone Peroxide (MEKP) as an accelerator. Mechanical properties of this synthetic composite were calculated using American Society's tensile in material manufacturing (ASTM) specifications. The cured composites were subjected to tensile strength, chemical resistance and water absorption tests. However, Scanning Electronic Microscopy (SEM) was used to determine the surface morphology of the synthesised composite samples analysis were used The results obtained shows an improvement in all the properties tested on alkali-treated and benzoylated baobab-fibre polyester composites.

Keywords: Baobab, Composite, Hand Lay-Up, Natural-fibre, Polyester.

1. Introduction

The use of natural fibres as reinforcement in the manufacturing of composites provides good incentives for the productive use of agricultural by-products. Natural fibres have many benefits over synthetic fibres, such as low weight, low density, low cost, recyclability, biodegradability and non-toxicity (Şahin and Mehmet, 2018; Montenegro, 2019).On the other hand, there are also disadvantages, such as lack of good interfacial adhesion, variations in quantity, low thermal stability and moisture absorption making the use of cellulosic reinforced fibre composites less attractive. Composite is a mixture of two different components, matrix and one of which is considered the reinforcing phase, e.g. threads, boards, pellets, etc. (Kalia *et al.,* 2011).

Limitations of natural fibre-reinforced composites can be resolved by surface alteration of the fibre as stated (Birniwa *et al*., 2019). Surface treatments are often used to improve the performance of natural fibre reinforced composites by bridging the compatibility gap between hydrophilic fibres and hydrophobic matrices. Natural fibres may be changed by physical or chemical processes. Physical treatment changes the structural and surface properties of fibres and their mechanical bonding with polymers. Chemical methods to alter natural fibre include the introducing a material that is compatible with both fibre and matrix. In particular, alkaline treatment, also called mercerisation, is a well-known method of enhancing lingo-cellulosic interfacial strength of fibre and resin with thermoset (Abdullahi *et al*., 2020). Mercerisation decreases the fibre diameter and eliminates padding, hemicellulose, wax and soil which covers the fibre surface. Moreover, it is well established that mercerisation contributes both to the creation of a rough surface topography resulting in improved fibre-matrix interface adhesion and an improvement in the number of celluloses exposed to the surface of fibres and polymer (Valander-Gonzalez,1999).

Fibre-reinforced plastic composites have long enjoyed a leading role with their high relative strength and modulus in a range of applications.

Because of their versatility, their lightness and the simplicity of producing complicated shapes with economical savings as compared to fibre-reinforced metal / alloys, fibre integrated plastics have been very common. Moreover, these composites can effectively substitute conventional materials in different fields, such as the building and furniture industries, transport and consumer goods (Xie *et al*., 2010;Vimalanathan, 2017; Birniwa 2018).

2. Materials and Methods

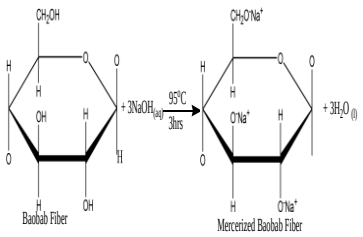
The polyester resin was obtained from NYCIL Nigeria limited, together with curing agents Methyl Ethyl Ketone Peroxide (MEKP) and cobalt octanoate as accelerator and catalyst respectively. Baobab plant as a source of the fibre was obtained from Kazaure in Jigawa state (Latitude: 12° 38' 54.46"N Longitude: 8° 24' 42.41" E).

2.1 Fibre Extraction

The bast fibre was immersed in water for seven days to achieve the ratting process. Then, the fibre was washed severally with tap water to remove the dissolved waxes and impurities. The fibre was brushed and combed using a nylon brush and allowed to dry at room temperature for three days which resulted to a fine strand of the material ready for treatment and composite fabrication (Morrinson *et al*., 1996; Velmurugan, *et al*., 2012; Gumel and Tijjani 2017).

2.2 Alkaline Treatment

The fibre was washed several times first with a non-ionic 2 % detergent solution. It was subsequently submerged in a thermostat unit in 5 % sodium hydroxide solution (NaOH) at 96 °C for three hours, in order to activate the hydroxyl (OH^-) group on the cellulosic fibre and introduce the sodium ion (Na^+) on the backbone of the fibre. Excess delignification of natural fibres can occur at higher concentrations above 5 %, resulting in weaker or damaged material. The fibre was then thoroughly washed with distilled water, dried for 24 hours in an air oven at 70 °C (Birniwa *et al.,* 2019; Montenegro *et al.,* 2019; Abdullahi *et al.,* 2020). Scheme 1 below showed the mercerization of baobab fibre.

