^{1,2}A. EFAOLA, ^{1.}I.O. OLADELE, ^{1.}B.O. ADEWUYI, ^{1.}A. OYETUNJI

DEVELOPMENT OF BIODEGRADABLE HIGH DENSITY POLYETHYLENE COMPOSITE FOR STRUCTURAL APPLICATION

¹Department of Metallurgical & Materials Engineering, Federal University of Technology Akure, Ondo State, NIGERIA ²National Agency for Science & Engineering Infrastructure (NASENI), Prototype Engineering Development Institute, Ilesha, Osun State, NIGERIA

Abstract: This study investigates high density polyethylene composites reinforced with yam stem particulate. Yam stem was obtained from the stem of harvested white yam tuber (*Dioscoreaspp*) and was treated with NaOH. Both treated and untreated yam stem fibres were pulverized to obtained particle sizes of <150 µm. The constituents were analyzed by gravimetric method whileThermogravimetric Analyzer (TGA) and X-ray Diffractograms (XRD) were used for characterization. The processed particles were used for the development of the high density polyethylene composites. The result showed that chemical treatment of fibre with NaOH enhances the removal of lignin and hemicellulose and, the retention of cellulose essential for reinforcement. TGA analysis revealed that the treated yam stem particulate were more thermally stable in comparison with the untreated while XRD analysis showed that alkaline treated peaks were more intense than the untreated due to the chemical treatment which was able to remove the unwanted fibre constituents from the fibre surface thus, exposing the cellulose which are highly crystalline. SEM analysis indicate improved adhesion in the morphology of the composites produced as there was no evidence of large agglomerate, delamination between the particles and the polymer matrix and pull out of the particles from the matrix. Results obtained from the mechanical tests showed that the mechanical properties of high density polyethylene composites while the treated performed better than the untreated composites while the treated performed better than the untreated composites while the treated performed of the HDPE; it therefore shows that good mechanical properties can be achieved by incorporating small amount of fillers into the matrix.

Keywords: biodegradable, high density polyethylene, mechanical properties, composites, structural applications

1. INTRODUCTION

Polyethylene (PE) is one of the most versatile and widely used thermoplastics in the world because of its excellent properties like toughness, near-zero moisture absorption, excellent chemical inertness, low coefficient of friction, ease of processing and unusual electrical properties. Polyethylene absorbs almost no water, Polyethylene is of low strength, hardness and rigidity, but has a high ductility and impact strength as well as low friction. Polyethylene is inert and translucent. This means that it allows light to pass through, but does not facilitate image formation, unlike in the case of transparent material. Polyethylene can be classified into several different categories but mostly do not depend on its density and branching. The main forms of PE are High-density polyethylene (HDPE), High molecular weight HDPE (HMW HDPE), ultrahigh molecular weight density polyethylene (UHMW-HDPE), linear low-density polyethylene (LLDPE), and very low-density polyethylene (VLDPE). These are divided based on density and branching. Generally, the most used PE grades are HDPE, low-density polyethylene (LDPE) and medium-density polyethylene (MDPE) [1].

There are various types of matrix that may be used to improve certain properties of composites. The selection of the type of additives for a certain composite depends on the application, the required properties, and the processing method [2]. High Density Polyethylene is used as matrix in this research.

Cellulose fibres are selected as reinforced filler for thermoplastics due to their superior mechanical properties and lower price compared to the thermoplastics [3]. The physical properties of fibre reinforced composite materials depend on the ability of the polymer matrix both to transmit stresses to the fibre reinforcement and to protect the fibres from damage. To promote these characteristics, fibres are treated with chemicals such as sodium hydroxide, potassium hydroxide and hydrogen peroxide in solution. Oladele *et al.*, [4] reported while comparing the various chemical treatments and molar concentrations that 1M concentration of sodium hydroxide is effective for the treatment of natural fibres.

