



EFFECTS OF NaOH MODIFICATION ON THE MECHANICAL PROPERTIES OF BAOBAB POD FIBER REINFORCED LDPE COMPOSITES

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ABSTRACT

In order to improve properties of natural fibers as reinforcement, different treatment methods have being adopted by researchers. However, the use of sodium hydroxide (NaOH) for the treatment of baobab pod fiber as reinforcement in low density polyethylene is sparsely reported. Therefore, this study, investigated the effect of 2 wt%, 4 wt% 6 wt%, 8 wt% and 10 wt% concentration of NaOH on baobab pod fibers as reinforcement for low density polyethylene (LDPE). Two roll mill machine and hydraulic press at a pressure of 10 kN and temperature of 120°C aided the production of the composite. FT-IR was used to analyze the functional groups of the treated and un-treated fibers. The result showed the disappearance of the peak 1550 cm⁻¹ corresponding to lignin after modification. Further, the composites were characterized for the following tensile strength (TS), modulus of elasticity (MOE), elongation at break, impact strength and water absorption. Preliminary studies on the effect of loading of the unmodified baobab fiber in the LDPE matrix showed desirable properties at 10 wt%, where fiber content was in the range of 5 wt% to 30 wt% at interval of 5 wt%. The composite produced from the 8 wt% NaOH modified fiber had the highest tensile strength, MOE, elongation at break. At this modification level, the tensile strength, MOE and elongation at break were about 75.48%, 92.18% and 28% respectively higher than the composite produced from unmodified fiber. Composite produced with 10 wt% NaOH modified fiber exhibited least water absorption of 1.80%, which was 50% lower than unmodified. These showed that the modification of the fiber improved the composite properties. These properties compared favorably with some reported properties for natural fiber reinforced polymer composites.

Keywords: Alkaline modification, Tensile strength, Modulus of elasticity, Baobab fiber, FTIR.

1. INTRODUCTION

The use of natural fibers as reinforcement in polymer composites is gaining attention recently. This is because of environmental issues, high cost and unsustainable nature associated with conventional synthetic fibers [1 – 5]. Further, natural fibers have some advantages over their synthetic counterparts in term of physical, mechanical and biological properties; these include lower density, higher strength to weight ratio, higher specific properties, renewability and biodegradability. These make them useful in manufacture of bearings and linkages, building and automobile structures such as sliding panels [6].

Despite these desirable properties the application of natural fibers as reinforcements in the composite materials has some inherent challenges. This includes incompatibility with hydrophobic polymer matrix because of hydrophilic nature of the fibers. Additionally,

fibers have tendency of forming aggregates. Consequently, researchers adopted different methods in the quest for improving the compatibility of natural fibers with hydrophobic matrix. One of such methods is the chemical treatment which activates hydroxyl chemical groups, to make the fiber compatible with the matrix [7]. Also, it alters the surface tension and the polarity of the fiber which aids the dispersion of the fibers in the matrix and improves adhesion of the fibers and the matrix [8].

Chemical methods such as mercerization (alkali treatment), silane treatment, benzylation, peroxide treatment, permanganate treatment and use of some mild acids have been reported [9-13]. Silane treatment aids stability of composite materials, benzylation caused reduction in the hydrophilicity of fiber hence interfacial adhesion is improved. Further, peroxide treatment helps to reduce fiber moisture content and

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also the thermal stability. On the other hand, alkali treatment is the cheapest and the most applied of all the methods; it removes lignin, hemicellulose, oils and waxes, with these components removed, cellulose is left, thereby increasing aspect ratio of the fiber, rough surface topology for proper adhesion of the fiber and the matrix [9]. Using these treatment methods on fibers, different levels of properties modification have been achieved when used to reinforce matrix.

Optimum concentration of 10 wt% NaOH treatment was reported for coir fiber reinforced polyester composite [14], for sisal fiber used as reinforcement in polyethylene 10 wt% was achieved as optimum [15], while 5 wt% was recorded for sisal reinforced polyester composite [12]. The use of other fibers such as banana [16], jute [17 and 18], hemp [19] in composites production has been reported. However, literature on the use of baobab (*Adansonia-digitata*) in this area of application is scanty. Presently, their use is only limited to making of rope, string, cord for musical instruments, snares, fishing-nets, mats and waterproof hats [20]. These fibers are obtained either from the pod or bark of the tree. Strong fibers can also be obtained from the root bark. Baobab has one of the highest percentage elongation at break compared with some other fibers [21]. This premised the need to investigate the suitability of this fiber for producing composite materials. Therefore, the aim of this work is to study the effect NaOH modification on baobab pod fiber reinforced LDPE composite. Its suitability will add to the list of renewable and sustainable bio-fibers and can serve as substitute to other fibers. Due to the low compatibility between the hydrophilic fiber and hydrophobic matrix, treating baobab fiber chemically is a novel technique that will surely add to the archives of information to the body of knowledge in natural fiber-matrix composite processing and application.

