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COMPARATIVE STUDY OF THE REINFORCEMENT EFFICIENCY OF COW BONE AND COW BONE ASH IN POLYESTER MATRIX COMPOSITES FOR BIOMEDICAL APPLICATIONS

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Abstract: *This work was carried out to study comparatively the reinforcement efficiency of cow bone and cow bone ash particles in polyester matrix composites in order to consider the suitability of the materials as biomaterial. Cow bone was procured from an abattoir, washed with water and sun dried for 4 weeks and a portion was burnt. The bone ashes and un-burnt bone portions were pulverized separately using the ball mill. Sieve analysis was carried out on the pulverized bone ash and bone particles into particle sizes of 75µm, 106µm and 300µm. Composite materials were developed by casting into tensile and flexural tests moulds using pre-determined proportions of 2, 4, 6, and 8 wt % for both the cow bone and cow bone ash. The samples after curing were striped from the moulds and were allowed to further cure at room temperature for 3 weeks before tensile and flexural tests were performed on them. The tensile test results showed that bone particles reinforced composites have the best tensile properties except in Modulus of elasticity where bone ash particles reinforced composite samples have higher values while the flexural test showed that bone ash particle reinforced samples has the best flexural properties.*
Keywords: *cow bone and ash, polyester, composites, mechanical properties, biomedical*

1. INTRODUCTION

The development of materials for any replacement application should be based on the understanding of the structure to be substituted. This is true in many fields, but particularly exigent in substitution medicine. The demands upon the material properties largely depend on the site of application and the function it has to restore. Ideally, a replacement material should mimic the living tissue from a mechanical, chemical, biological and functional point of view.

Mineralised tissues such as bones, tooth and shells have attracted considerable interest as natural anisotropic composite structures with adequate mechanical properties. In fact, nature is and will continue to be the best materials scientist ever. Who better than nature can design complex structures and control the intricate phenomena (processing routes) that lead to the final shape and structure (from the macro to the ultra-structural level) of living creatures? Who can combine biological and physico-chemical mechanisms in such a way that can arrive to ideal structure–properties relationships? Who, else than nature, can really design smart structural components that respond, in-situ, to exterior stimulus adapting the microstructure and correspondent properties? In the described line of thinking, mineralized tissues and biomineralization processes are good examples to learn from for the materials scientist of the future. This is especially true for engineers that want to develop composites to replace mineralized tissues [1].

Structurally, the bone matrix consists of type I collagen fibres reinforced by hydroxyapatite nano-crystals precipitated along the collagen fibres [2]. The mineral part is responsible for the stiffness whereas the collagen is responsible for its flexibility. A demineralised bone becomes very flexible being easily twisted, whereas a bone without collagen is very brittle [3].

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The major component of compact bone is called the osteon. Organised in concentric lamellar matrix, the osteons create cylindrical conduits known as Haversian canals, which provide access for the circulatory and nervous systems. The capillaries within the Haversian canals originate from arteries and veins within the marrow cavity. It is known that the structure of bones is continuously adapted to the stresses applied to it [4]. Thus, any substitution implant material should be compatible and not disturb significantly the stress environment of the surrounding living tissue [5]. Materials that are biocompatible are called biomaterials, and the biocompatibility is a descriptive term which indicates the ability of a material to perform with an appropriate host response in a specific application. This definition has been further extended by Wintermantel and Mayer [6], and they distinguished between surface and structural compatibility of an implant [7]. Surface compatibility means the chemical, biological, and physical suitability of an implant surface to the host tissues while structural compatibility is the optimal adaptation to the mechanical behavior of the host tissues. From all the above discussion it becomes evident how difficult it is to design and produce materials that can be used on replacement and fixation of bones or for filling bone defects, especially those that must work under load bearing conditions. That explains why synthetic materials are only about 10% of the bone grafting market, where autografts and allografts still reign. Biomaterials are finding increase use in problem areas like replacement of deceased or damage parts.