The mechanical properties of particulate–polymer composites depend strongly on the particle–matrix interface adhesion, stress–strain behaviour of the filler or the matrix, and volume of particle loading [5]. The most important factor to obtain a good fiber reinforced composite is the adhesion between the matrix and the fiber. Due to the presence of a hydroxyl group in natural fibers, the affinity to moisture absorption is high which leads to poor wettability and weak interfacial bonding between the fibers and the matrices. Therefore, in order to develop composites with better mechanical properties, it is necessary to impart the hydrophobicity to the fibers by suitable chemical treatments. The effect of alkali on the cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes and hence, there is a lattice transformation.

Liu *et al.,* [6] analysed the effect of alkali treatment on the structure, morphology and thermal properties of the grass fibers based composite. The analysis shows that the fibers extracted from the grass can be used as a reinforcement material. Li

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et al.,[7] made a comprehensive study on various types of chemical treatments applied for the surface modification of the natural fibers. It is found that each treatment has its own advantages and disadvantage. Of the various chemical methods, alkali found to be most economical and easier way of achieving the improved properties by enhancing the better compatibility between fiber and matrix. Alsaeed *et al.,* [8] found that higher concentration of chemical treatment might decrease the strength of natural fiber. Cao *et al.,* [9] investigated the effect of alkali (NaOH) on the mechanical properties of bagasse fiber-reinforced polyester composites. Of the various concentration of NaOH used, superior properties were obtained for the composites made from 1% NaOH treated bagasse fibers. Vilay *et al.,* [10] compared the effect of NaOH and Acetic Acid on the mechanical and dynamic mechanical properties of the bagasse–polyester composite. The results indicate that the chemical treatment has improved the storage modulus and water resistant capability of the composite. Venkateshwaran *et al.,* [11] concluded in his research that; of the various percentages of alkali treatments, 1% provides better mechanical properties of the composite. From the above literature, it is found that alkali (NaOH) is an effective and cheaper method of chemical treatment employed to modify the fiber surfaces.

2. MATERIALS AND METHOD

Materials

The materials used for this work were white yam *(Dioscorearotundata*) stem obtained from Ilesa located in geographical coordinates of latitude 7° 36′ 95″ North and longitude 4° 42′ 90″ East, Ilesa West, Osun State, Nigeria which serves as the reinforcement and High density polyethylene was supplied by Safripol in South Africa. The high density polyethylene has a melt flow index (MFI) of 8.5 g/10 mins and a density of 0.953 g/cm³ with feed and melting extrusion zone temperatures of 170 °C and 220-230 °C, respectively. Other materials used were maleic anhydride grafted polyethylene (MAPE) G-2608

which serves as a compatibilizer for high density polyethylene supplied by Eastman chemical company, in South Africa while sodium hydroxide (NaOH) was sourced from Pascal Scientific Laboratory, Akure, Ondo State, Nigeria.

Methods

Fibre extraction and preparation

Yam fibre was obtained from the stem of harvested white yam tuber. The stem was removed from the yam stakes twined together as shown in Figure 1 when the stem was green or dried with the use of cutlass. Nodes were cut off by the use of pen knife in order to prevent the formation of weak points in the composite to be produced. The internodes were sliced into two; the fluffy materials in the core were scrapped making the spines at the bark to drop naturally. The yam fibre was washed thoroughly with distilled water to remove any adhering undesired soil and sun-dried. This was further chopped into smaller length using pen knife.

Chemical treatment

Chopped yam stem fibre was chemically treated with 1M concentration of NaOH solution at a temperature of 70 °C for 2 hours in a shaker water bath as shown in Figure 2 while some were left as untreated samples before pulverization. The treated fibre was washed and rinsed with distilled water until the pH of the rinse solution stabilized at 7. The same washing condition was carried out on the

untreated fibre. The fibres were later oven dried for two days at 65 °C before pulverized.

Production of yam stems particulate

Both the treated and untreated chopped yam stem fibres were pulverized using domestic marlex grinder followed by sieving with sieve shaker from where particle sizes of $<150 \mu$ m were collected and used as shown in Figure 3.

