

# Development of bone particulate reinforced epoxy composite for biomedical application

## Abstract

Mechanical properties of particulate goat bone reinforced epoxy composite were investigated in this research. This was aimed at assessing the suitability of the developed composites for biomedical applications. The combination of the two materials for the development of the composites was targeted to meet good surface compatibility in addition to the structural compatibility needed as biomaterials. The composites were developed by compounding the matrix and the reinforcement in predetermined proportions and were fabricated by open mould technique. Mechanical properties such as tensile, flexural and hardness properties as well as wear behaviour were tested. From the results, XRF analysis showed that the main elements present in the goat bone ash are calcium and phosphorus with 71.6 and 16.6% contents, respectively. It was observed that improved mechanical properties were obtained for higher wt. percent reinforced composites from 16-20wt. percent compared to lower wt. percent composites from 2-14wt. percent. Bone particulate reinforced epoxy composite developed from 16wt. percent gave the best tensile and flexural properties and also possesses good and favourable hardness properties. However, improved wear resistance was observed at low wt. percent fraction of the reinforcement within 2-6wt. percent. Examination of the fractured surfaces by Scanning Electron Microscopy showed that the particulates were well dispersed within the matrix and the reason for the enhancement in the properties. Sample with optimum performance was 16 wt. percent bone particulate reinforced epoxy composite which has the best combination of mechanical and wear properties. The combination of good properties obtained made the developed composite to be suitable for biomedical application and, therefore, selected as the best composite material for biomedical application having met the structural conditions necessary.

**Keywords:** composites, biomaterial, epoxy, goat bone, mechanical properties, wear property

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Oladele IO,<sup>1</sup> Isola BA<sup>1</sup>

<sup>1</sup>Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, Nigeria.

**Correspondence:** Oladele IO, Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, Nigeria, Email wolesuccess2000@yahoo.com

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## Introduction

A progressive deterioration of all tissues with age is the major contributor to the need for spare parts for the body. Bone tissue is susceptible to fracture as a result of trauma, pathology and resorption Rabie et al.<sup>1</sup> This effect is especially severe in women due to the hormonal changes associated with menopause. Bone density decreases because bone-growing cells become progressively less productive in making new bone and repairing micro-fractures. The lower density greatly deteriorates the strength of bones and an unfortunate consequence is that many old people fracture their hips or have collapsed vertebrae and spinal problems Hench et al.<sup>2</sup>

Bone fixation and repair devices traditionally are fabricated with metals and used clinically Goodrich et al.<sup>3</sup> Stainless steel, titanium and its alloys have been employed for the majority of fracture fixation treatments Eglin & Alini et al.<sup>4</sup> However these metallic devices and implants are not biodegradable, and often require a second surgery in order to remove these from the body. This not only increases the hospitalization time and health care cost but also elevates chances of infection and complications. Also, due to the mismatch between the mechanical properties of these devices and the natural bone, mechanical forces and loads are retained by implants and are not transferred to the healing bone which results in unwanted bone resorption and implant loosening Sumner et al.<sup>5</sup>

Metals are known for high strength, ductility, and resistance

to wear, however, the shortcomings of many metals includes; low biocompatibility, corrosion, too high stiffness compared to tissues, high density, and release of metal ions which may cause allergic tissue reactions. Ceramics are known for their good biocompatibility, corrosion resistance and high compression resistance, nevertheless, the drawbacks of ceramics include; brittleness, low fracture strength, difficult to fabricate, low mechanical reliability, lack of resilience and high density. The above said complexities give way for the next generation bone implants that are polymeric based which have better biocompatibility. One of the substitute material based that are gaining attention is the utilization of abundantly available natural materials as reinforcement materials in polymer matrix composite. Goat bone being a natural material from animal source is expected to have good surface compatibility in addition to the structural compatibility needed as biomaterials. Composite is a material comprised of two or more metal, polymer or ceramic structures which are separated by an interface. Composite materials have been widely used for a long time in innovative technological applications due to their superior mechanical properties Manivasagam et al.<sup>6</sup>