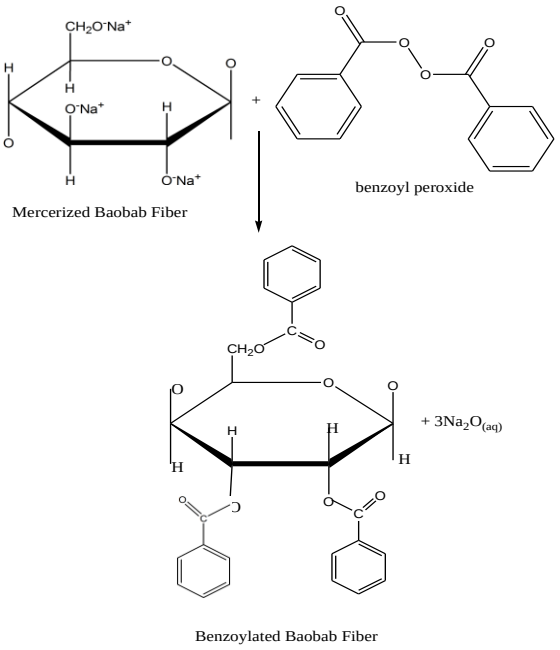


(Mohd *et al.,* 2012; Edeerozy, 2004)

Scheme 1. Alkaline Treatment of Baobab Fibre

2.3 Peroxidation

The fibre was cleaned with 2% non-ionic detergent solutions and soaked for 1 hour in 2% NaOH solution, then dissolved for 30 minutes in acetone in benzoyl peroxide (at 6% concentration). Instead of benzoyl peroxide some part was soaked in hydrogen peroxide for contrast with them. Complete peroxide decomposition was achieved by heating the solution at a temperature of 180^{o}C higher. Baobab fibre has been classified as treated with benzoyl (Lingtong *et al*., 2018). A conversion of mercerised baobab fibre into benzoylated fibre was shown in scheme 2.



Scheme 2. Benzoylation Treatment of Baobab Fibre

2.4 Polymer Matrix

The polyester resin is a type of thermosetting polymer that interlinks in the presence of a cross linker (hardener) when polymerised. The matrix material was developed in 2:1 volume ratio with a mixture of the polyester resin and hardener (Niharika and Acharya, 2013).

2.5 Composite Fabrication

A 140 x 35 x 10 mm size mould was typically used to cast the composite sheet. The standard method of hand lying (Abdurohman *et al*., 2018) was used to fabricate the composite samples as shown in Figure 1. A calculated quantity of polyester resin (100ml) and cobalt octanoate (1.6ml), MEKP (2.6ml), has been thoroughly mixed stirred gently to minimise air trap. A mould release agent was applied at the mould's inner surface for quick and easy removal of composite sheets. A thin layer of the mixture was poured after holding the mould onto glass sheet. A calculated quantity of the untreated and treated fibre necessary was then spread on the blend. The prepared resin mixture

was poured into the mould cavities gently to avoid air bubbles. The mould had been allowed to cure for 15 minutes at room temperature. The samples were later taken out of the mould and then retained for further testing.

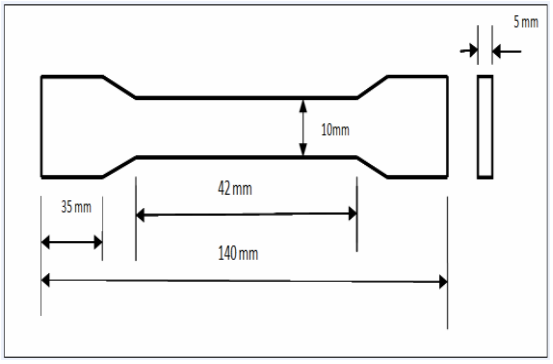


Figure 1. Schematic representation of the mould used.

2.6 Tensile strength Test

Following ASTM D3039 / D3039M-00 (2006) method, traction strength of dog bone shape specimens of 140 to 35 to 10 mm, was performed using the standard ultimate testing machine (UTM), Shimadzu (Model AG-1) with 20 mm / min cross head speed (Niharika and Acharya, 2013).

