Effect of Cow bone and Groundnut Shell Reinforced in Epoxy Resin on the Mechanical Properties and Microstructure of the Composites

O. I. Rufai, G. I. Lawal, B. O. Bolasodun, S. I. Durowaye, J. O. Etoh

Abstract—It is an established fact that polymers have several physical limitations such as low stiffness and low resistance to impact on loading. Hence, polymers do not usually have requisite mechanical strength for application in various fields. The reinforcement by high strength fibers provides the polymer substantially enhanced mechanical properties and makes them more suitable for a large number of diverse applications. This research evaluates the effects of particulate Cow bone and Groundnut shell additions on the mechanical properties and microstructure of cow bone and groundnut shell reinforced epoxy composite in order to assess the possibility of using it as a material for engineering applications. Cow bone and groundnut shell particles reinforced with epoxy (CBRPC and GSRPC) was prepared by varying the cow bone and groundnut shell particles from 0-25 wt% with 5 wt% intervals. A Hybrid of the Cow bone and Groundnut shell (HGSCB) reinforce with epoxy was also prepared. The mechanical properties of the developed composites were investigated. Optical microscopy was used to examine the microstructure of the composites. The results revealed that mechanical properties did not increase uniformly with additions in filler but exhibited maximum properties at specific percentages of filler additions. From the Microscopic evaluation, it was discovered that homogeneity decreases with increase in % filler, this could be due to poor interfacial bonding.

Keywords—Groundnut shell reinforced polymer composite (GSRPC), Cow bone reinforced polymer composite (CBRPC), Hybrid of ground nutshell and cowbone (HGSCB).

I. INTRODUCTION

T is an established fact that polymers have several physical limitations such as low stiffness and low resistance to impact on loading. Hence, polymers do not usually have requisite mechanical strength for application in various fields. The reinforcement by high strength fibers provides the polymer substantially enhanced mechanical properties and makes them more suitable for a large number of diverse applications. Polymeric materials reinforced with synthetic fibers such as glass, carbon, and aramid provide advantages of high stiffness and strength to weight ratio as compared to conventional construction materials, i.e. wood, concrete, and steel. Despite these advantages, the widespread use of synthetic fiber-reinforced polymer composite has a tendency

O. I. Rufai is with the Department of Metallurgical and Materials Engineering, University of Lagos, Akoka, Lagos, Nigeria (phone: +2348088776521, e-mail: islamlolade@yahoo.com). to decline because of their high-initial costs, their use in nonefficient structural forms and most importantly their adverse environmental impact [1]. Hence there is an increased interest in the use of natural fibers as reinforcement in plastics to substitute conventional synthetic fibers in some structural applications and this has become one of the main concerns to study the potential of using natural fibers as reinforcement for polymers. The interest in natural fiber-reinforced polymer composite materials is rapidly growing both in terms of their industrial applications and fundamental research [2]. Natural fiber reinforced thermoplastic and thermoset composites constitute an important class of materials with wide variety of applications. The major types of thermosetting materials are epoxy resins and unsaturated polyesters (UP), phenolic resins (including phenol-formaldehyde ones), amino resins, (e.g. melamine-formaldehyde and urea-formaldehyde ones) and polyurethane [3].

Natural-fiber composites with thermoplastic and thermoset matrices have been used for various applications such as car manufacturing and suppliers for door panels, package trays, dashboards and interior parts. Natural fibers cultivation depends mainly on solar energy. For the natural fiber production, processing and extractions, relatively small amount of fossil fuel energy is required. While in comparison, the production of synthetic fiber depends mainly on fossil fuels and needs nearly ten times more energy as compared to natural fiber. As a result, the pollutant gas emissions to the environment from synthetic fiber production are significantly higher than that from the natural fiber production [4]. Naidu [5] examined the mechanical properties of metal matrix composites based on groundnut shell fiber. The results emphasized the increasing hardness value and reducing density of composites. It was seen that the hardness is decreasing with the increase in fiber length up to 20mm. However further increase in fiber length increased the micro hardness value. The tensile strength of the composite increased with increase in fiber length. An increase in fiber length led to a gradual increase in the tensile modulus of the coir fiber reinforced epoxy composite. The flexural strength increased with increase in fiber length and the resistance to impact loading of groundnut coir fiber reinforced epoxy composites improved with increase in fiber length. It was also concluded that hardness of Aluminum - Groundnut Shell Ash composites increased with increase of groundnut shell ash composition.