The use of by-products as reinforcement is a modern technology for producing relatively inexpensive materials of high strength from suitable homogeneous matrix bases. Therefore, cow bones and its ash were used in this research. These materials have constitutes a waste of natural resources since the physical and mechanical properties are yet to be effectively brought to the attention of modern designers. Cow bones which are obtainable from slaughtered cows in abattoirs are usually burnt or sold to feed mill for the production of animal feeds. However, this by-product in some cases are left to waste but can be used as reinforcement in polymer to produce composite materials for biomedical use due to its good mechanical properties[8]. This work was carried out to investigate comparatively the mechanical properties of cow bone particles and cow bone ash particles. This was done in order to study the effect of biocompatibilization treatment on the mechanical properties of the developed composites.

2. MATERIALS AND METHODS

The main materials that were used for this work are as follows: unsaturated polyester resin, cow bones, methyl ethyl ketone peroxide (MEKP) used as the catalyst, cobalt 2% in solution used as the accelerator, polyvinyl acetate used as the mould releasing agent, and ethanol used as a cleaning agent.

***2.1. Material Preparation**. The cow bone was procured from the abattoir, washed with water so as to remove the dirty particles that might have been stuck to the bone, and sun dried for 4 weeks. The bones were separated into two portions; one portion is burnt into ashes while the other portion was crushed with hammer. The two portions were pulverized separately using Denver laboratory ball mill to further reduce the particle sizes. The particles from the process were sieved with sieve shaker 16155 models into 75, 106, and 300 µm sieve sizes.*

***2.2. Mould Production.** Tensile mould of gauge length 90 × 5× 3 mm of a dumb-bell shape and flexural mould of 150 × 50× 3 mm were used for the production of tensile and flexural samples respectively*

***2.3. Production of Composites**. To develop the composites, 1.5 g each of catalyst and accelerator was added to 120 g of the polyester resin while bone particulate was varied in a predetermined proportions of 2, 4, 6, and 8 wt % for all the particle sizes. After proper stirring, the homogenous slurry is poured into the mould and allowed to be cured at room temperature before it is removed. Same procedure was also adopted for the bone ash particles for all the three different particle sizes. Three (3) samples were produced for each mechanical property that was carried out from each proportion. The striped samples are left to be cured*

further at room temperature for 3 weeks before the mechanical tests were carried out.

2.4. Mechanical Testing of Cast Samples. *Following the moulding of the composites samples were prepared for tensile and flexural tests.*

(a) Determination of the Tensile Properties of the Materials. In the present study, tensile tests were performed on INSTRON 1195 at a fixed crosshead speed of 10mmmin−1. Samples were prepared according to ASTM D412 (ASTM D412 1983) and tensile strength of the standard and conditioned samples was calculated.

(b) Determination of the Flexural Property of the Materials. Flexural test was carried out by using Tensiometric Universal Testing Machine in accordance with ASTM D790. To carry out the test, the grip for the test was fixed on the machine, the sample that has been cut into the test piece dimensions of 150mm × 50mm × 3mm was hooked on the grip, and the test commenced. As the specimen is stretched, the computer generates the required data and graphs. The flexural test was performed at the speed of 100 mm/min.

3. RESULTS AND DISCUSSIONS

3.1. Variation of Tensile Properties with Particle Sizes and Particle Contents

3.1.1. Variation of E-Modulus with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Figure 1 shows the variation of E-Modulus of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites. The modulus of elasticity is a measure of the stiffness of the material and is the rate of change of strain as a function of stress within an elastic limit. The results show that the modulus was enhanced by cow bone ash than cow bone particles in all the particle sizes. The best results were obtained from cow bone ash reinforced samples of 300 µm particle size having 8 and 6 wt % with the highest values of 4597.56 Mpa and 4454.38MPa respectively. This was closely followed by 8 wt % of 75 µm cow bone ash reinforced samples with a value of 4450.49 MPa while the unreinforced polyester material has a value of 3966.15 MPa.