2. EXPERIMENTAL

2.1 Materials

The Baobab pod fibers were sourced, extracted and treated with NaOH in National Research Institute for Chemical Technology (NARICT) Basawa, Zaria, Kaduna State, Nigeria while the low density polyethylene (Virgin) was purchased from Steve Moore Chemical Store Enterprise, Zaria Kaduna State Nigeria and used as purchased.

2.2 Methods

2.2.1 Treatment of baobab fiber

Sodium hydroxide solutions were prepared in 500 ml beaker by diluting 8 g, 16 g, 24 g, 32 g, and 40 g pellets of sodium hydroxide in 400 ml (400g) of distilled water

respectively to obtain concentration of 2, 4, 6, 8 and 10 wt% [22]. Baobab pod fibers of 1 mm size were soaked in the prepared 2 wt% of NaOH solution and heat at 40 °C for 1200s on a regulated hot plate under a continuous stirring to ensure even modification. The fibers were rinsed severally with distilled water until a neutral pH prior to drying in an oven at 50°C for 1200s. The same procedure was employed on baobab fibers at different concentration (4, 6, 8 and 10 wt %) of NaOH.

2.2.2 Composite production

Preliminary studies on the effect of loading of the unmodified baobab fiber in the LDPE matrix showed desirable properties at 10 wt%, where fiber content was varied from 5 wt% to 30 wt% at interval of 5 wt%. Consequently, the fiber weight was maintained at 10 wt% of the total composite weight for both the modified and unmodified baobab fibers. The fibers were mixed with LDPE in a two-roll mill machine model 5183, by Reliable Rubber Machinery Company, USA. The machine was preheated at the melting temperature of LDPE, 120°C in 1800s. LDPE constituting 90 wt% of the composite was poured into the preheated machine to melt for about 300s. Thereafter, the baobab fiber constituting 10 wt% was poured gradually into the melted LDPE until homogenous mixture was achieved. Finally, the compounded baobab/LDPE was removed from the mill in a form sheet.

Subsequently, a preheated (120 °C for 1800s) hydraulic press model number 12000, by Carver Incorporation, USA was used to produce the composites. Pre-sized compounded sheet placed in molds were placed on the preheated hydraulic press and compressed at pressure of 10 kN for a period of 360s. Thereafter, the mold was allowed to cool and the composite sample removed from it. This procedure was used to produce composite samples from the modified baobab fiber of varying NaOH concentrations.

2.3 Characterization

2.3.1 Fourier Transforms Infrared Spectroscopy (FTIR)

The functional groups of the modified and unmodified baobab fibers were determined using Shimadzu machine (model: FTIR-84005) FT-IR spectrometer, Japan. The samples were dried in an oven at 60°C. After which about 0.2 mg of the specimens were placed on a KBr plate and inserted into the infrared barrel. The infrared spectra of these samples were measured in a transmission wavelength number range between 4500 and 500 cm⁻¹.

2.3.2 Mechanical Properties and Water Absorption Tests

Tensile testing of the composite specimens was carried out using an Instron Machine Model 3369, System

Number 3369K1781, Capacity 50kN, USA. ASTM D638 [23] was adopted and three samples were tested and the mean recorded as representative value. The tensile test; from the test, the tensile strength, MOE and elongation at break were obtained. The impact test was conducted using impact testing machine serial number 412-07-15269C, by Norwood Instrument Limited, Great Britain. The test was conducted according to ASTM D256 [23]. ASTM D570-98[24] was used to carry out water absorption capacity of the samples and Equation (1) used to calculate the percentage of water absorbed.

$$\% \text{water absorbed} = \frac{W_f - W_i}{W_i} \times 100 \quad (1)$$

In (1), W_i (g) is the initial weight of dry sample; W_f (g) is final weight of the sample after soaking.

Three samples were used for each test and the mean value of the water absorption and their corresponding standard error was calculated.