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Agunsoye [6] studied the effects of particulate cow bone additions on the mechanical properties and tribological behavior of cow bone reinforced polyethylene composite in order to assess the possibility of using it as a new material for engineering applications. The results revealed that tensile strength and the hardness values of the composite increased with increase in wt.% cow bone particles while the impact strength and rigidity decreased. The study also revealed that the additions of the particulate cow bone have the most significant main effect on the wear behavior of the composite while the interactions between load and time has no significant. Hence, cow bone particles could be used to improve the strength and wear properties of recycled low density polyethylene (RLDPE).

Isiaka [7] investigated the influence of cow bone particle size distribution on the mechanical properties of polyester matrix composites in order to consider the suitability of the materials as biomaterials. It was discovered that fine cow bone particles lead to improved strength while coarse particles lead to improved toughness. The results also showed that these materials are structurally compatible and are being developed from animal fiber based particle. It is expected to also aid the compatibility with the surface conditions as biomaterials. From the literatures, it is clear that natural fibres can be used to reinforce polymeric materials and get composite material with improve mechanical properties. In this research, the relationship between the microstructure and mechanical properties of groundnut shell, cow bone and a hybrid of groundnut shell and cow bone reinforced with epoxy is investigated in order to evaluate their uses as an engineering material and a biomaterial respectively.

II. EXPERIMENTAL DETAILS

A. Preparation of the Groundnut Shell, Cow Bone – Epoxy Matrix Composite

The unsaturated epoxy resin was weighed using an electronic weighing machine. A beaker is placed on the weighing machine and the epoxy is added gradually into the beaker, the weight indication is observed as more epoxy are continually added. Pouring of the epoxy into the beaker is stopped when the desired weight of epoxy necessary for a particular formulation is achieved. The beaker is removed from the weighing machine and is placed aside. The formulation used for the epoxy is:

X = P/100 x Basis

where X is weight of epoxy; P is percentage of epoxy; Basis: 50grams.

The two groundnut shell and cow bone particulates were weighed using an electronic weighing machine based on the weight percentage of the particulate to be added to the epoxy resin. A petridish is placed on the electronic weighing machine and the particulates are added gradually into the petridish, the weight indication is observed as more particulate are continually added. Pouring of the particulate into the petridish is stopped when the desired weight of particulate necessary for a particular formulation is achieved. The process is repeated for other weight fractions of particulate needed.

The petridish is removed from the weighing machine and is placed aside. The formulation used for the particulate is:

$$Y = Q/100 x Basis$$

where Y is the weight of the filler; Q is the percentage of filler; Basis: 50grams.

The hardener was weighed using an electronic weighing machine. A beaker is placed on the weighing machine and in it is placed a test tube the hardener is added gradually into the test tube with the help of a syringe, the weight indication is observed as more drops of hardener are continually added. Pouring of the hardener into the test tube is stopped when the desired weight of hardener necessary for a particular formulation is achieved. The test tube is removed from the weighing machine and is placed aside. The formulation used for the hardener is:

Basis: 50grams

Ratio: 2 parts of matrix (epoxy) to 1 part of hardener

$$Z = X/100 x Basis$$

where Z is the weight of the hardener X is the weight of epoxy

TA	BLE I			
VALUES OF WEIGHT OF EPOXY,	REINFORC	EMENT A	ND HARDEN	VEF
Percentage of filler (%)	X (g)	Y (g)	Z (g)	
0	50.0	0.0	25.00	
5	47.5	2.5	23.75	
10	45.0	5.0	22.50	
15	42.5	7.5	21.25	
20	40.0	10.0	20.00	
25	37.5	12.5	18.75	



Fig. 1 Groundnut shell particulate



Fig. 2 Cow bone particulate



Fig. 3 Mould filled with GSRPC samples



Fig. 4 Mould filled with CBRPC samples



Fig. 5 Mould filled with HRPC samples

The mixture was poured into a mold already coated with paper tape which acted as our poly vinyl alcohol (PVA) and allowed to cure. This procedure is repeated for all samples produced with changes in the particular percentage. After curing the samples are stripped from the mold.

