Morphological and Tensile Properties of Modified Surface Ukam Fiber Reinforced Polyester Composites

[1]Okpanachi George Echiye, [2]Temidayo Christopher Tofade,[3] Iheanacho George Ikenna, [4]Oyewole Eyitayo Oluwaseun, [5]Yahaya Mohammed Lawal Isa,[6] Adikankwu Henry
Centre for Satellite Technology Development, Abuja,
okpanachi.george@cstd.nasrda.gov.ng, +2348039718721

Abstract— Polyester resin composites reinforced with treated ukam fiber at various fiber contents are prepared by hand lay-up process. The microstructures of the composites are characterized by Scanning Electron Microscopy (SEM and their mechanical properties are investigated by tensile strength. The morphological and mechanical results indicate an improvement in adhesion between the polyester resin and ukam fiber upon alkali treatment. The mechanical properties of the composites show a significant increase in tensile strength with the addition of fiber, a gain of 86% is marked compared to neat polymer. Morphological studies reveal that the incorporation of fiber in polymer results in an increase in tensile strength. From the images, obviously the fibre distribution size of the treated composites varied from, 722.76 nm – 6.09 µm and they were smaller than that of the untreated composite that ranged from 894.20 nm - 8.69 µm. A reduction in size of the treated fibres leads to better mechanical properties of the composites than those of the untreated composites due to an increase in number of reinforcing fibres that were packed in a cross section of the matrix. This research offers an ecological alternative to upgrade the valorization of abundant and Nigeria resources. The use of plant fibres as a reinforcement in polyester matrices requires the issue of compatibility between the two phases to be addressed because plant fibres presents hydrophilic surfaces and polyesters are generally hydrophobic, good fibre matrix dispersion and wetting of the fibres by the matrix may result. As a consequence, the mechanical properties of the composites are severely reduced. This study considers the effect of fibre treatment by chemical modification of the fibres using silane, alkali, acetylation and potassium permanganate producing the composites at different volume fraction of 5%, 10%, 15%, 20% and 25%. The results showed an improvement in the mechanical properties of the composites due to the surface modification of the fibres.

Index Terms— Ukam fiber, Polyester resin, tensile strength, hand lay-up process

1 INTRODUCTION

In recent decades composite materials based on polymer matrix have attracted great interests for both industrial applications and fundamental research [1]. Due to the high cost of the petroleum derived products, to environmental hazard and to public concern for energy security [2], a growing effort has emerged in the research of polymer composites reinforced with natural fillers from renewable natural resources (fibers) instead of the synthetic fillers (carbon or glass) [3]

Natural fibres have the potential to replace glass fibres in polymer composite materials because of their low costs, low density, unlimited availability, biodegradability, renewability and recyclability [4]. These characteristics make natural fibres attractive as reinforcement of engineering polymer systems [5].

Natural fiber reinforced composites have comparable specific properties with glass fibre components and specific weight greater than synthetic fibre components [6] The use of natural fibres in thermoplastic composites provides a further benefit because the strength and toughness of the plastic is markedly improved [7]. However, certain drawbacks such as incompatibility with the hydrophobic polymer matrix, the tendency to form aggregates during processing and poor resistance to moisture greatly reduce the potential of natural fibres to be used as reinforcements in polymers [8].

The major advantage of using natural fiber as a reinforcement agent to polymer matrix is the improvement of the physical properties of the manufactured materials in comparison with the neat polymer [9]. However, there are several parameters affecting the mechanical properties of the manufactured materials. A good dispersion and interfacial adhesion between the matrix and natural fiber are two critical factors for the composites to achieve improved mechanical properties. A good dispersion/distribution can be achieved by effective compounding of various components and by a suitable compounding process [10]. In the present research, ukam fiberr was used as the reinforcing material since it is abundant in nature and has a minimal impact on the environment due to its biodegradable properties [11]. The ukam tree, (Cochlospermum planchonii), is a tropical plant, which belongs to the Sapotaceae family. Thus, the aim of this research is to prepare composite materials reinforce with treated ukam fiber as reinforcement at different concentrations (5 wt. %, 10 wt. %, 15 wt. %, 20 wt. % and 25 wt. %). The effects of treated fiber loading on the mechanical and morphological properties of composites are reported.