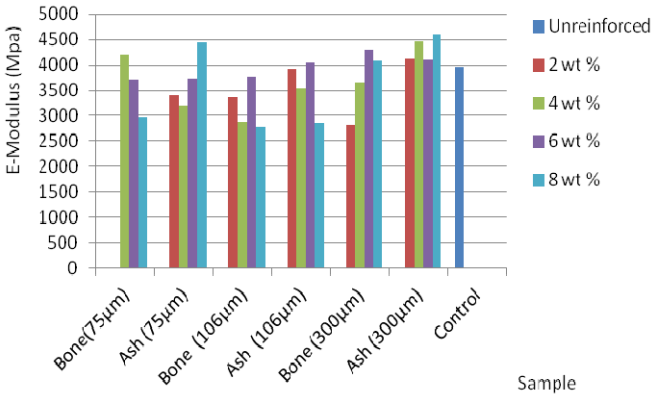


Figure 1. Variation of E-Modulus with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

3.1.2. Variation of Tensile Stress at Maximum Load with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites.

Figure 2 shows the variation of the tensile stress at maximum load of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites.

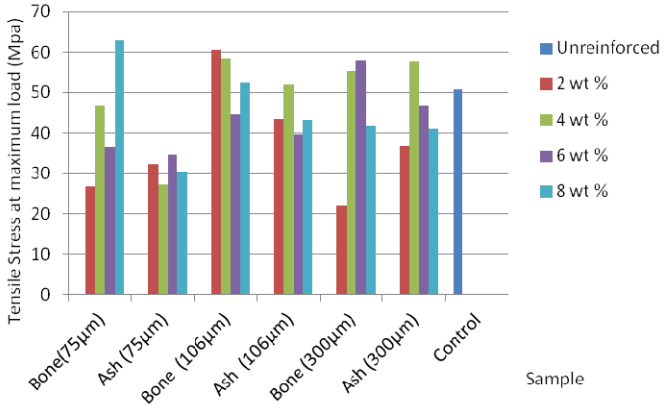


Figure 2. Variation of Tensile Stress at Maximum Load with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Tensile stress at maximum load is the maximum stress that a material can withstand while being stretched or pulled before necking. The trend show that the tensile stress at maximum load increases as the particle size increases for cow bone ash reinforced samples while it decreases as the particle size increases for cow bone particles. This shows that the tensile stress at maximum load is enhanced as the particle size increases for cow bone ash while it decreases as the particle size increases for cow bone particle size. However, the results show that the tensile stress at maximum load for the cow bone reinforced samples were better enhanced compared to cow bone ash samples. From the result, it was observed that sample reinforced

with 8 wt % of particle size 75 µm cow bone has the highest value of tensile stress at maximum load of 63.04 MPa followed by 2 and 4 wt % particle size of 106 µm with a value of 60.72 MPa and 58.54 MPa respectively compared to the unreinforced polyester material with a value of 50.76MPa.

3.1.3. Variation of Tensile Strain at Maximum Load with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Figure 3 shows the variation of the tensile strain at maximum load of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites. Tensile strain at maximum load is the maximum strain that a material can withstand while being stretched or pulled before necking. The trend showed that the tensile strain at maximum applied load decreases as the particle size increases for both cow bone and cow bone ash reinforced samples except for the increase that was observed for the particle size of 106 µm cow bone reinforced sample. From the results, it was observed that cow bone reinforced sample was better enhanced than cow bone ash reinforced samples. The best result was obtained from 4 wt % of particle size 106 µm with a value of 0.025 mm/mm followed by 0.024 mm/mm from 8 wt % of particle size 75 µm cow bone reinforced samples compared to the unreinforced polyester material with a value of 0.01623 mm/mm.

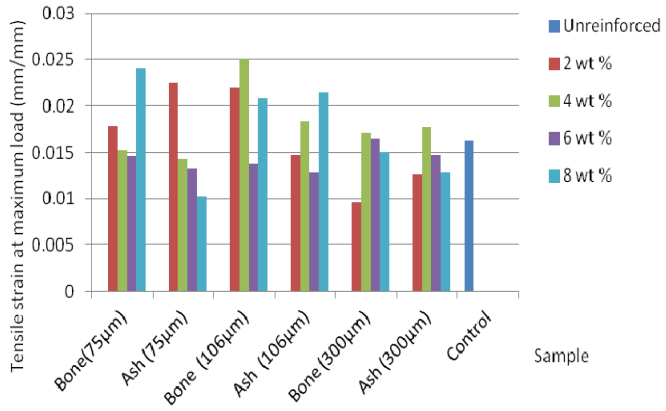


Figure 3. Variation of Tensile Strain at Maximum Load with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