B. Mechanical Characterization

The tensile testing was performed using an Instron universal testing machine operated at a cross head speed of 10mm/min. The tensile test specimen preparation and testing procedures were conducted in accordance with the American Standard testing and measurement, method D412 (ASTM D412 1983), using dumbbell test piece. Each tensile specimen is positioned in the instron universal tester and then subjected to tensile load, as the specimen stretches the computer generates graph as well as all the desired parameters until the specimen fractures. A graph of load versus extension is plotted automatically by the tester and various property of the specimen determined are; tensile strength, tensile strain, modulus, tensile strain at break.

Three point flexural testing were conducted using testometric testing machine with serial number 25257 and capacity M500-25KN. The flexural test was carried according to ASTM D 7264 at a cross-head speed of 20mm/min, maintaining a span of 100mm. This test was conducted at room temperature. The flexural test specimens were of 120 X

50 X 10 mm. The testometric machine was used to carry out the three point bending flexural test on the polymeric material composite at different filler content at 0, 5, 10, 15, 20, and 25% of filler content.

The sample were cut in dimension, their initial weights were taken with the aid of an electronic weighing scale. Each of the samples was immersed in a beaker containing water and the new weights of the samples were recorded.

Water absorption which is a measure of material ability to absorb moisture (water) was obtained by immersing the specimen for 72 hours. After immersion, the surfaces of the specimens were cleaned dry and weighed immediately to measure their wet weight. The increase in weight is recorded as percentage gained and is expressed by;

 $\frac{finalweight - initialweight}{initialweight} \times 100$

The hardness property of samples produced was determined using Brinell hardness tester. The specification of the Brinell hardness machine is ball indenter of diameter 20mm and the maximum load of 4000N. The hardness test was carried out on the composite material at different filler percentage at 0, 5, 10, 15, 20 and 25% of filler content.

Impact test is a standard method of determining the impact resistance of materials. An arm held at a specific height (constant potential energy) is released. The arm hits the sample and breaks it. From the energy absorbed by the sample, its impact energy is determined. A notched sample is generally used to determine impact energy and notch sensitivity. Impact test is used to study the toughness of a material. A material's toughness is a factor of its ability to absorb energy during plastic deformation.

FTIR is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivit y or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. FTIR offers quantitative and qualitative analysis for organic and inorganic samples. FTIR identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The peaks in IR spectrum reveal the functional groups present in the molecule. An IR peak is characterized by its stretching frequency, intensity (strong or weak) and also the shape of the peak (broad or narrow).The machine used was an FTIR-8400S series.

C. Optical Microscopy

The optical microscope often referred to as the "light microscope", is a type of microscope which uses visible light and a system of lenses to magnify images of small samples. The image from an optical microscope can be captured by normal light-sensitive cameras to generate a micrograph.

III. RESULTS AND DISCUSSION



Fig. 6 Fourier transform infrared spectroscopy of epoxy resin

From Fig. 6, the highest peak of the epoxy resin was observed at 1510.31 cm⁻¹. This is within the carbonyl region (1500-1700 cm⁻¹) which is typical for epoxy resin as they show prominent C=O peaks in this region.

 TABLE II

 Result of Mechanical Tests on 0% Cow Bone, Groundnut Shell and Hybrid Reinforcement

Bending strength	Young's	Impact	Brinell	Percentag
at peak/break	modulus	strength	Hardness	e water
(MPa)	(MPa)	(Joules)	(BHN)	absorption
57.576	227.049	6.53	54.8	0.156
57.576	227.049	6.53	54.8	0.156
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	Bending strength at peak/break (MPa) 57.576 57.576 57.576	Bending strength at peak/break Young's modulus (MPa) (MPa) 57.576 227.049 57.576 227.049 57.576 227.049	Bending strength at peak/break Young's modulus Impact strength (Joules) 57.576 227.049 6.53 57.576 227.049 6.53 57.576 227.049 6.53 57.576 227.049 6.53	Bending strength at peak/break Young's modulus Impact strength Brinell Hardness (MPa) (MPa) (Joules) (BHN) 57.576 227.049 6.53 54.8 57.576 227.049 6.53 54.8 57.576 227.049 6.53 54.8 57.576 227.049 6.53 54.8