2 MATERIALS AND METHODS

2.1 Materials

Ukam fibres were sourced from Ikem in Isiuzo LGA of Enugu. The ukam plant fibre was obtained from the stem which consists of wood core with bast fibres. In this stem are a number of fibre bundles each containing individual fibre cells. The Ukam fibres was purchased from Juneng Nigeria Limited, Nsukka and Polyester resins, Polyvinyl alcohol (PVA), Cobalt octane, Methyl ethyl Ketone Peroxide (MEKP), Sodium hydroxide, acetic acid, silane, Benzoyl chloride and potassium permanganate (KMnO4) was supplied by Moore Chemicals, Zaria, Nigeria.

2.2 Fibre Surface Treatment

Alkaline treatment, silane treatment, potassium permanganate treatment and acetylation were used for chemical modification of the ukam fibres.

Alkali treatment

Alkaline treatment was carried out using NaOH solution in accordance with [12]. The Ukam fibres were soaked in 5 % of NaOH for 24 hours at room temperature. The fibers were washed several times with water to remove any alkali solution sticking to the fibers surface, neutralized with dilute acetic acid and then washed again with water. Finally, the Ukam fibres were sun dried for 48 hours with the fibres straight with no slacking thus preventing intersection of fibre within the matrix.

Silane treatment

Silane treatment was carried out using silane solution dissolved in water [13]. This contains 60 % ethanol and 40 % water mixed well and allowed to stand for an hour. The PH of the solution is 9.0.

Extracted Ukam fibres were allowed to react with silane by immersing in silane dissolved in a water- ethanol mixture for 3 h. After which the solution was decanted and the fibres were sun dried.

Potassium permanganate (KMnO4) treatment

The potassium permanganate treatment was carried out in accordance with [14]. The ukam fibres were thoroughly washed with water and soaked in 2 % NaOH for an hour. The alkali treated fibers were soaked in 0.5 % KMnO4 in acetone for half an hour. The permanganate soaked fibre was put into distilled water for 10 minutes to catalyze the reaction. The fibers were then decanted and sun dried.

Acetylation

Ukam fibres were soaked in demineralized water for an hour, filtered and placed in a flask, containing acetylating solution [15]. Acetylating solution consists of 250 ml toluene, 125 ml acetic anhydride and a small amount of catalyst perchloric acid (60 %). The process temperature of acetylation was 60 ^{o}C and duration of 1 to 3 hour. After modification, the fibre was washed thoroughly with distilled water until acid free. Finally modified ukam fibres were sun dried before preparation.

2.3 Sample Preparation

Ukam fiber was used as reinforcement and general purpose polyester resin as a matrix for the composite material of the laminated specimens. The steps of manufacturing the composite using the hand lay-up process are described below (Mohammed et. al. 2010). The hand lay-up process is an open molding technique. The surface was thoroughly cleaned for use by removing any dust and dirt from it. After the mold surface has been cleaned, the release agent was applied and the mold surface was coated with free wax using a smooth cloth. Then a film of polyvinyl alcohol (PVA) is applied over the wax using sponge. PVA is a water-soluble material and 15% solution in water is used. When water evaporates a thin film of PVA is formed on the mold surface. PVA film was dried completely before the application of resin coat. This is very important because it helps in the removal of the laminates from the mould. The matrix material was prepared using general purpose polyester. Cobalt octane (0.35% by volume of resin) is added to act as accelerator and Methyl Ethyl Ketone peroxide (MEKP) (1% by volume) is added to act as catalyst. Resin, accelerator and catalyst were thoroughly mixed. The use of accelerator is necessary because resin does not cure properly. After adding the accelerator and catalyst to the polyester resin, it is left for some time so that bubbles formed during stirring may not die out. The amount of added accelerator and catalyst is not high because a high percentage reduces gel time of polyester resin and may adversely affect impregnation. A unidirectional ukam fiber with thickness (0.3 mm) was used as a reinforcement, which was cut in layers of required dimensions for each specimen. To prepare the laminate, the Ukam fiber is laid in the mould, the resin is spread uniformly over the fiber by means of a brush until fibers are immersed in the resin. External pressure plate is used to apply pressure while curing to enhance uniform thickness reached. The casting was carried

out at room temperature for 24 hours and firmly removed from the mold to get a fine finished composite plate.