3 RESULTS AND DISCUSSION

3.1 FTIR Spectra of Modified and Unmodified Baobab Fiber

The FT-IR spectra of the unmodified and sodium hydroxide modified baobab fibers are shown in Figure 1 while the peak positions and their corresponding assignments are given in Table 1. Changes were observed

in the FT-IR spectra of the modified baobab fibers when compared to the unmodified fibers. There was change in the intensity of the characteristic peak (3850 cm^{-1}) of C-H group stretching corresponding to hemicelluloses in the unmodified fiber to 2533 cm^{-1} after modifying. The peak 1683 cm^{-1} of bonded OH groups in the unmodified fiber changed to 1669 cm^{-1} in the modified fiber.

The band at 1669 cm^{-1} attributed to the stretching vibrations of OH groups disappeared after the modification process at 10 wt%. Its absence in the 10 wt% modified fiber indicated that lignin had been removed through the modification [25, 26 and 27]. The use of alkaline treatment reduces or removes the amount of lignin, wax and oils covering the outer cell wall of the fiber, depolymerizing cellulose hence exposing the short length crystallites. The disturbance of the hydrogen bond in the network structures increases the surface roughness of the fiber [9]. It is reported that one way of improving fiber-matrix adhesion at the interface is through good mechanical keying [28]. Therefore, the changes resulting from modifications as shown by the FT-IR will lead to increase in surface roughness, hence good chance of mechanical keying and probably better mechanical properties of treated fiber reinforced polymer composites.

Table 1: Infrared transmittance peaks (cm^{-1}) and possible assignments of chemical groups in unmodified and NaOH modified baobab fiber

Wave number (cm^{-1})	NaOH modified	Peak assignment
Unmodified Fiber Peak	Fiber Peak	
3850	-	C - H stretching in cellulose
2495	2425	C - H stretching C H and CH ₂ in hemicelluloses
1683	1666	Stretching vibrations of OH groups
1528	1526	CH ₂ symmetric bending
1193	1205	C - H ₂ bending in cellulose and hemicelluloses.

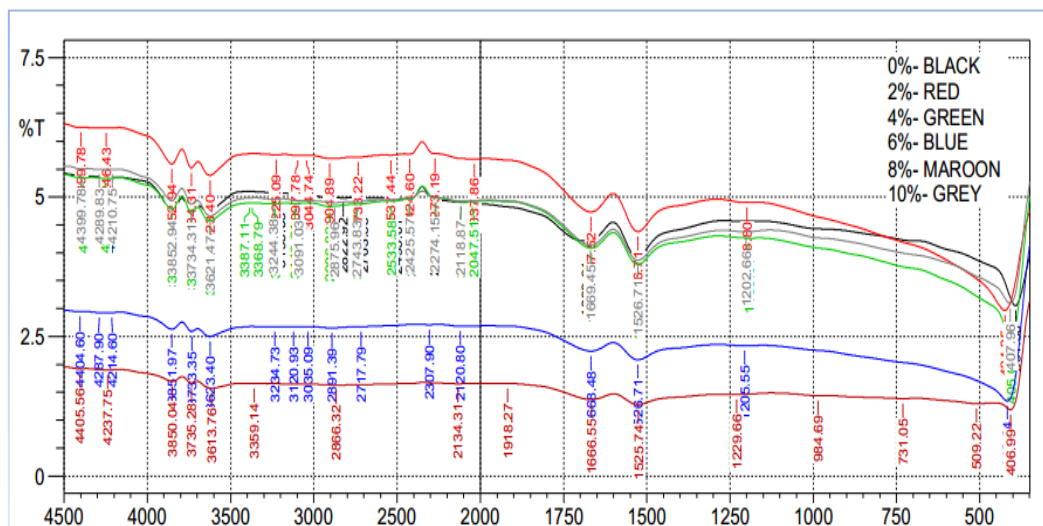


Figure 1: FT-IR spectra of unmodified and NaOH modified baobab fiber

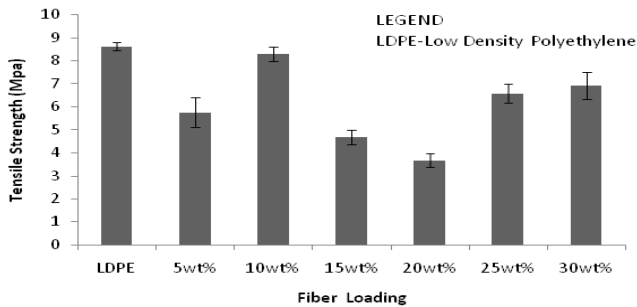


Figure 2: Effect of fiber loading on the tensile strength of unmodified fiber/LDPE composite