TABLE III Result of Mechanical Tests on 5% Cow Bone, Groundnut Shell and Hybrid Reinforcement

Reinforcement	Bending	Young's	Impact	Brinell	Percentage
	strength at	modulus	strength	Hardness	water
	break (MPa)	(MPa)	(Joules)	(BHN)	absorption
Cow bone	44.064	195.92	5.31	76.2	0.054
Groundnut shell	36.768	212.81	4.75	63.2	0.169
Hybrid	57.036	275.71	6.12	80.8	0.037

TABLE IV Result of Mechanical Tests on 10% Cow Bone, Groundnut Shell and Hybrid Reinforcement

Reinforcement	Bending	Young's	Impact	Brinell	Percentage	
	strength at	modulus	strength	Hardness	water	
	break (MPa)	(MPa)	(Joules)	(BHN)	absorption	
Cow bone	33.432	281.44	5.36	65.5	0.616	
Groundnut shell	25.512	222.19	4.89	60.6	0.096	
Hvbrid	28.164	222.99	8.93	52.2	0.121	

TABLE V Result of Mechanical Tests on 15% Cow Bone, Groundnut Shell and Hybrid Reinforcement

Reinforcement	Bending	Young's	Impact	Brinell	Percentage
	strength at	modulus	strength	Hardness	water
	break (MPa)	(MPa)	(Joules)	(BHN)	absorption
Cow bone	28.176	247.16	5.44	51.3	0.066
Groundnut shell	31.116	191.55	5.32	43.9	0.113
Hybrid	24.228	139	16.3	46.4	0.107

 TABLE VI

 Result of Mechanical Tests on 20% Cow Bone, Groundnut Shell

AND HYBRID REINFORCEMENT						
Reinforcement	Bending	Young's	Impact	Brinell	Percentage	
	strength at	modulus	strength	Hardness	water	
	break (MPa)	(MPa)	(Joules)	(BHN)	absorption	
Cow bone	40.704	298.09	5.31	49.2	0.151	
Groundnut shell	31.704	374.06	5.44	47.5	0.093	
Hybrid	32.208	89.04	12.2	48.3	0.043	

TABLE VII	

RESULT OF MECHANICAL TESTS ON 25% COW BONE, GROUNDNUT SHELL AND HYBRID REINFORCEMENT

Reinforcement	Bending	Young's	Impact	Brinell	Percentage	
	strength at	modulus	strength	Hardness	water	
	break (MPa)	(MPa)	(Joules)	(BHN)	absorption	
Cow bone	19.848	227.030	4.89	43.9	0.214	
Groundnut shell	24.636	194.510	8.43	48.6	0.193	
Hybrid	1.944	10.978	10.3	21.8	0.450	

From Fig. 7 the bending strength at break of all reinforcements fell from 0-5%, continually dropped up to 15% for cow bone, rose at 20% and decreased again. It dropped because the increase in the weight percentage of filler reduced the deformability of the matrix, reducing the ductility of the composite thereby forming a weak structure. As the amount of reinforcement increases there is reduction in the total surface area available for matrix-filler interaction. The bending strength for groundnut shell reduced uniformly to 15%, rose at 20% and reduced again. The bending strength is highest at 5% for cow bone, groundnut shell and hybrid.



Fig. 7 Bending strength at break against filler concentration



Fig. 8 Young's modulus against filler concentration

The Young's modulus for the cow bone and groundnut shell showed a wavelike pattern while the hybrid decreased steadily in Fig. 8. The ultimate strength of a composite depends on the weakest fracture path throughout the material. Hard particles affect the strength in two ways. One is the weakening effect due to the stress concentration they cause, and another is the reinforcing effect since they may serve as barriers to crack growth [8]. The strength of composites is determined by the fracture behaviors which are associated with the extreme values of such parameters as interface adhesion, stress concentration and defect size/spatial distributions. Thus, the load-bearing capacity of a particulate composite depends on the strength of the weakest path throughout the microstructure, rather than the statistically averaged values of the microstructure parameters.

The tensile strength is highest at 20% of groundnut shell, this could be due to absence of void or porosity and good interfacial bond while the lowest is at 25% of hybrid, which could be due to poor stress transfer between the particlematrix interface.