Many matrix and reinforcement components of composite materials have been tried by several researchers in tissue engineering to advance the mechanical features, biological functions and to deliver special molecules. Biocompatible polymers have been mostly applied as matrix for composite materials associated with ceramic fillers in tissue engineering. Although ceramics are generally stiff and

brittle materials, polymers are known to be flexible and exhibit low mechanical strength and stiffness. Composites aim to combine the properties of both materials for medical applications Park et al.<sup>7</sup>

The interest in natural fiber-reinforced polymer composite materials is rapidly growing both in terms of their industrial applications and fundamental research. They are renewable, cheap, completely or partially recyclable, and biodegradable. Their availability, renewability, low density, and price as well as satisfactory mechanical properties make them an attractive ecological alternative to glass, carbon and man-made fibers used for the manufacturing of composites Murali et al.<sup>8</sup>

Biomaterials are materials of natural or man-made origin that are used to direct, supplement, or replace the functions of living tissues of the human body Black et al.<sup>9</sup> They involve complete or part of a living organism or biomedical device which performs, augments or replace any natural function Hermawan et al.<sup>10</sup> Nowadays, biomaterials are commonly used in various medical devices and systems; synthetic skin; drug delivery systems; tissue cultures; hybrid organs; synthetic blood vessels; artificial hearts; cardiac pacemakers; screws, plates, wires and pins for bone treatments; total artificial joint implants; skull reconstruction; dental and maxillofacial applications.

Researchers have begun to focus attention on natural fiber composites (i.e., bio composites) which are composed of natural or synthetic resins reinforced with natural fibers. Oladele et al.<sup>11</sup> 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 since they are developed from animal fiber based particle, it is expected to also aid the compatibility with the surface conditions as biomaterials. Agunsoye et al.<sup>15</sup> 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. percent of the 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 effect. Hence, bone particles could be used to improve the strength and wear properties of recycled low density polyethylene (RLDPE).

A lot of research has been done on natural fiber reinforced polymer composites but researches on goat bone based polymer composites have not been exploited. Against this background, the present research was undertaken with an objective to explore the potential of goat bone polymer composites as biomedical materials.

## Materials and method

### Materials

The main materials for the investigation are goat bone which serves as the particulate reinforcement phase and Epoxy resin and hardener which serves as the matrix phase.

### Method

**Preparation of goat bone particulate:** The goat bone was scraped

to remove meat remnants, washed thoroughly to remove any oil and then sun dried for 2 weeks. The goat bone were calcined in the furnace at 400°C for 4 hours to eliminate any protein present and then cooled in air. The calcined bone was reduced to smaller sizes with the use of hammer prior to pulverizing in the ball mill. The bone were pulverized into finer particles with a ball mill followed by sieving using the sieve shaker to obtain the required grain size of 75µm undersize particulate.

**Composites production:** The composite was produced using the open mould technique Oladele et al.<sup>12</sup> A mixing ratio of 2:1 (epoxy: hardener) was selected based on the epoxy-hardener manufacturer's instruction. Bone particulate was varied in a predetermined proportion of 2, 4, 6, 8, 10, 12, 14, 16, 18 and 20 wt.%. The three moulds used for the fabrication were for tensile, flexural and wear specifications. The samples were left to cure in the mould for four hours after which it is removed and dried in air for 28 days before testing.

**Mechanical test:** Three tests were performed for each composite composition to guarantee reliability of the data generated from the average values that were used as the representative values Oladele et al.<sup>13</sup>

**Tensile Test:** Tensile test was carried out in accordance to American Standard Testing and Measurement Method D412 (ASTM D412 1983) on Instron Universal testing machine. The tests were carried out using the displacement control at a rate of 5mm/min. Composite samples with 3mm thick and of gauge length 150mm were used. Three identical samples were tested for each weight fraction from where the average values were used as the representative values.

**Flexural test:** Three point bend tests were performed in accordance to ASTM D 790M to measure flexural properties using Instron Universal testing machine Oladele et al.<sup>14</sup> The tests were carried out using the displacement control at a rate of 10mm/min. The samples were of 150x50x3mm. Three samples were tested for each weight fraction used and the average values were taken to represent the actual values.