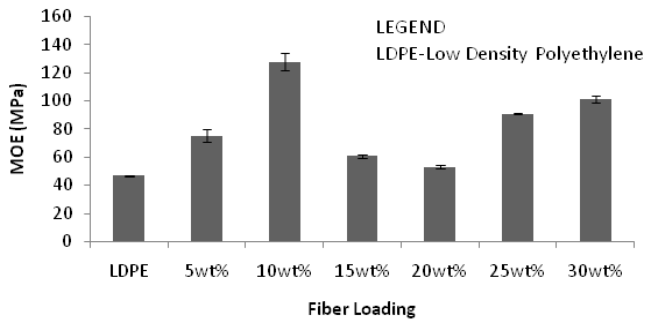


Figure 3: Effect of Fiber loading on MOE of unmodified fiber/LDPE composite

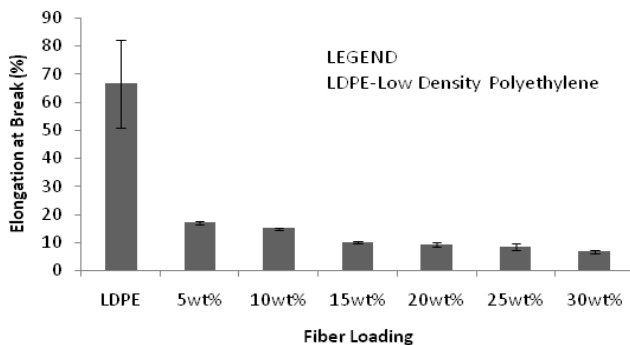


Figure 4: Effect of fiber loading on elongation at break of unmodified fiber/LDPE composite

3.2 Mechanical Properties of Unmodified Fiber/LDPE Composites

3.2.1 Tensile strength of unmodified Fiber/LDPE composites

Figure 2 presents effect of fiber loading on the properties of the composites produced from the unmodified baobab pod fiber. The unreinforced low density polyethylene (LDPE) had the higher tensile strength than the LDPE reinforced with unmodified baobab fiber. The decrease in the tensile strength between the reinforced and unreinforced LDPE might be due to the weak fiber and matrix interface resulting from the presence of impurities, lignin and waxes on the fiber. However, 10 wt% baobab fiber reinforced LDPE had the highest tensile strength among the reinforced LDPE, which decreased with increasing the fiber loading. The decrease could be as a result of fiber-fiber interaction at higher fiber content whereby load was not effectively

transferred through the matrix to the fiber [17]. At higher fiber content, poor interfacial bonding between the fiber and the matrix may occur due to excess fiber, hence, reduction in properties. The 8.28 MPa obtained for 10 wt% was lower than the optimal value of 10.4 MPa for Dum palm fiber-polyester composites [29] and 9.5 MPa for luffa-polyester composite [30].

3.2.2 MOE of Unmodified Fiber/LDPE composites

The effect of fiber loading on the Modulus of elasticity (MOE) produced composites is presented in Figure 3. As indicated in the figure, 10 wt% fiber loading exhibited the highest modulus of elasticity of 127.8 MPa. This suggests that the 10 wt% fiber loading composite exhibit highest degree of stiffness within the fiber loadings investigated. However, the drop in MOE after 10 wt% loading might be attributed to poor dispersion of fiber in the matrix at higher loading in addition to poor fiber-matrix interaction [17].

3.2.3 Elongation at Break of Unmodified Fiber/LDPE Composites

The result of the effect of fiber loading on the elongation at break of composites as presented in Figure 4 indicated that the elongation of the unreinforced LDPE was clearly higher than any of the reinforced baobab fiber composites. The elongation at break decreases with increasing fiber loading, with the elongation at break of 5 wt% baobab fiber reinforced composite slightly higher than that of 10 wt%. Increase in fiber content could result to this as the fiber is stiffer than the matrix hence polymer-polymer chain is broken by the fiber, hence reduction in elongation. The highest elongation at break of 17.13%, obtained in this work was higher than 1.98% and 0.94% elongations for Dum palm and luffa fiber reinforced polyester composites respectively [29, 30].