Fig. 9 Brinell hardness (BHN) against filler concentration

From Fig. 9, it was noticed that both the highest and lowest hardness was exhibited by the same reinforcement (hybrid) at 5 and 25% respectively while other reinforcements such as cow bone and groundnut shell both showed undulating patterns. The unpredictable pattern of the hardness may be probed to be caused by the poor interfacial bonding or surface adhesion of the fillers and epoxy resin.



Fig. 10 Impact strength against filler concentration

Fig. 10 shows the amount of energy the samples can absorb prior to fracture. It was observed that the cow bone and hybrid samples can only absorb maximum energy at 15% filler concentration. However, the maximum amount of energy absorbed was by the hybrid at 15% reinforcement. The impact strength of the groundnut shell increased uniformly as the filler concentration was increased.

For the cow bone and hybrid, the impact strength reduced after 15%. This may be due to the reduction of elasticity of the

material due to filler addition and thereby reducing the deformability of matrix. An increase in concentration of filler reduces the ability of matrix to absorb energy and thereby reducing the toughness, so impact strength decreases.

There is no explanation to this but it could be attributed to discontinuity of matrix phase in the composite.



Fig. 11 Percentage water absorption against filler concentration

The percentage of water absorption for all the composites is shown in Fig. 11. It can be seen that increasing the filler content, the water absorption becomes quite unpredictable although all the composites are shown to be hydrophilic. The poor wettability and interfacial adhesion between the reinforcements and polyester resin are attributed to the hydrophilic nature of the fillers [9]. As shown in the chart below, it is being noticed that the most hydrophilic of all is the 10% cow bone reinforcement, while the least hydrophilic of all is the 5% hybrid reinforcement.

From the micrographs (Figs. 12-27), it can be seen that homogeneity decreases with increase in % filler. This could be due to poor interfacial bonding between the filler and the reinforcement. More research needs to be done in the aspect of improving the interfacial bonding so as to have an improved microstructural result.



Fig. 12 Micrograph of control sample



Fig. 13 Micrograph of 5% cow bone filler

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Fig. 14 Micrograph of 5% groundnut shell filler



Fig. 15 Micrograph of 5% hybrid filler



Fig. 16 Micrograph of 10% cow bone filler



Fig. 17 Micrograph of 10% groundnut shell filler



Fig. 18 Micrograph of 10% hybrid filler



Fig. 19 Micrograph of 15% cow bone filler



Fig. 20 Micrograph of 15% groundnut shell filler



Fig. 21 Micrograph of 15% hybrid filler



Fig. 22 Micrograph of 20% cow bone filler



Fig. 23 Micrograph of 20% groundnut shell filler



Fig. 24 Micrograph of 20% hybrid filler



Fig. 25 Micrograph of 25% cow bone filler



Fig. 26 Micrograph of 25% groundnut shell filler



Fig. 27 Micrograph of 25% hybrid filler

IV. CONCLUSION

It was seen that the mechanical properties of epoxy can be greatly improved by these reinforcements. From Fig. 7 it can be seen that the hybrid sample of 5% reinforcement showed the highest resistance before shattering relative to other samples the flexural test was performed on. This implies that the hybrid reinforcement of 5% can be used in place of the pure epoxy for applications where flexibility is a major consideration.

From Fig. 8, it can be seen that the groundnut shell sample of 20% reinforcement showed the highest stiffness before shattering relative to other samples the tensile test was performed on. Therefore, the groundnut shell reinforcement of 20% can be used in place of pure epoxy where stiffness is a major concern.

From Fig. 9 it can be seen that the hybrid sample of 5% reinforcement showed to have the highest surface hardness compared to all other samples being tested. This implies that the hybrid reinforcement of 5% can be used in place of the pure epoxy for applications where surface hardness is a major consideration.

From Fig. 10 it can be seen that the hybrid sample of 15% reinforcement showed to absorb the highest amount of energy before shattering relative to other samples the impact test was performed on. Therefore, the hybrid reinforcement of 15% can be used in place of pure epoxy where impact strength is a major concern.

From Figs. 7-11 it is seen that as the filler concentration increased, the shapes of the reinforcement became larger, changed from spherically shaped to irregularly shaped and they became more closely packed.

The microstructural analysis of all the test samples showed decrease in homogeneity with increasing %filler addition. This could be due to poor interfacial bonding.

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