Determination of the yam stems particulate constituents

The Goering and Van Soest procedure for the determination of total Neural Detergent Fibre (NDF) was used to test for lignin, cellulose and hemi-cellulose so as to ascertain the efficiency of each of the treatment given to the particulates in the removal of lignin

and hemi-cellulose and retention of cellulose which was the fibre needed for the reinforcement [4]. The main effect of washing a natural fibre with a mild alkaline solution is the removal of waxes, hemicelluloses and a partial removal of the lignin present on their surface. This exposes the cellulosic content of the particulate for proper bonding of the matrix with



Plate1: Yam stem twinned around stakes



Plate 2: Samples in water shaker bath



Plate 3: Yam stem particulate

the particulate. Also, Faola *et al.*, [12] reported that chemical treatment of fibres with potassium and sodium hydroxide is effective for removing the lignin and hemi-cellulose and retention of the cellulose.

Drying of the yam stem particulate

The pulverized yam stem of both treated and untreated was bagged in a cellophane nylon and placed inside the oven at a temperature of 65°C for 24 hours to further remove the moisture content before mixing/impregnating.

Compounding of the composite materials

A good dispersion and interfacial adhesion between the matrix and bio-fillers are two critical factors for composites to achieve improved mechanical properties and this can be achieved by effective compounding of various components and by a suitable compounding process [13-14].

Predetermined proportions of HDPE, yam stem particulate (each for treated and untreated) were mixed together with High density polyethylene and MAPE. Each mixed samples were melt- blended together using twin-screw extruder at a Jone high-speed tumbler mixer for 10 minutes to obtain homogeneous mixture. Temperature of between 180-200°C and, rotor speed of 40 rpm for HDPE were used. The extrudates after air cooling were granulated into pellets with a guillotine grinding machine, bagged in a cellophane nylon and placed inside the oven at a temperature of 65°C for 24 hours.

Production of yam stem particulate reinforced HDPE composites

The pellets were poured in a tensile mould with guage length of 14 mm and a rectangular metallic mould of dimension 150 x 100 x 4 mm. The filled mould was placed in between the lower and the upper plates of the Carver laboratory press maintained at 190°C under a pressure of 10 tons for 10 minutes. Trapped gas was released during compaction by degassing the compact at about 2 minutes of compaction while the composites produced were immediately transferred to a cold press to cool down for 10 minutes.



Plate 4: Production process flow chart

Testing and structural characterization of samples

Composite samples were prepared for hardness, tensile, flexural, impact toughness tests and morphological tests (SEM, TGA and XRD) in order to study the behavioral performance of the composite in service condition. These tests were carried out as follows.

Tensile test

Dumbell-shaped cutter was used to punch out the tensile specimens from the composite samples. The test was carried out using Instron Universal Tensile Testing Machine in accordance with ASTM D638-10 standards. The test piece which is of gauge length 25 mm was fixed at the edges of the upper and lower grip of the machine and the test commenced. As the test piece is being extended, graph is being plotted automatically and important tensile properties data were generated. The load applied was 25 KN at a crosshead speed of 5 mm/min. Six pieces was tested for each sample from where the average value was taken.

Hardness test

The hardness of the neat polymer and composites were measured with the aid of micro-hardness tester, model 900-390, in accordance with ISO 868:2008 standards. This machine measures the resistance to penetration by measuring the depth of impression. The test was carried out by indenting the sample with the instrument at a test load of 50 gf, dwell time of 10 seconds and X50 magnification before taking the reading that was displayed on the monitor. Six values were taken for each sample and the average was taken as the representative value.

Flexural test

Flexural strength was carried out using Tensometric Universal Testing Machine in accordance with ASTM D790. Each sample of dimension 150 x 50 x 4 mm cut out from composite samples was firmly mounted on the Tensometric machine and as the sample stretched, the computer generate the graph as well as the desired parameters. Three samples were tested from where the average value was determined.

Impact test

The test was carried out on a Charpy impact testing machine (Instron CEAST 9050), in accordance with ISO179 standard. Samples cut into dimension 80 x 10 x 2 mm were placed horizontally on the machine, the notched surface is directly

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opposite the swinging pendulum, the initial reading of the sample gauge length and the thickness were entered into computer system attached to the machine and the machine was switched on. The pendulum of the machine swung freely through angle 150°C and fractured the sample. Six specimens were tested from where the mean value was evaluated.