3.2.4 Impact strength of unmodified Fiber/LDPE composites

Figure 5 presents the effect of fiber loading on impact strength of unmodified fiber reinforced LDPE composite. Lower energy was required to break the composites material as compared to the virgin LDPE composite. The impact strength of the composites decreased with increasing fiber content, from 4.20 J/mm² to 1.57 J/mm². The trend observed could be as a result of increase in the stiffness of the composite as the fiber content was increased [31]. The highest impact was obtained at 5 wt% fiber loading, however the overlap in the error bar between 5 wt% and 10 wt% fiber loading, indicated that there was no significant difference in the impact strength of the two composites.

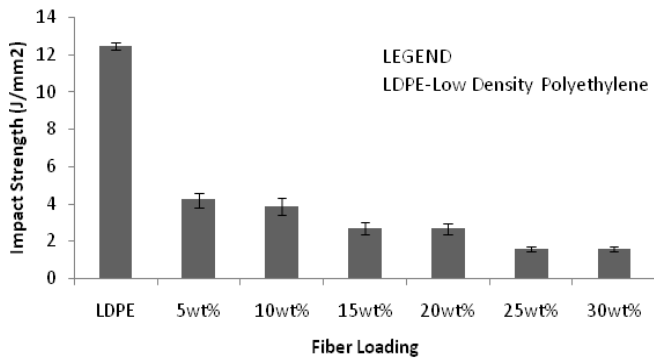


Figure 5: Effect of fiber loading on impact strength of unmodified fiber/LDPE composite

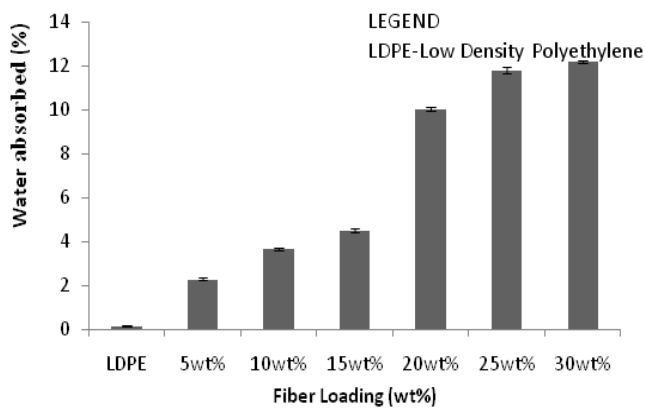


Figure 6: Effect of fiber loading on water absorption of unmodified fiber/LDPE composite

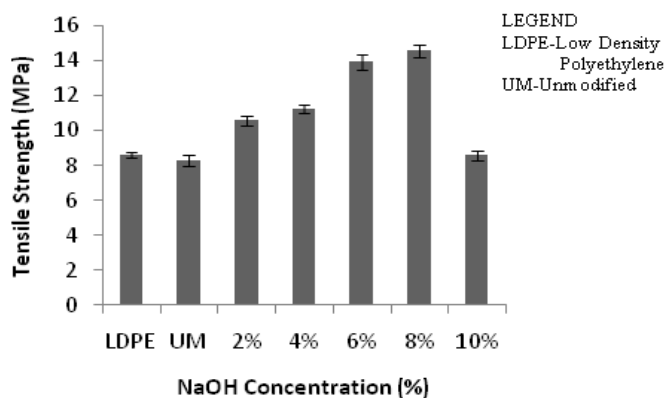


Figure 7: Effect of fiber modification on the tensile strength of the composite

The highest impact strength of 4.20 J/mm² obtained in this work was lower than the optimal value of 4.50 J/mm² at 5 wt% fiber loading for PALF-polyethylene composite [32], 4.80 J/mm² at 5 wt% fibre content for palm-epoxy composite [33] and 5.6 J/mm² at 5 wt% fiber loading in jute-coir fiber reinforced hybrid polypropylene composite [17], but higher than impact strength value of 0.008 J/mm² at 5 wt% NaOH concentration treated luffa fiber reinforced epoxy matrix composite [34]. These could be as a result of the difference in the nature of the fibers or the matrix used.