**Hardness test:** Rockwell hardness test was conducted on the specimen using a standard Rockwell hardness tester, Indentec, AK4150. The sample were mounted on the hardness tester support and then indented with a load of about 60Kgf for 10 seconds. The hardness value was displayed on the digital surface of the machine that shows the readings. Each sample was indented in three places and the average value was used as the representative values.

**Wear Test:** The wear resistance test was carried out with Taber Abrasors, Model ISE-AO16. The standard load used was 500g and revolution of 150RPM, center hole of 10mm was made on the sample, so as to fix the test piece on the machine. The samples were measured using an analytical weighing balance to take the initial weight of the samples. The sample was secured to the instrument platform, which is a motor driven at a fixed speed. Thereafter, the weight was measured as the final value and the difference between the initial and the final value was noted and recorded against each samples. The average values were used as the representative value Oladele et al.<sup>16</sup>

**X-ray Fluorescence spectroscopy (XRF):** X-ray fluorescence was also carried out on the bone particles to determine the elemental chemical composition of the goat bone particulate. SKYRAY Instrument was used with EDX3600B X-Ray fluorescence spectrometer been applied to conduct fast and accurate analysis. The instrument was calibrated using pure silver sample before being used.

**Microstructural characterization:** The fractured surfaces of the pro-

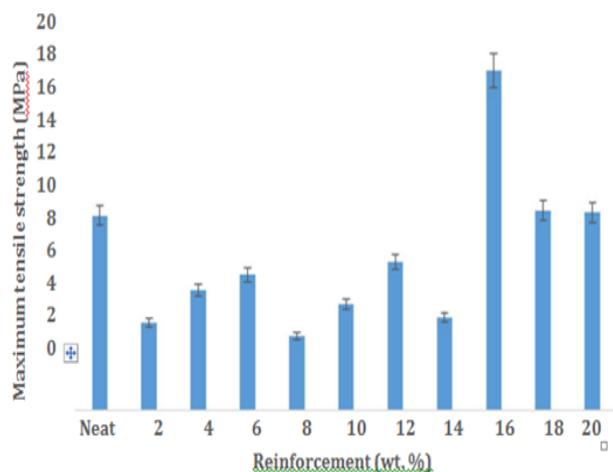
duced composite samples were examined by Phenom ProX scanning electron microscope (SEM) operated at 15Kv. Samples were mounted on to aluminium stubs with carbon tape and then sputter coated with platinum and palladium using Quorum coating machine (Q150res) to make them conductive prior to SEM observation.

## Result and discussion

The XRF analysis showed that the main elements present in goat bone ash are calcium and phosphorus with 71.6% and 16.6%, respectively.

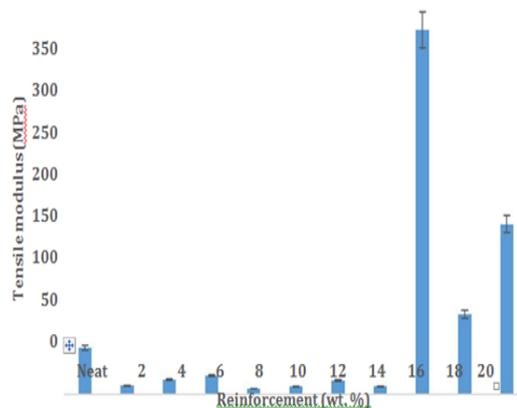
### Mechanical behaviour

Figure 1 shows the variation of tensile strength with reinforcement and the neat sample. Three distinct stages were noticed where different trends were observed. From 2-6wt. percent, the strength tends to increase as the reinforcement content increases while from 8-14wt. percent, similar trend was observed but with a decrease in tensile strength compared to the neat sample. These first two stages were characterized by decrease in tensile strength compared to the neat sample. This decrease in tensile strength might be due to either poor adhesion or direct contact of particles and void formation Muthuel. However, from 16-20wt. percent, contrast trend was noticed where the strength tends to decrease as the reinforcement content increases and higher enhancement of the tensile strength was obtain in this stage. It was observed that the sample with 16wt. percent reinforcement has the highest tensile strength of 17.30MPa followed by 18wt. percent with a value of 10.16. This culminated to 74.6% enhancement in tensile strength for the sample with 16wt. percent reinforcement compared to the neat sample.