2.7 Fourier Transforms Infrared Spectrophotometric Analysis (FT-IR)

The Agilent technology (Cary 630 FITR, Japan Fourier Transformed Infrared Spectroscopy machine was used. The spectra of all the treated and untreated (baobab fibre) from 4000-650cm^{-1} were acquired. Spectroscopic analysis of the FTIR was performed on the natural fibre *Adansonia digitata*.

2.8 Chemical Resistance Test

The chemical resistance test was also conducted on the composites, under ASTM C868-02 (2012), three cured composites from each sample were tested and the average results were evaluated.

$$\%\, weight\ gain/loss = \frac{W_2 - W_1}{W_1} \times 100\% \quad \text{Equation 1.}$$

(Egwaikhideetal.,2007)

Where; W_1 = initial weight of the sample

W_2 = final weight of the sample

2.9 Water Absorption Test

The water absorption analysis was carried out by standard test procedure ASTM D570-98, (1998) for composite water absorption. Actual weight of each specimen was calculated before testing. At various temperatures (23, 50, and 100 ^{o}C, respectively) three sample specimens of each shaped composite were submerged in distilled water, after 24 hours the sample specimens were withdrawn from the water and cleaned with a soft tissue paper ensured it is dried before calculating final weight. The weight of the specimens was measured, and they were immersed again in water, the water absorption test was continued for several hours until a constant weight of the specimens were reached. The results are reported in Table 1.

The sample weight gain percentage was calculated at regular intervals, and the composite weight loss/weight percentage was assessed using equation 1.

2.10 Scanning Electron Microscopy (SEM)

The tensile research samples were broken in liquid nitrogen for 20 minutes of freezing in liquid nitrogen to examine the morphological properties of the fibre-matrix composite samples. Throughout an automated sputter coater (JEOL JFC-1600), the broken surfaces were coated with a thin film of metallic gold to make the base conductive and examined by a scanning electron microscope (JEOL JSM 6390 LV). The SEM picture was obtained with the following specifications: voltage acceleration, 20 KV; image mode, secondary electron image; 20 mm working distance; and 500 X magnification. The surface morphologies of the fibre samples were also analysed using the same microscope before and after chemical treatments. The corresponding picture is shown in Figure 4.

3. Results and Discussion

The chemical fibre alteration was carried out to enhance the performance of the fibre by introducing more reactive groups. It enabled the smooth bonding between the fibre and the polymer resin. The resultant material would therefore improve the mechanical properties of baobab fibre (Abdullahi *et al*., 2020). This was intended to promote the existence of complex groups on the fibre sheet. As such, interfacial properties can be enhanced, while better performance will boost the mechanical properties of natural fibre-reinforced polymer composite, the addition of aqueous NaOH in the hydroxyl group (Scheme 1) into the natural baobab fibre stimulated alkoxide ionisation. Washing of the fibre later, allowed Oxygen to react with H_2O to produce additional hydroxyl groups on the surface of the fibre. This created hydrogen bonding and also enabled a neutral pH to be achieved (Vimalanathan, *et al* 2017).

Table 1. Water Absorption for both Treated and Untreated Baobab Composite

Baobab Fibre	Initial Weight	Final Weight	% Gain/Loss
Treated	15.0	15.0	0
Untreated	15.0	15.3	+2

From the results obtained in Table 1, it is stated that the untreated baobab fibre composite absorbs a high amount of water compared to the treated baobab fibre composite, this could be attributed to the high content of impurities (such as lignin, pectin, waxes etc.), in the untreated fibre due to its hydrophilic nature compared to the treated fibre which has fewer impurity (hydrophobic) (Aderonke, *et al*., 2013).

Table 2. Chemical resistance test for both treated and untreated baobab fibre

Chemical Used (Conc.)	Treated Baobab (g)		Weight Lost/Gain (g)	Untreated Baobab (g)		Weight Lost/Gain (g)
	Initial Weight	Final weight		Initial Weight	Final weight	
10% H_2SO_4	15.0	14.8	-1.3	15.0	14.9	-0.6
	15.0	14.7	-2	15.0	14.8	-1.3
10% HCl.	15.0	15.1	+0.6	15.0	15.2	+1.3
	15.0	15.0	0	15.0	15.3	+2
10% KOH	15.0	15.0	0	15.0	15.3	+2
	15.0	15.0	0	15.0	15.3	+2