3.2.5 Water Absorption of Unmodified Fiber/LDPE Composites

It was observed that the unreinforced virgin low density polyethylene absorbed the minimal percentage of moisture due to its hydrophobic nature and on reinforcing with the baobab fiber, the capacity to absorb moisture increases as a result of the introduction of the hydrophilic baobab fiber. The water absorption of the composites increased with increasing fiber content. Similar trend has been reported [35]. Natural fibers are known to have hydroxyl groups which easily absorb moisture because of the formation of hydrogen bonding [36]. Higher fiber content could also result in higher voids entrapped in the composites, hence, higher water accumulation at the interface between fiber and matrix [37].

3.3 Mechanical Properties of Modified Fiber/LDPE Composites

3.3.1 Tensile Strength

Figure 7, shows the effect of fiber modification on the tensile strength of baobab fiber reinforced low density polyethylene composite. It was observed that the tensile strength of the composites produced with fibers modified with NaOH solution concentrations of 2 wt%, 4 wt%, 6 wt% and 8 wt% were higher than the unmodified after which there was a drop in tensile strength. This can be considered as a positive effect of modification thereafter a negative effect. The composite resulting from 8 wt% concentration of NaOH solution modified baobab fiber exhibited the highest tensile strength of 15 MPa, which was 75.48% higher than the unmodified baobab fiber composite.

The improved tensile strength observed at 2 wt%, 4 wt%, 6 wt% and 8 wt% of NaOH solutions may be attributed to the improved fiber-LDPE interface, resulting from better adhesion after the fiber was chemically modified, while the observed decrease in tensile strength could be attributed to fiber damage at higher concentrations of NaOH which could decrease the strength of the composite [14].

The highest tensile strength (TS) value of 15 MPa obtained at 8 wt% NaOH solution modified fiber was higher than 12 MPa reported for coir fiber reinforced polyester composite in which 10 wt% fiber and 10 wt% alkali treatment were used [14]. The TS was also higher than the 7.65 MPa reported for 20 wt% crushed luffa fiber reinforced recycled low density polyethylene (rLDPE) [22]. The TS obtained in this work was generally lower than reported values for synthetic fibers such as glass that is commonly used. The TS in this work was higher than 1.601 MPa for woven mat reinforced polyester, but much lower than 395.8 MPa for woven

mat reinforced with siloxane modified polyester, 64.4 MPa virgin glass fiber reinforced polyester [38].

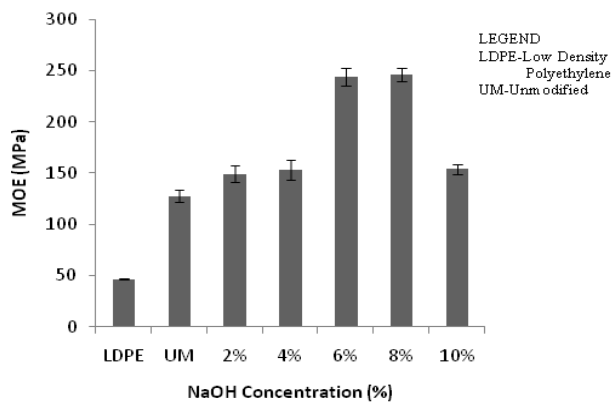


Figure 8: Effect of baobab fiber modification on MOE of the composite

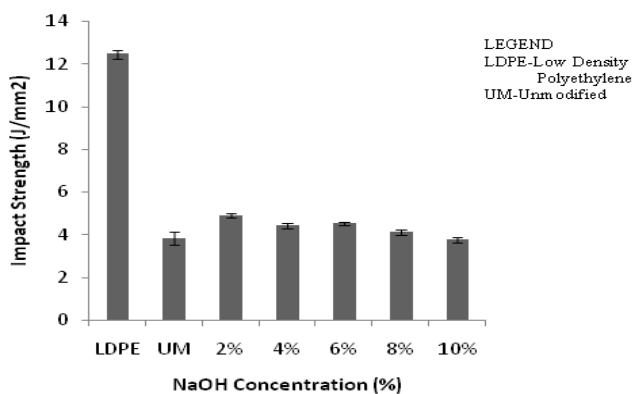


Figure 9: Effect of baobab fiber modification on the impact strength of the composite