3.1.4 Variation of Tensile Stress at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites.

Figure 4 shows the variation of the tensile stress at fracture of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites. Tensile stress at fracture is the tensile stress corresponding to the point of rupture. The same trend as that of tensile stress at maximum load was observed except that tensile stress at fracture was better enhanced for cow bone ash samples at 300 µm. From the result it was observed that 8 wt % of particle size 75 µm of cow bone has the highest value of 63.04 MPa followed by 2 wt % cow bone particle size of 106 µm with a value of 60.72 MPa compared to the unreinforced polyester material with a value of 50.52 MPa.

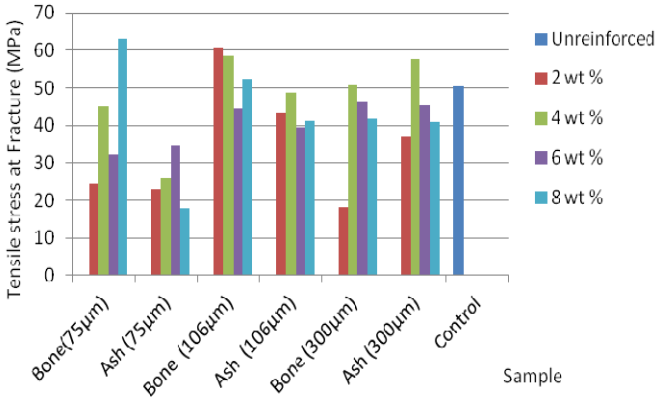


Figure 4. Variation of Tensile Stress at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

3.1.5. Variation of Tensile Strain at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Figure 5 shows the variation of the tensile strain at fracture of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites. Tensile Strain at fracture is the strain strength of the material at the point of rupture. The trend shows that the tensile strain at fracture decreases as the particle size increases for both the cow bone and cow bone ash reinforced samples. This implies that tensile strain at fracture decreases as the particle size increases. Considering the results from this work, it can be deduced that particle size distribution from different reinforcement materials has diverse effect on the properties of the composites. From the results, it was observed that cow bone reinforced sample of 2 wt % from 75 µm has the highest value of 0.03361 mm/mm followed by 2 wt % bone ash reinforced sample from 75 µm with value of 0.02723 mm/mm compared to unreinforced polyester material with a value of 0.01626 mm/mm.

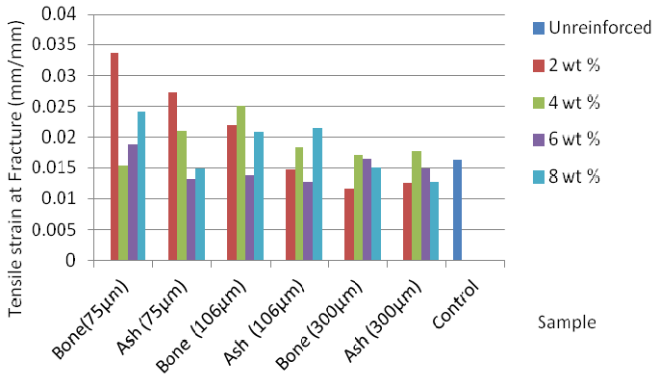


Figure 5. Variation of Tensile Strain at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

3.1.8. Variation of Impact Energy at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Figure 6 shows the variation of impact energy at fracture for the composite samples and the control. The impact energy at fracture is the energy that the specimen has absorbed up to the point of failure. From the Figure, it was observed that the impact energy at fracture decreases as the particle size increases for cow bone particle reinforced samples while cow bone ash reinforced samples gave its optimum result at 106 µm. The best results were obtained from 75µm cow bone with 8 and 4 wt % reinforcements with values 1.49 J and 1.37 J respectively compared to the unreinforced polyester material.