Thermo gravimetric analysis (TGA) of the particulate and composites

Thermo gravimetric analysis (TGA) was used to measure the weight loss of particulate as a function of rising temperature. It was carried out on theyam stem particulate and, particulate reinforced composites developed to measure its level of weight loss as a function of increasing temperature performed under nitrogen atmosphere. The samples were heated from room temperature at nitrogen gas flow rate of 60 ml/min.

X-ray Diffraction analysis of particulate and composites

X-ray diffraction analysis was carried out on the particulate and the composite produced. Each of the treated and untreated particulate were compressed into disks using a cylindrical steel mould of ($\emptyset = 15$ mm) with an applied pressure of 20 MPa. A Phillips X'Pertdiffractometer fitted with a ceramic X-ray diffraction tube was used to determine the crystallinity. The diffracted intensity of Cu Ka radiation (wavelength of 0.1542 nm) was recorded between 5° and 40° (2 θ angle range) at 40 kV and 40 mA.

SEM observation

Field Emission Scanning Electron Microscope of Model JEOL JSM-7600F was used for the morphological characterization of the composite samples surfaces. This was carried out to investigate the miscibility of the particulate with the matrix at the fractured surfaces. Samples cut with bench vice were cleaned thoroughly, air-dried and glued on sample holder before coated with thick irradium in JEOL sputter ion coater and observed.

3. RESULTS AND DISCUSSION

Figure 1 shows the yam stem particulate constituents values for lignin, hemicellulose and cellulose. It was revealed that untreated sample has higher value of lignin and hemicellulose as compared with the treated.





Figure 1: Variation of yam stem particulate constituents in both treated and untreated conditions



The essence of pretreatment of the fibre was due to their hydrophilic nature as they are derived from ligno-celluloses which contain strongly polarized hydroxyl groups making their rate of water absorption to be high. The high water absorption rate leads to the degradation of the fibres and the fibre-matrix interface resulting in loss of mechanical performance. Since the lignin keeps the water in fibres [15], therefore, to reduce the water absorption rate of composite materials the lignin content of the fibre has to be reduced and this could be achieved by the pretreatment of the fibre with chemicals that can help in reducing the lignin content. From the result, the lignin content was reduced by 32% after treatment. Also, during chemical treatments, constituent like hemicellulose was hydrolyzed by the action of alkaline solutions [4]. In this research, the treatment causes the reduction in hemicellulose by 74%. The cellulose was reduced by 5% after the treatment which implies less reduction in the cellulose content of the fibre compared to other constituents. Figure 5, therefore, revealed the effectiveness of NaOH solution in the removal of lignin and hemicellulose and, the retention of cellulose which is the required constituent for the reinforcement.

Figure 2 shows the thermo gravimetric analysis of the treated and untreated yam stem particulate. From the result, thermal decomposition of the particulates as measured by weight loss and temperature was observed between 100-600 °C for both treated and untreated yam stem particulates. Weight loss in fibers occurs due to the decomposition of cellulose, hemicellulose and lignin constituents during heating [16]. It was observed that the untreated starts to lose weight earlier than the treated. This is attributed to the higher moisture content of untreated yam stem particulate whereby, the presence of hemicelluloses has caused higher moisture absorption of the composite [17]. The decomposition process was very mild until around 250 °C for both the treated and the untreated. However, a large weight loss occurred at temperatures above 250 °C up to 400 °C. At these temperatures, the cellulose was entirely decomposed due to the breaking down of its molecular structures. The decomposition temperature of the treated was higher than that of the untreated. This was due to the fact that most of the cellulose structure was crystalline, which is strong and resistant to hydrolysis in which the crystalline structure contained strong intramolecular and intermolecular hydrogen bonding that required higher energies to be broken down making the treated to be thermally stable than the untreated [18-19]. This

corresponds with the findings of Kim and Eom [20] and Nair *et al.*, [21] that higher decomposition temperatures give greater thermal stability. It was also observed that the untreated particles degrade at lower temperature compared with the treated. This was also reported by Beckerman and Pickering [22] 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.