3.3.2 Modulus of Elasticity (MOE)

Effect of fiber modification on the MOE of baobab fiber reinforced LDPE is presented in Figure 8. The 8 wt% NaOH modified baobab fiber composite exhibited the highest modulus of elasticity of 246 MPa. This suggests that the 8 wt% modified baobab fiber composite exhibit highest degree of stiffness compared to the other composites, i.e. composites reinforced with unmodified baobab fiber and those produced with fibers treated with 2 wt%, 4 wt%, 6 wt% and 10 wt% NaOH solutions. The enhancement in the MOE after the NaOH modification could be attributed to the fact that the modification resulted to increase in the roughness of the fiber, hence increase contact area with the matrix. Crystallinity index of the fibers may also have been improved because of the removal of cementing materials such as lignin. It was reported that removal of cementing materials led to better packing of the cellulose chains, therefore improved mechanical properties of fibers [29]. The decreased value of MOE observed after the 8 wt% concentration of NaOH may be attributed to the damage on the fiber surface as a result of high concentration of chemical used for the modification thereby making fibers lose their characteristics [9].

Optimal NaOH treatment of 8 wt% for luffa- rLDPE composite has been reported [22] and 10 wt% was reported for short sisal polyethylene composites [15]. The highest MOE obtained in this work was higher than reported MOE of Dum-palm fiber polyester composite of 64.15 MPa [29], 18.98 MPa established for crushed luffa fiber reinforced rLDPE [22]. However, it was much lower than 500 MPa reported for epoxy reinforced luffa fiber [32]. Fiber type, matrix type and fiber loading may be responsible for the differences noted [39]. The MOE was lower than glass fiber reinforced polyester in all cases. It was lower than 80.5 MPa for woven mat reinforced polyester, but much lower than 18000 MPa for woven mat reinforced with siloxane modified polyester, 7200 MPa virgin glass fiber reinforced polyester [38]. This is expected as synthetic fibers are generally stiffer than natural fibers.

3.3.3 Impact strength

The effect of fiber modification on the impact strength of the baobab reinforced LDPE is presented in Figure 9.

A positive modification effect was observed with the modified fiber composites compare to the unmodified fiber composite. There was about 23% increase in the impact strength of the composite made with baobab fiber modified with 2 wt% NaOH solution compared to the unmodified baobab fiber composite. The increase in the impact strength may be attributed to the increase in the fiber surface roughness, resulting in better mechanical interlocking and the increased amount of cellulose exposed on the fiber surface [40].

There was decrease in impact strength after the 2 wt% NaOH solution modification. It was reported that if the NaOH concentration is higher than the optimum condition, the excess delignification of the fibers can take place resulting to weakening or damaging of fibers and consequent decrease in impact strength of the composite may occur [9].

The highest impact strength of 4.91 J/mm² obtained from 2 wt% NaOH modified baobab fiber was higher when compared with the impact strength value of 0.008 J/mm² at 5 wt% NaOH concentration treated luffa fibre reinforced epoxy matrix composite [34]. The value was also higher than 4.50 J/mm² at 5 wt% fiber loading for PALF-polyethylene composite [29], 4.80 J/mm² at 5 wt% fiber content for palm-epoxy composite [33] but lower than 5.6 J/mm² at 5 wt% fiber loading in jute-coir fibre reinforced hybrid polypropylene composite [17]. The variation could be as a result of the difference in the properties of the fibers or the matrix used. The impact strength obtained in this work was generally higher than the reported values for glass fiber reinforced polymers. It was higher than 0.017.6 J/mm² reported for chopped

strand glass fiber reinforced epoxy resin and 0.1535 J/mm² for glass fiber reinforced polyester modified with sodium montmorillonite [38]. Glass fibers are known to be stiffer than the natural fibers, hence the reason for the observed superior impact strength of the composites from natural fiber.

3.3.4 Elongation at Break

Figure 10 presents the effect of fiber modification on the elongation at break of the composite. The composite produced from fiber modified with 8 wt% concentration of NaOH solution exhibited the highest percentage elongation at break of 20.75%. This was about 28% higher than the unmodified baobab composite. The enhanced percentage elongation on modification could be as a result of the removal of hemicelluloses, lignin, wax and pectin from the fiber. The removal of the impurities may have exposed more areas on the fiber surface for better contact with the matrix [41].