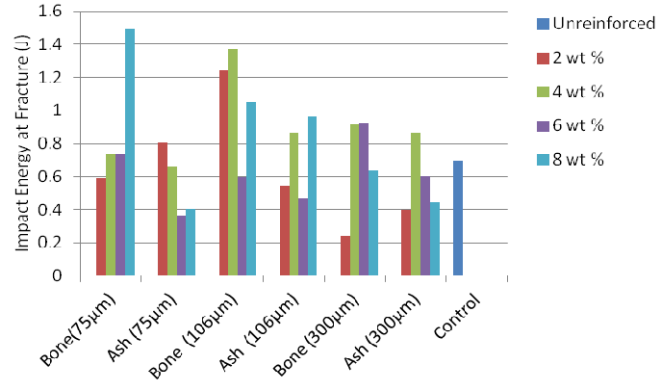


Figure 6. Variation of Impact Energy at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

3.2. Variation of Flexural Properties with Particle Sizes and Particle Contents

3.2.1. Variation of Bending Strength at Peak with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Figure 7 shows the variation of bending strength at peak of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites. Bending strength at peak represents the highest stress experienced by the material when subjected to bending stress before it ruptured. The trend shows an increase that was followed by reduction for the cow bone reinforced samples as the particle size increases while it decreases for cow bone ash reinforced composite as the particle size increases.

From the result, cow bone ash reinforced sample from 8 wt % of particle size 75 µm has the highest value of 68.24 N/mm² followed by 4 wt % of particle size 106 µm cow bone ash reinforced sample with a value of 66.23 N/mm² compared to the control with a value of 43.25 N/mm².

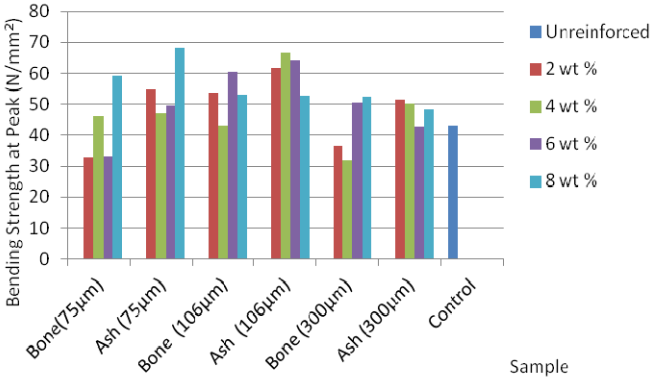


Figure 7. Variation of Bending Strength at Peak with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

3.2.2. Variation of Bending Modulus with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Figure 8 shows the variation of Bending Modulus of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites. Bending modulus also known as flexural modulus of elasticity is the ratio of maximum fibre stress to maximum strain within elastic limit of stress-strain diagram obtained in flexure test. The result revealed that cow bone ash reinforced sample had better enhancement compared to that of cow bone particle reinforced samples.

The best result was obtained when 8 wt % of 300 µm particle size was used with an optimum value of 9137 N.mm² followed by sample with 8 wt % from 75 µm particle size cow bone reinforced sample with a value of 9103 N.mm² compared to the control sample which had a value of 7451.8 N.mm².

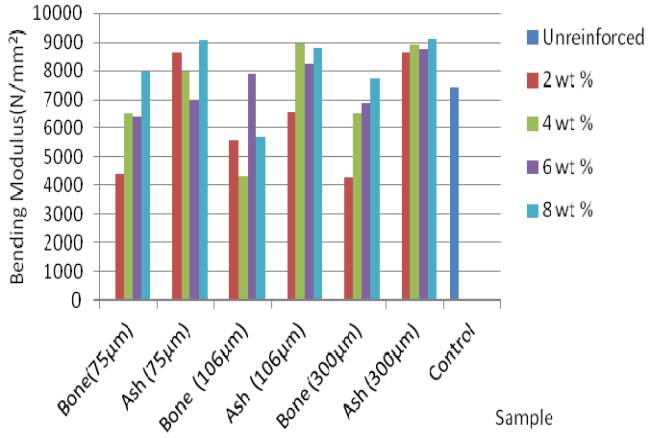


Figure 8. Variation of Bending Modulus with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

3.2.3. Variation of Impact Energy at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

Figure 9 shows the variation of impact energy at fracture of the samples with different particle sizes for both cow bone and cow bone ash reinforced composites. The impact energy to fracture is the energy that the specimen was able to absorb before it failed. Similar trend was observed for both cow bone particles used where 106 µm particle sizes gave the optimum result.