2.4 Characterization

Tensile test

This was carried out according to ASTM D 638 - 03 [17]. Hounsfield (Monsanto) Tensometer (Universal Testing Machine) (model No. S/N 8889) was used to determine the elastic modulus, ultimate tensile strength and percentage elongation in length of the materials. The specimen geometry was in a dumb-bell shape and the dimensions were ascertained using the vernier calipers. Uni-axial load was applied to each ends of the respective samples until it failed. Maximum stress, strain, percentage elongation and modulus of elasticity were obtained as follows:

$\sigma(MPa)=P/A$

Study of Morphological Surface of the Composites

Scanning electron microscopy (SEM) was employed in order to observe the morphology of the sample's surface of the composites specimen with a field-emission Scanning Electron microscope Phenom Prox. Model No. MVEO 16477830 manufactured by Phenom World Netherlands.

The samples were cleaned properly and rubbed with sand paper to remove unwanted particles sticking to the surface. It was then air-dried and coated with a thin layer of gold using sputter coater to avoid electron accumulation. The samples were coated to make them conductive. They were then placed on a sample holder in vertical orientation in such a way that the surface was observed directly under the microscope (at 15 kV) and the morphology of the produced samples were viewed and photographed.

3 RESULTS AND DISCUSSION

3.1 Surface morphology of the composites

Figures 1-4 displays surface morphology of composites with different treatments. Figure 1 showed morphology of the untreated sample. From Figure 1 it was revealed that the fibres are not well compacted with polyester matrix. Figures 2-4 showed the SEM of the treated composites. The morphologies reveals continuous surface along the fibers as well as no fibrillation occurred. The fiber surface area increase after treatment, each single fibril can be clearly observed because of removal of some impurities through treatment. The smoothest fiber surface is exhibited in Alkali and Silane treated fibers as shown in Figures 1-4.

Figures 1-4 show surface morphology of composites at 25 % fibre loading for all the treatments. From the images, obviously the fibre distribution size (See Figure 1b -4b) of the treated composites varied from, 722.76 nm – 6.09 µm and they were smaller than that of the untreated composite that ranged from 894.20 nm - 8.69 µm. A reduction in size of the treated fibres leads to better mechanical properties of the composites than those of the untreated composites due to an increase in number of reinforcing fibres that were packed in a cross section of the matrix. The morphology of the untreated composite (See Figure 4b) shows that the fibres are not well compacted with polyester matrix, with continuous surface along the fibers as well as non-occurrence of fibrillation.

Alkali system exhibited similar behaviour to those of silane system. This may be due to the increment in the effective fiber surface area and surface roughness which are available for interaction with the matrix after removal of some lignin and hemicellulose by treatment. Treatment surface has been obviously covered by polymer matrix leading to better stress transfer in this composite system. Good fiber dispersion in the all composites has been obtained in spite of fiber content as high as 25%wt[16].

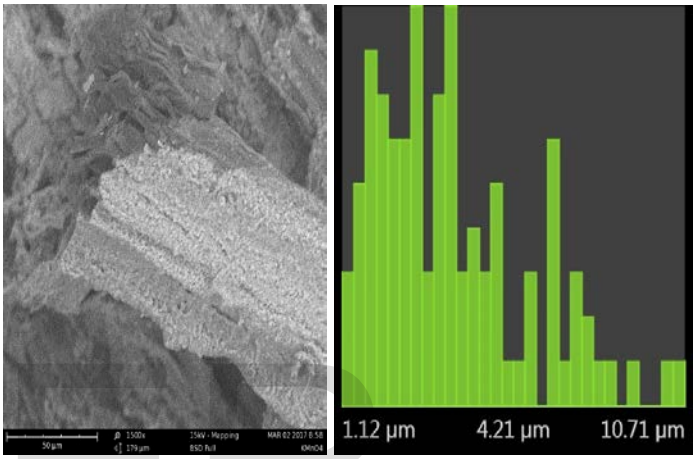

Figure 1: Scanning electron microscopy image of acetylation treated ukam fibre reinforced polyester composites at 25% fibre loading