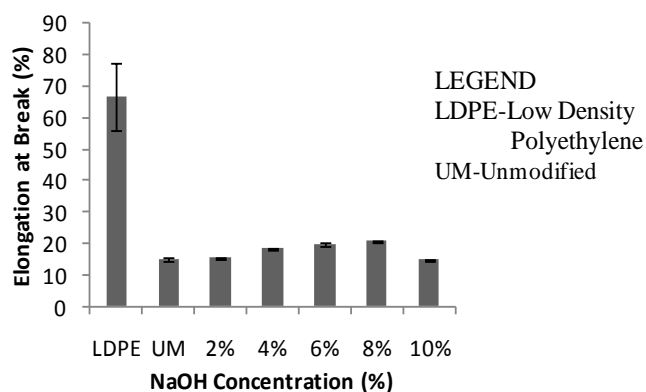


Figure 10: Effect of baobab fibre modification on the elongation at break of the composite

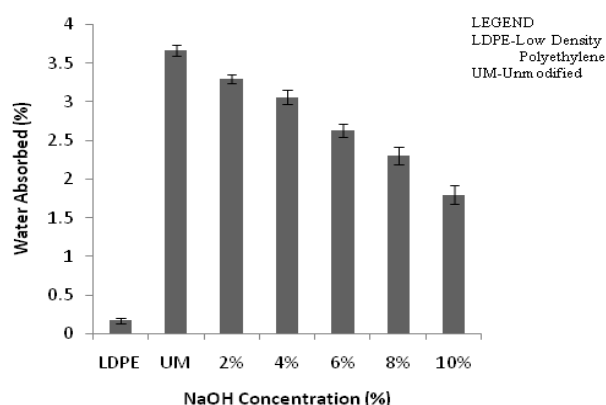


Figure 11: Effect of baobab fibre modification on water absorption of the composite

However, at very high NaOH modification concentration of 10 wt%, deterioration in the fiber properties could have occurred. This may be due to excess delignification of the fibers, which results in weakening or damaging of the fibers [9]. An optimal of 10 wt% NaOH treatment was reported for randomly arranged sisal-polyethylene

composites with 27% elongation at break [15], this was higher than the highest elongation at break obtained in this work. The elongation at break in this work was generally higher than values reported for glass fiber composites. It was higher than 3.9% for woven mat reinforced with siloxane modified polyester, 1.8% virgin glass fiber reinforced polyester, but lower than 20% for woven mat reinforced polyester [38].

3.3.5 Water Absorption

Figure 11 presents the effect of fiber modification on water absorption of the composite. As shown in the figure, water absorption capacity of the composites dropped from 3.7% to 3.3%, when the fibers were modified with 2 wt% concentration of NaOH solution. With further increase in NaOH concentrations, there was further decrease in water absorption of the composites as compared to the unmodified. The reduction in the water absorption could result from the surface modification of the fiber [8]. It was reported that on treatment of biofibers with alkali, sensitive hydroxyl groups are weakening hence easily react with water molecules and removed from the structure of the fiber [42]. The fiber's moisture resistance property was improved as a result of the reduced hydrophilic hydroxyl groups. The least water absorption of 1.8% recorded at 10 wt% NaOH modification in this study was lower than the reported 2.8% at optimal treatment concentration of 10 wt% NaOH [27]. This was higher than 0.5531% reported for 5 wt% NaOH luffa fiber reinforced epoxy [35]. The water absorption of glass fiber reinforced polyester composite was reported as 0.3962% [39]. The one obtained in this work was higher.

4. CONCLUSION

The present study established the potential of baobab pod fiber as bio-fiber as reinforcement in LDPE to produce composites. The effects that chemical modification by NaOH has on the bio-fiber, and hence the composites were presented. The following conclusions are therefore made:

The FT-IR analysis of unmodified and modified baobab fiber showed that, there was reduction intensity of the band representing hemicelluloses and disappearance of the lignin band in the NaOH modified fiber. This resulted to improved properties of the composites made from the modified fiber.

Most of the studied composites properties were highest at 10 wt% unmodified baobab pod fiber loading. Baobab fiber composites produced from 8 wt% NaOH solutions modified fibers exhibited 75.48%, 92.18% and 28% tensile strength, modulus of elasticity (MOE) and elongation at break respectively higher than unmodified

baobab fiber composite. Composite produced from 2 wt% NaOH modified fiber showed highest impact strength of 4.91 J/mm². This was 23% higher than the composite from the unmodified fiber. Composites produced from modified fiber at 10 w% NaOH solutions had the least water absorption and this was 50 % lower than the unmodified. This was an indication that the modification had effect on the properties of the composites and the results compared favorably with other reported works.

Consequently, the forgoing attests to the promising potential of fiber as reinforcement in composite materials production. This succinctly shows the economic importance of the study.

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