Figure 3 shows the X-ray difractograms of the treated and untreated yam stem particulate. The low intensity peak represents the amorphous phase (lam) and the higher intensity peak represents the crystalline phase with crystallographic plane (002). The plot exhibit two prominent peaks obtained at 2θ values of 32° and 35° . It was observed that the crystallinity index of the treated particle was higher than that of the untreated. It was reported by Mangesh and Akshay, [23] that this may be attributed to removal of amorphous materials such as hemicellulose, lignin and pectin by the alkali treatment. This invariably results in close packing and stress relaxation of the cellulose chains, hence, an increase in the degree of crystallinity. Though, crystallinity index is useful only on a comparison basis as it is used to indicate the order of crystallinity of the crystalline regions [24]. The crystallinity index (CrI) of the treated and untreated yam stem particulate was calculated using the formula in equation (1) [25].



Figure 3: X-ray Diffractograms of treated and untreated yam stem particulate

Table 1: Crystallinity index values for both treated and untreated Yam stem particulate

Particulate	I_{am} (2 θ =	I ₀₀₂ (2θ	Crystallinity
	32.5)	=35.5)	index%
Treated	2991	11250	73.4
Untreated	2691	9250	70.9

$$CrI(\%) = \frac{I_{002} - I_{am}}{I_{002}} X \, 100$$

where, I_{002} and I_{am} are the peak intensities of crystalline and amorphous materials, respectively. I_{002} is the maximum intensity of diffraction of the (0 0 2) lattice peak at a 2 θ angle 35.5° and I_{am} is the intensity of diffraction

of the amorphous materials which is taken at a 2θ angle 32.5° where the intensity is at a minimum (Beckerman and Pickering, 2008).

Table 1 shows the crystallinity index for both treated and untreated yam stem particulate. Cellulose crystallinity of the yam stem particulate increases as a result of the influence of alkaline treatment of the particle. During chemical treatment, fiber constituents were removed from the fiber surface and, hence, the cellulose content and crystallinity index of the particle increases. Alkaline treatment removes the lignin and hemicellulose which are mostly amorphous from the fiber surface and hence, increases the crystallinity fraction of the cellulose which also loses part of the amorphous with respect to the peak characteristic of the cellulose system.

Figure 4 shows the ultimate tensile strength of treated and untreated yam stem particulate reinforced high density polyethylene composites. Tensile strength indicates the ability of a composite material to withstand forces that pull it apart as well as the capability of the material to stretch prior to failure. It was noticed from the plots that, the composites samples were of higher tensile



Figure 4: Ultimate tensile strength of particulate reinforced HDPE composites



Figure 5: Tensile Modulus of Particulate Reinforced HDPE Composites

strengths than that of the control sample. However, treated samples were slightly enhanced compared to the untreated ones. Sample reinforced with 20 wt% gave the optimum result with a value of 34.26 MPa while the untreated samples have their tensile strength values reduce as the reinforcement content increases from 10 to 40 wt% with values 31.58 and

(1)

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30.18 MPa, respectively. This correlates with the findings of Deshmukh *et al.,* [24] that tensile strength of composite decreases with increase in particulate filler concentration.

Figure 5 shows the tensile modulus for neat and YSP HDPE composites. Modulus reduces with increase in reinforcement content up to 30 wt% for treated HDPE composites; thereafter, the modulus value goes up for 40 wt% with a value of about 1873 MPa. This observed results was due the influence of proper dispersion of particulate in the matrix and the reinforcement weight content used while for the untreated, the modulus reduces with increase in reinforcement up to 20 wt% thereafter, an increase in modulus occurs. However, tensile modulus of the treated is higher than that of the untreated composites. This correspond with the findings of Bledzki and Gassan [25] that increase in tensile modulus of the kenaf treated fibre reinforced composites improves over the untreated fiber reinforced composites due to alkaline treatment attributed to improved bonding between the fibre and matrix.