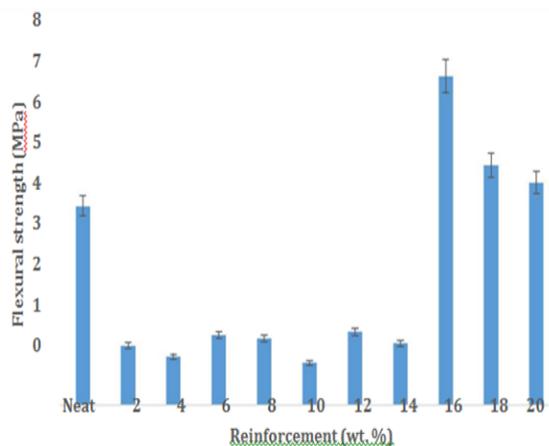
**Figure 1** Variation of tensile strength with reinforcement and the neat sample.

The variation of tensile modulus with reinforcement and the neat sample was presented in Figure 2. The results show that the modulus was enhanced for the composite sample containing 16-20 wt. percent reinforcement. It was observed that the sample with 16wt. percent reinforcement has the highest tensile modulus with a value of 298.93MPa followed by 20wt. percent with a value of 139.66MPa. It can also be seen that the tensile modulus of the composite follow similar trend with the tensile strength in Figure 1. Similar effect of cow bone ash on tensile properties has been observed by Oladele et al.<sup>16</sup> in the case of bone ash and bone particulate reinforced polyester composites for biomedical applications.



**Figure 2** Variation of tensile modulus with reinforcement and the neat sample.

Figure 3, shows the variation of flexural strength with reinforcement and the neat sample. The results showed that the flexural strength of the composites from 2-14wt. percent was lower than that of the neat sample while that of 16-20wt. percent was higher but in a decreasing order. The decrease in flexural strength within 2-14wt. percent may be due to low amount of reinforcement, poor interfacial adhesion between the bone particulates and the epoxy matrix or non-uniform dispersion of the particulate within the epoxy that led to lower resistance to the applied flexural force leading to quick rupture.

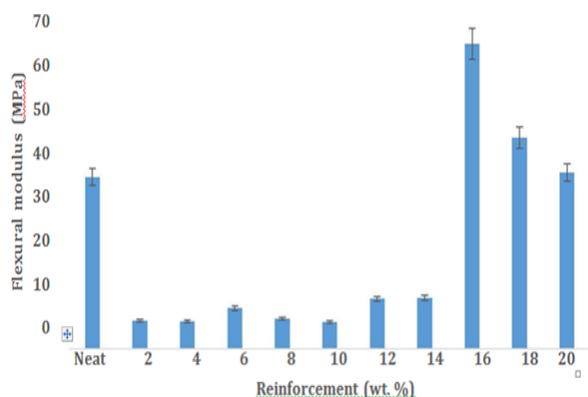


**Figure 3** Variation of flexural strength with reinforcement and the neat sample.

Nevertheless, it was observed from the results that, the sample with 16wt. percent reinforcement has the highest flexural strength with a value of 7.07MPa which amounted to 65% enhancement compared to the neat sample followed by the sample with 18wt. percent reinforcement with a value of 5.16MPa.

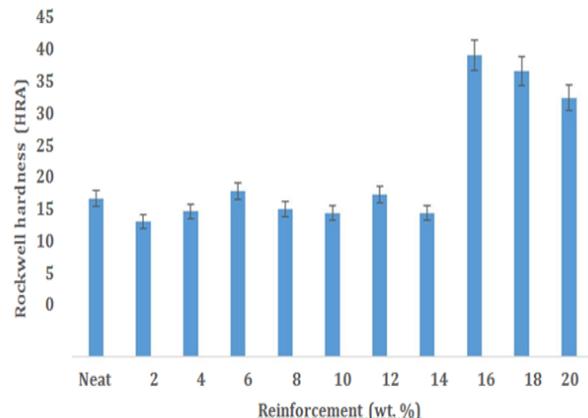
From Figure 4, which shows the variation of flexural modulus with reinforcement and the neat sample, it was observed that the sample with 16wt. percent reinforcement has the highest flexural modulus with a value of 61.49MPa that culminated to 77.6% enhancement in flexural modulus compared to the neat sample followed by the sample with 18wt. percent reinforcement which has a value of 42.59MPa and a 23% enhancement. The response of the materials to flexural modulus was similar to that of the flexural strength. It was observed