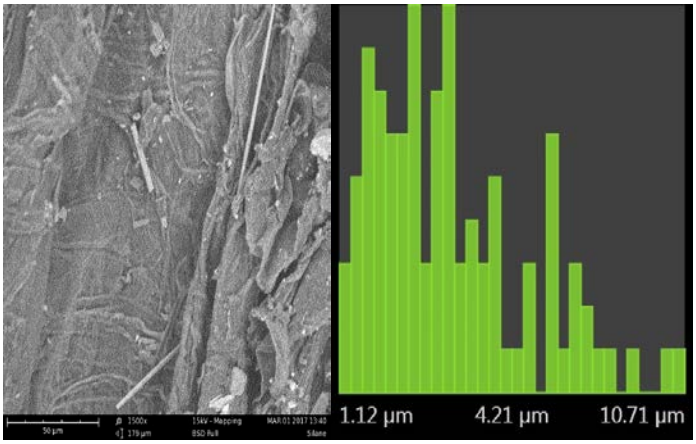

Figure 2: **Scanning electron microscopy image of alkali treated ukam fibre reinforced polyester composites at 25% fibre loading**

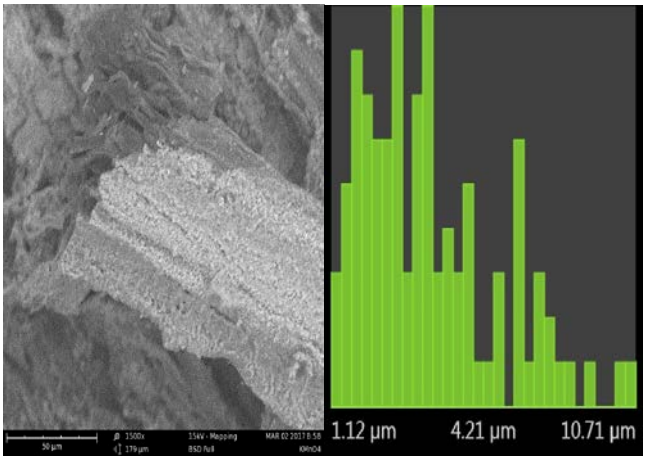

Figure 3: Scanning electron microscopy image of $KMNo_4$ treated ukam fibre reinforced polyester composites at 25% fibre loading

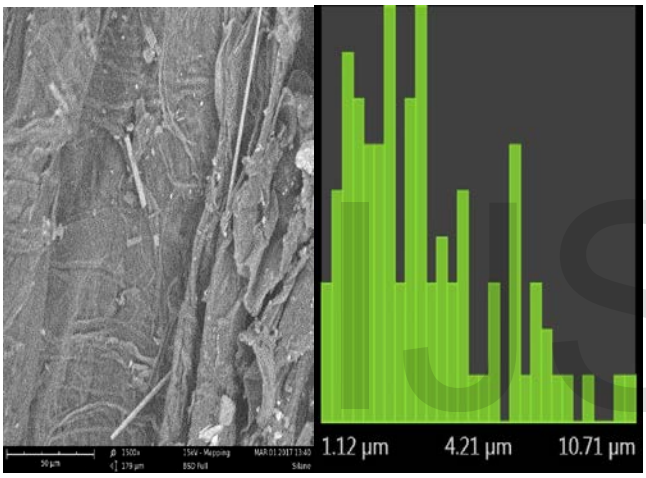

Figure 4: Scanning electron microscopy image of silane treated ukam fibre reinforced polyester composites at 25% fibre loading