Figure 7: Impact Properties of Particulate Reinforced High Density Polyethylene Composites

Figure 6 showed the hardness properties of both

the treated and untreated YSP reinforced High Density Polyethylene composites. Here, an increase in hardness strength was observed from 10 to 20 wt% before a decrease was observed at 30 wt% and a slight increase at 40 wt% making 20 wt% to have the highest hardness value of about 44 MPa for the treated particulate reinforced HDPE composites. The same trend was also observed for the untreated particulate reinforced HDPE composites.

Figure 7 showed the impact properties of the treated and untreated YSP reinforced High Density Polyethylene composites. It was noticed from the results that, there was increase in impact strength from 10 to 20 wt% particulate loading before a decrease was observed from 20 to 40 wt% for treated YSP reinforced HDPE composites. This was also observed in the untreated YSP reinforced HDPE composites with the same trend as the treated ones. Though, the impact strength of treated YSP reinforced composites were more enhanced than the untreated ones for each of the reinforcement content used, but 20 wt% YSP content have the highest impact strength for both treated and untreated HDPE composites with values of about 107 and 86 Mpa, respectively.



Figure 8: Flexural Strength of Particulate Reinforced High Density Polyethylene Composites



Flexural strength is the ability of the composite material to withstand bending forces applied perpendicular to its longitudinal axis. Figure 8 shows the flexural strength of the treated and untreated YSP reinforced Hiah Density Polyethylene composites. It shows an increase in flexural strength from 10 to 20 wt% before it starts to decrease from 20 to 30 wt%. The untreated HDPE composites also follow the same trend as the treated ones except for a remarkable decrease from 20 to 40 wt% that was observed. However, treated HDPE composites have higher flexural strength of 23 MPa compared to untreated HDPE composites and the neat sample with 21 MPa. Figure 9 shows the flexural modulus for neat and YSP HDPE composites. Modulus increases with increase in reinforcement content up to 20 wt% for treated HDPE composites with a value of 387 MPa; thereafter, the modulus value reduces for 30

and 40 wt% which may be due to improper

Figure 9: Flexural Modulus of Particulate Reinforced HDPE Composites

dispersion of particulate in the matrix. Also for the untreated, the modulus increases with increase in reinforcement up to 20 wt% before decreasing. However, flexural modulus the treated samples were higher than that of the untreated composites in all the variations similar to the flexural strength results with the neat sample having a value of 321 MPa.



Plate 5: SEM of 20 wt% Treated



Plate 6: SEM of 20wt% Untreated







Plate 8: SEM of 40 wt% Untreated

Plates 5-8 show the surface morphology for the yam stem particulate (YSP) from where it was noticed that the chemical treatment has actually removed some of the lignin that may hinder proper interfacial bonding as a smooth constituent, therefore, leaving the surface of the treated yam stem particulate wrought compared to the untreated particulate which has their surfaces smooth. However, there were agglomerations and voids in some of the samples as shown in the plates which were responsible for the poor mechanical properties in the developed composites. The composites from untreated particles were observed to posse these features. While small agglomerates and voids were observed at low filler content, large agglomerates of particles were noticed for higher filler content.

4. CONCLUSIONS

Yam stem particulate reinforced high density polypropylene composites were developed and characterized. The results obtained from the mechanical and morphological analysis carried out show that:

- Chemical treatment was found to be effective for the reduction of lignin and hemi-cellulose as well as retention of the cellulose constituents of the yam stems particulate.
- Composites developed from treated yam stem particulate perform better than the untreated composites. However, both composites gave better results than the unreinforced samples from HDPE in all the tests carried out.
- HDPE composites developed exhibited higher thermal stability as well as crystallinity compared with the neat sample.
 Lamellar thickness (L) of HDPE increases with the addition of YSP which indicates that YSP aid the formation of more perfect crystals. This can be attributed to that fact that the particulate act as heterogeneous nucleating agent inducing HDPE to form perfect and stable crystals.
- The reinforcement with optimum value was obtained at 20wt% yam stem particulate addition being the samples with the best results in tensile, hardness, impact, flexural, TGA and XRD which implies that best results can be obtained by incorporating small amount of the particulate into the matrix.

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