The result shows that 2 wt % of 106 µm particle size from both cow bone ash and cow bone particle reinforced samples gave the highest values of 1.1904 N.m and 1.0804 N.m respectively compared to the control sample which has a value of 0.5684 N.m.

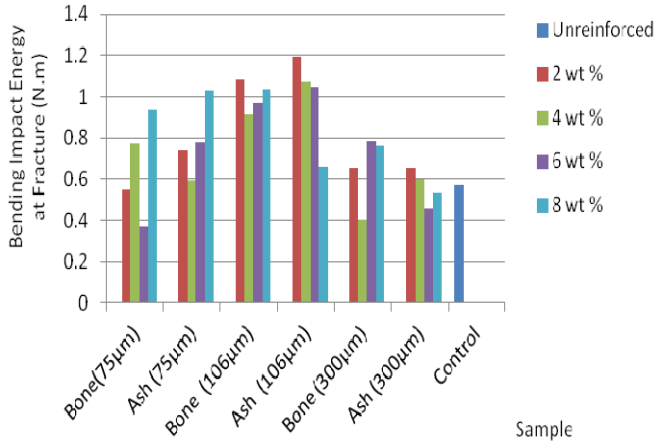
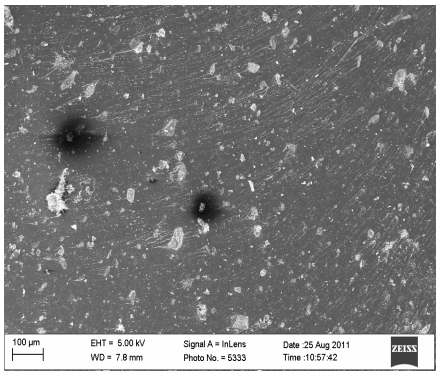
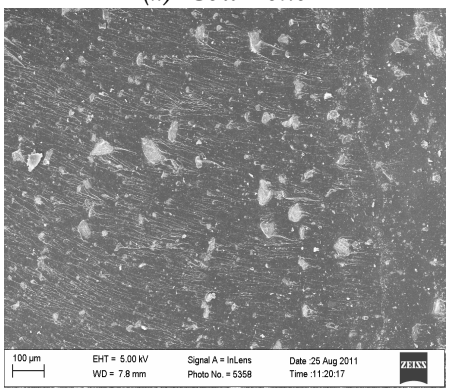


Figure 9. Variation of Bending Impact Energy at Fracture with Particle Sizes for both Cow Bone and Cow Bone Ash Reinforced Composites

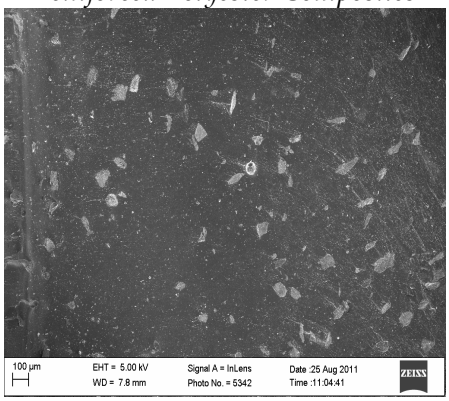


(a) Cow Bone

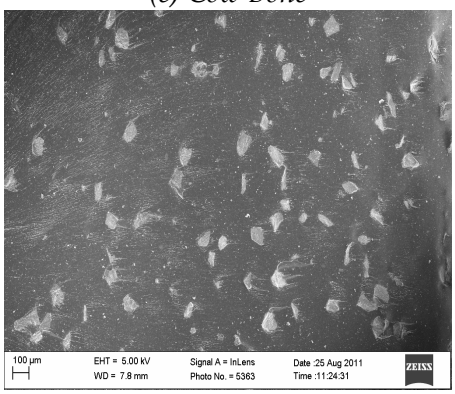


(b) Cow Bone Ash

Plates (a-b). SEM of Fractured surfaces of 8 wt % from 75 µm particle size Cow Bone and Cow Bone Ash-Reinforced Polyester Composites