3.2 Tensile properties

The results of the tensile modulus, tensile strength and % elongation of the treatment is showed in Figures 5. From the results, it can be observed that chemical treatment have serious effect in the tensile properties, for example the tensile strength of 217.22, 181.85, 213.83. 158.97, 149.43 MPa and elastic modulus of 1.0217, 1.5276, 1.0196, 1.0071, 1.3238 MPa were obtained for the untreated, alkali, silane, acetylation and KNNO4 treatment at 5wt% fibres respectively.These values increased to maximum at 15wt% fibres for the untreated with values of 221.10 and 1.4852Mpa for tensile strength and elastic modulus, 20wt% Fibres for Alkali treatment with values of 333.2 and 1.9578MPa for tensile strength and elastic modulus. For Silane treatment 25wt% optimum of 224.59 and 2.7521MPa for tensile strength and elastic modulus, 25wt% for the $KMNO_4$ treatment with values of 223.22 and 1.906MPa for tensile strength and elastic modulus, 25wt% for the Acetylation treatment with values of 172.81 and 1.7677MPa for tensile strength and elastic modulus.

The results showed that the composite has a relatively higher Young's modulus, tensile strength and %elongation, as shown in Figures 5. The Young's modulus of the composites with Ukam fibres increment is attributed to the inherent high stiffness strength of the reinforcement[17].

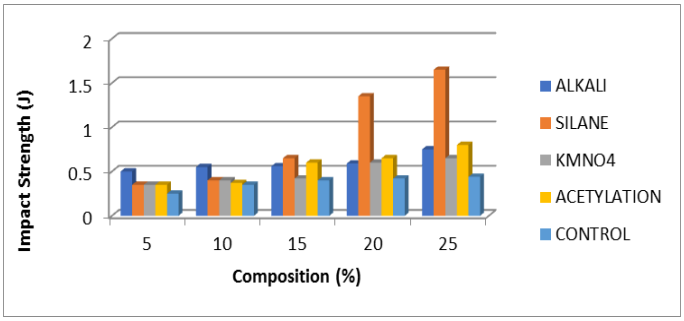

Figure 4: Variation of Impact strength with wt% Ukam fibres

.

CONCLUSION

Scanning electron microscope (SEM) investigation show that surface modifications improves the ukam fibre/matrix adhesion.The fiber surface area increased after treatment, each single fibril can be clearly observed because of removal of some impurities through treatment. Tensile strength of the Ukam fibres reinforced composites has been clearly improved when compared to that of neat polyester. These can be ascribed by strong interfacial adhesion and consequently good load transfer between the treated fiber and matrix which lead to high tensile strength

Acknowledgment

Iam greatly indebted to my project supervisors, Engr. Prof. A.Ashwe, Engr. Prof. G.B. Nyior and Engr. Dr. H.A. Iortyer, for instilling in me their zeal for academic thoroughness. Thanks also go to all the lecturer and staff of the Department of Mechanical Engineering, Federal University of Agriculture, Makurdi. Also worthy of mention is Dr Abdulwahab of the Department of Materials and Metallurgical, Ahmadu Bello University, Mr Danladi and Mallam Abubakrar of the Mechanical Engineering Department who assisted me in carrying out the mechanical property tests on the specimens and the Heads of Department for the permission to use the equipment in the various workshops in the University.My appreciation also to Professor S.O Mohammed, the Director General, National Space Research and Development Agency, Abuja, Engr Dr Spencer Onuh, MNSE, Director/CEO, Centre for Satellite Technology Development, Abuja and all management staff for

granting me the study leave which gave the opportunity to undergo this programme.