from the results that the properties were enhanced from 16-20wt. percent reinforcement addition in a decreasing order.



**Figure 4** Variation of flexural modulus with reinforcement and the neat sample.

The hardness values of the composites are presented in Figure 5. Similar response mode to other mechanical properties was observed with a slight difference. Here, it was observed that some samples within 2-14wt. percent were seen to possess marginal improvement in hardness property compared to the neat sample. Still, 16-20wt. percent range possess better improved hardness property. Sample with 16wt. percent reinforcement has the highest hardness value of 39.7HRA which may be due to uniform dispersion of particles and good bonding strength followed by the sample with 18wt. percent reinforcement with a value of 37.6HRA.



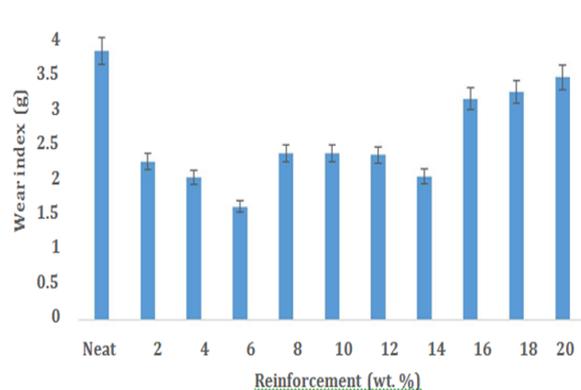
**Figure 5** Variation of Rockwell hardness with reinforcement and the neat sample.

The enhanced mechanical properties, particularly within 16-20wt. percent reinforcement content may be due to one or combinations of the following factors which are; higher weight reinforcement content added, good blending and wetting between the goat particulate and the epoxy matrix, reduced or absence of voids or sound interfacial bonding strength that allow easy transfer of load from the matrix to the reinforcement and, the presence of calcium and phosphorous in higher amount.

### Wear behaviour

The behaviour of the materials under the influence of load that tends to wear them is as presented in Figure 6. Three stages as

observed in mechanical properties were also obvious here but with a difference. The best wear resistance was observed within the first stage where the resistance increases as the reinforcement content increases. Also, similar trend was obtained in the second stage but with lesser wear resistance when compared to the first stage. The third stage possess the least wear resistance and in an inverse order. Though, all the developed composites possess better wear resistance than the neat. In order words, the goat bone ash reduced the wear rate of the composite. From the results, 6wt. percent possess the best wear resistance with wear index of 1.39g followed by 4wt. percent with a wear index value of 1.75g compared to the neat sample with a wear index of 3.29g.



**Figure 6** Variation of wear index with reinforcement and the neat sample.

Considering the stage with the best mechanical properties and sample from 16wt. percent that was the best among them, this sample was the best also with the highest wear resistance with a value of 2.71g. This amounted to about 18% enhancement, therefore, it implies that sample developed from 16wt. percent is the best for this application.

**Table 1** Variation in percent composition and weight of produced samples

Composition (%)	Reinforcement (g)	Epoxy (g)	Hardener(g)
Neat	—	220	110
2	6.6	216	108
4	13.2	211	106
6	19.8	206	103
8	26.4	202	101
10	33	198	99
12	39.6	194	97
14	46.2	190	95
16	52.8	185	92
18	59.4	180	90
20	66	176	88

### Microstructure

Plate 1: Scanning electron microscope image of the fractured surface for (a). Neat epoxy (b). 8wt. percent and (c). 16wt. percent bone particulates reinforced epoxy composite showing particles dispersed in the epoxy matrix.