Table 3. FT-IR test of frequency of treated baobab fibre

Frequency cm^{-1}	**Bond**	**Functional Group**
1500-1400	C-C Stretch	Aromatics
1452.92		
1760-1690	C=O Stretch	Carbonyl
1723.92		
3640-3310	O-H Stretch	Carboxylic acids
3322.94		
Spectra of untreated baobab fibre		
3287.97	O-H Stretch	Free hydroxyl groups
2918.97	C-H Stretch	Methylene

The finding in Table 2 indicates that both treated and untreated baobab composite are resistant to chemical attack, i.e. hydrochloric acid and potassium hydroxide except for H_2SO_4. This is attributed to the strong contact of fibre and resin in the case of treated fibre, since both the handled fibre and resin are hydrophobic, in contrast, untreated fibre contains high levels of impurities, and there is no good resin compatibility with untreated fibre (Aderonke, *et al*., 2013).

All FT-IR spectral bands for treated and untreated baobab fibre as shown in Figure 2 and Table 3 are the same, except the presence of new peak at 1452.92cm^{-1}stretching vibration which is due to the presence of C-C bond in the aromatic ring and 1721.93cm^{-1}stretching vibration which is as a result of C=O for carbonyl compounds. These imply the treatment of the fibre has been achieved (Mosadeghzad, *et al.,* 2009).

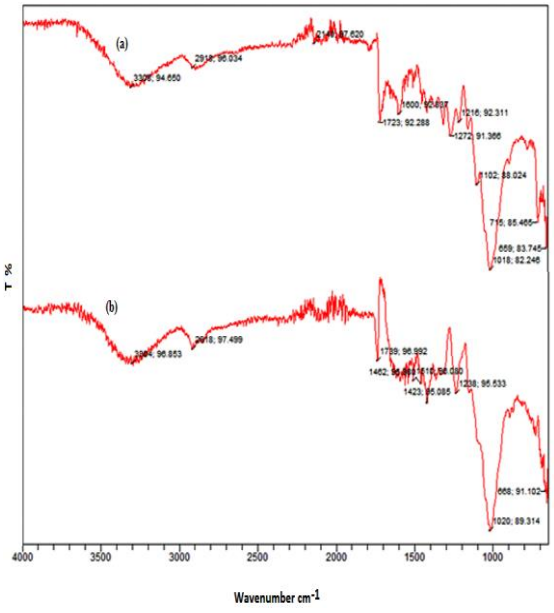


Figure 2. FT-IR Spectra of Treated (a) and untreated (b) baobab Fibre.

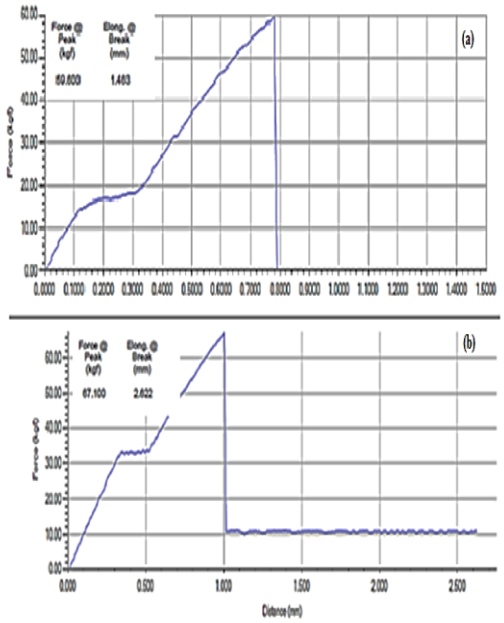


Figure 3 (a). Tensile spectra of untreated baobab reinforced polyester composite (b). The result tensile strength of treated baobab reinforced polyester composite.

Table 4. Tensile force at peak and elongation at break of the fibre samples.

Samples	FORCE at PEAK(kgf)	ELONGATION AT BREAK(mm)
Treated Baobab composite	67.100	2.622
Untreated Baobab composite	59.600	1.463

Tensile spectra of untreated baobab reinforce polyester composite

The result of the tensile strength of treated and untreated baobab composite is shown in Figure 3 (a) and (b), Table 4 reveals that the treated composite has higher tensile strength compared to untreated composite; this is due to both treated fibre and polyester resin are naturally hydrophobic; hence there is good compatibility between them. While untreated baobab fibre contain high level of impurities (hydrophilic) and polyester resin is hydrophobic; thus there is no good compatibility between them (Abdulsalam *et al*., 2014, Aderonke, *et al.,* 2013).