REFERENCES

1 Thomas, C., Borges, P., Panzera, T., Cimentada, A., and Lombillo, I.: 'Epoxy composites containing CFRP powder wastes', Composites Part B: Engineering, 2014, 59, pp. 260-268

2 Malha, M., Nekhlaoui, S., Essabir, H., Benmoussa, K., Bensalah, M.O., Arrakhiz, F.E., Bouhfid, R., and Qaiss, A.: 'Mechanical and thermal properties of compatibilized polypropylene reinforced by woven doum', Journal of Applied Polymer Science, 2013, 130, (6), pp. 4347-4356

3 Kakou, C., Arrakhiz, F., Trokourey, A., Bouhfid, R., Qaiss, A., and Rodrigue, D.: 'Influence of coupling agent content on the properties of high density polyethylene composites reinforced with oil palm fibers', Materials & Design, 2014, 63, pp. 641-649

4 Joseph, P., Joseph, K., Thomas, S., Pillai, C., Prasad, V., Groeninckx, G., and Sarkissova, M.: 'The thermal and crystallisation studies of short sisal fibre reinforced polypropylene composites', Composites Part A: Applied Science and Manufacturing, 2003, 34, (3), pp. 253-266

5 Joffe, R., Andersons, J., and Wallström, L.: 'Strength and adhesion characteristics of elementary flax fibres with different surface treatments', Composites Part A: Applied Science and Manufacturing, 2003, 34, (7), pp. 603-612

6 Westman, M.P., Fifield, L.S., Simmons, K.L., Laddha, S., and Kafentzis, T.A.: 'Natural fiber composites: a review', in Editor (Ed.)^(Eds.): 'Book Natural fiber composites: a review' (Pacific Northwest National Laboratory (PNNL), Richland, WA (US), 2010, edn.), pp.

7 Misra, S., Misra, M., Tripathy, S., Nayak, S., and Mohanty, A.: 'The influence of chemical surface modification on the performance of sisal-polyester biocomposites', Polymer Composites, 2002, 23, (2), pp. 164-170

8 Puglia, D., J, B., and J.M, K.: 'A Review of Natural Fibers Based Composites- Part II', Journal of Natural Fibers, 2005, 1, (3), pp. 23-65

9 Paul Ihom, A., and O Onah, D.: 'The Effects of Ukam (Cochlospermum Planchonii) Plant Fiber Variation on the Properties of Polyester Matrix Fiber Reinforced Composite' (2016. 2016)

10 Nekhlaoui, S., Essabir, H., Bensalah, M., Fassi-Fehri, O., Qaiss, A., and Bouhfid, R.: 'Fracture study of the composite using essential work of fracture method: PP–SEBS–g–MA/E1 clay', Materials & Design, 2014, 53, pp. 741-748

11 Echiye, O.G., Ashwe, A., Nyior, G., and Iortyer, H.A.: 'Dynamic Mechanical Analysis of Ukam (Cochlosperum Planchonii) Fibre Reinforced Polyester Composites'

12 Liu, L., Yu, J., Cheng, L., and Qu, W.: 'Mechanical properties of poly (butylene succinate)(PBS) biocomposites reinforced with surface modified jute fibre', Composites Part A: Applied Science and Manufacturing, 2009, 40, (5), pp. 669-674

13 Herrera-Franco, P., and Valadez-Gonzalez, A.: 'A study of the mechanical properties of short natural-fiber reinforced composites', Composites Part B: Engineering, 2005, 36, (8), pp. 597-608

14 Paul, S.A., Boudenne, A., Ibos, L., Candau, Y., Joseph, K., and Thomas, S.: 'Effect of fiber loading and chemical treatments on thermophysical properties of banana fiber/polypropylene commingled composite materials', Composites Part A: Applied Science and Manufacturing, 2008, 39, (9), pp. 1582-1588

15 Bledzki, A.K., Mamun, A.A., Lucka-Gabor, M., and Gutowski, V.S.: 'The Effects of Acetylation on Properties of Flax Fiber and its Polypropylene Composites', Express Polymer Letters, 2008, 2, (6), pp. 413-422

16 Jähn, A., Schröder, M., Füting, M., Schenzel, K., and Diepenbrock, W.: 'Characterization of alkali treated flax fibres by means of FT Raman spectroscopy and environmental scanning electron microscopy', Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 2002, 58, (10), pp. 2271-2279

17 Roe, P., and Ansell, M.P.: 'Jute-reinforced polyester composites', Journal of Materials Science, 1985, 20, (11), pp. 4015-4020