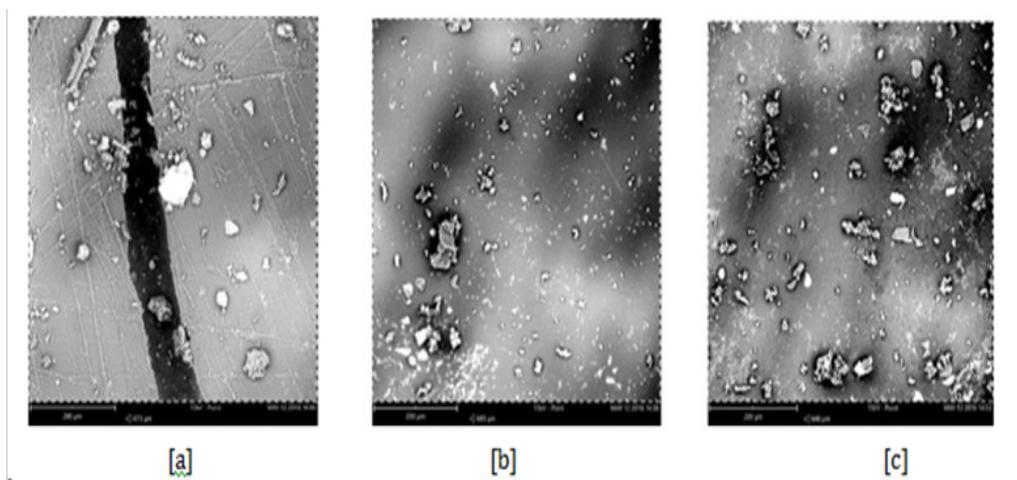
Plates 1(a-c) show SEM micrograph for the neat epoxy material produced and sample from the two broad categories (2-14 and 16-20wt. percent) of the developed composites where there is reduced and improved properties, respectively.

Plate 1 (a) revealed the fractured surface from the neat sample the presence of some voids due to entrapped air and crack was seen. This observed crack was due to the insufficient strength from the matrix to bear the applied load which led to crack propagation with the continuous application of load.

Plate 1(b) shows SEM micrograph for 8wt. percent composite sample that represent the categories of samples with reduced strength compared to the neat. The whitish part indicates the bone

particulates while the dark part indicates the epoxy matrix. Though, crack propagation was not noticed due the possibility of the goat bone particulate blocking and hindering the propagation of such failure mode, however, the reinforcement content were small and were not uniformly distributed. This may be part of the reasons for the weak mechanical properties obtained.

Plate 1 (c) shows SEM micrograph for the 16wt. percent bone particulate reinforced epoxy composite produced that represent the improved composites categories. It was seen from the image that the reinforcement were present in high amount and were well dispersed. This was part of the reasons for the enhancement in properties obtained.



**Plate 1** Scanning electron microscope image of the fractured surface for (A). Neat epoxy (B). 8wt.% and (C). 16 wt.% bone particulates reinforced epoxy composite showing particles dispersed in the epoxy matrix.

## Conclusion

The work has been carried out to investigate the possibility of exploiting goat bone as reinforcement in epoxy matrix for biomedical application. This animal bone is from one of the highly consumed meat in Nigeria which has been regarded as waste. From the research, the following conclusions have been made.

- i. The presence of calcium in higher proportion of about 71.6% in calcined particulate goat bone as a natural reinforcement material confirm its suitability as a good reinforcement material in epoxy matrix for the development of composites for biomedical applications.
- ii. Reinforcement of epoxy with 16-20wt. percent particulate goat bone gave the best mechanical properties with 16wt. percent emerging as the best among them.
- iii. Wear resistance was highly enhanced within 2-6wt. percent particulate goat bone reinforcement with 6wt. percent evolving as the best weight fraction.
- iv. Composite sample with best combination of mechanical and wear properties was 16wt. percent goat bone particulate reinforced epoxy matrix composite. This feat positioned the sample as the most suitable material for biomedical application having met the structural conditions necessary.

## Acknowledgements

None.

## Conflict of interest

The author declares no conflict of interest.

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