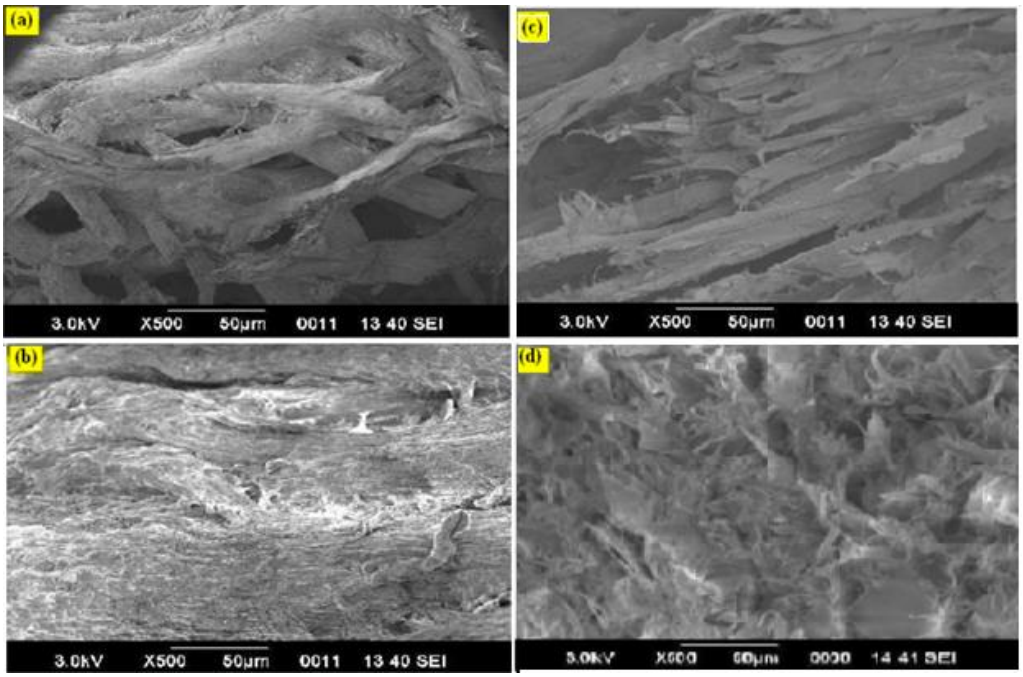


Figure 4. Scanning Electron Microscopic of baobab fibre.

SEM is an excellent method for studying the morphological composition of the fibre surfaces. Figure 4 (micrographs a through d, respectively) reveals the waxes, oils, and small particles that formed a protective layer on the untreated and alkali-treated fibre surface Figure 4b indicates a higher surface cracks and fibre bundle divergence as opposed to Figure. 4a. Furthermore, Figure 4a shows clear intercellular spaces that are distinguished and the unit cells are partly visible, which was not apparent in untreated baobab fibres. These traits could have been attributed to the gradual elimination of wax and sticky substances and the lack of cementing materials as lignin and hemicelluloses during treatment with sodium hydroxide.

Figure 5c indicate that the interfacial adhesion between the untreated fibre and the matrix was bad due to the weak bonding and the absence of any physical interaction between the two components. The fibres were taken out of the epoxy matrix and then separated the matrix and fibres by the tensile power. Because of the lack of interaction at the terminal, holes emerged from debonding along the fibre, resulting in poor stress transfer between matrix and fibre. Nevertheless, fracturing the samples did not cause the baobab fibre to crack, But, in Fig. 5d indicates a direct connection between the baobab fibre and the matrix owing to the lack of waxy content impurities and a close link between them. The mechanical properties of the composites have been corroborated with morphological proof.

4. Conclusion

Modification of fibre composite is promising for use as reinforcement in natural fibre material, as was evident from the results obtained in the hydrochloric acid absorption of water and tensile tests performed. The manufacture of the composite of baobab reinforced polyester resin is effective. The effects of FT-IR treatment have been reported as alkaline and benzoyl peroxide has also been effective in forming hydrophobic fibres and high tensile strength composites. The composite shows strong resistance to indentation and other severe deformations that the composite can show when used. However, the tensile and flexural measurements show that bonding strength is quite high compared to other managed and untreated composites, which is why the fibre / matrix adherent and reliability have improved. In view of the result obtained and the nature of the baobab fibre, there is need for further investigation through subjecting to high temperature in order to establish its thermal stability using either thermo gravimetric analysis or differential scanning calorimetric.

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Conflict of interest

The authors declare no conflict of interest.

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