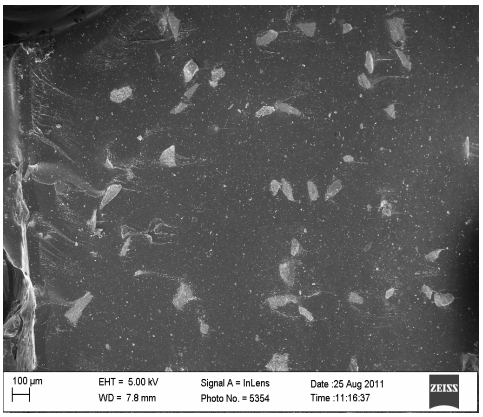


(c) Cow Bone

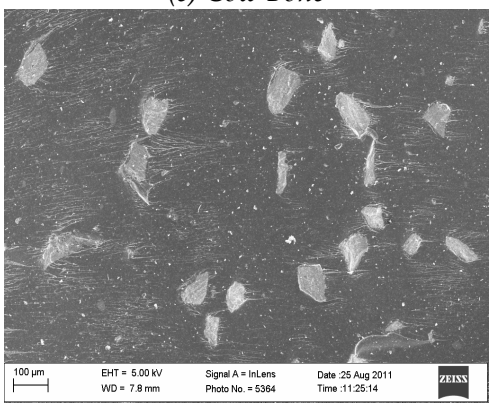


(d) Cow Bone Ash

Plates (c-d). SEM of Fractured surfaces of 8 wt % from 106 µm particle size Cow Bone and Cow Bone Ash-Reinforced Polyester Composites



(e) Cow Bone



(f) Cow Bone Ash

Plates (e-f). SEM of Fractured surfaces of 8 wt % from 300 µm particle size Cow Bone and Cow Bone Ash-Reinforced Polyester Composites

Plates (a- f) depict the SEM micrographs of cow bone and cow bone ash particulates reinforced polyester composites (a-f). From the micrographs, it was observed that both cow bone and cow bone ash particles were well dispersed (white particle) in the polyester matrix (black surface). However, the influence of the cow bone ash particles on the matrix was more pronounced as this affects the dark coloration of the matrix by causing it to be more whitish (plates b, d, f) than that of the cow bone particle reinforced samples (plates a, c, d). By turning the cow bone into ash is one of the biocompatibility treatment expected to be carried out on cow bone for it to be suitable as biomedical implants [8], this observation shows that, the treatment has influenced the matrix structure and, hence, the expected properties. From the mechanical tests results stated above, it was revealed that better enhancement of the properties were achieved from the composites developed compared to the unreinforced polyester material due to proper dispersal of the particles in the polyester matrix.

4. CONCLUSION

The investigation carried out from this research work has revealed that both cow bone ash and cow bone particles can be used as reinforcement in polyester matrix in order to develop composites materials that is suitable as biomaterials. The work showed that variation in particle sizes as well as biocompatibilization treatment has pronounced influence on the microstructure and mechanical properties of the materials. The following conclusions were also drawn out;

- ✓ *Cow bone ash (biocompatibilization treated) particle reinforcement gave better improvement in flexural/bending strength properties while cow bone particle reinforcement gave better enhancement in tensile strength properties except for modulus of elasticity where cow bone ash particle reinforcement offered better enhancement. This confirmed the fact that biocompatibilization treatment offer improved enhancement for the mechanical properties. Hence, it remains a promising material for biomedical applications.*
- ✓ *The enhancement of the mechanical properties of composites was observed to be reducing as the particle sizes increases. Optimum results were obtained from 75 µm particle sizes followed by 106 µm particle sizes. However, 300 µm particle sizes from cow bone ash gave the best enhancement for both tensile and bending modulus. This implies that coarse particle from biocompatibilization treatment offer improved enhancement in modulus property.*
- ✓ *By considering the fibre content, optimum results were obtained from 8 wt % addition followed by 2 wt %.*

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