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Development of Agro-Based Particulate Reinforced Epoxy Composites for Helmet Application

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Abstract

This work attempts to make an improvement in the current helmet manufacturing materials and methodology. The composite was developed using epoxy matrix reinforced with cow bone and snail shell particles that were mixed in predetermined proportions. It was aimed at discovering filled epoxy with superior properties using naturally occurring particles from cow bone and snail shell for the production of safety helmet. The cow bone and snail shell were ground separately and sieved using 38 microns sieve sizes. Elemental composition analysis using X-Ray fluorescence (XRF) of both cow bone and snail shell revealed that snail shell has higher percentage composition of calcium compared to cow bone while manganese oxide and chlorine were present in snail shell only. Cow bone and snail shell particulate reinforced epoxy composites were prepared by varying the cow bone and snail shell particles from 5 to 25 wt% with 5 wt% intervals. Mechanical (flexural, tensile, impact and hardness) properties of the developed epoxy composites were evaluated. The results revealed that the mechanical properties exhibited maximum values at specific percentages of filler additions. From the result, hybrid samples within 15-20 wt% reinforcement showed the highest resistance before shattering relative to other samples. Therefore, for applications where impact strength is a major factor, hybrid sample of 15-20 wt% reinforcement can be used in place of pure epoxy. Elemental compositions were discovered to be part of the reasons for the observed responses from the developed composites.

Keywords: Sustainable materials, hybrid composites, cow bone, snail shell, helmet

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- **Introduction**

The ever-growing need for sustainability, innovations, and energy-efficient technology propels researchers and engineers to take to the production of natural biodegradable polymer composites in place of the synthetic ones in a bid to promote sustainable development. Natural fibers from plants and animals have gained much attention because of this [11; 4]. Natural fibers are gotten from readily available renewable sources usually plants and animals. Some of the investigated natural fibers are sisal, bagasse, pineapple-leaf, rice husk, wheat husk, bamboo, banana, animal hairs/fur, silk, horn, bones, feathers and many more. Natural fibres are attractive fillers because of their promising properties such as low density, recyclability, biodegradability, non-toxicity and eco-friendliness [9; 14;16]. These properties offer the advantage of producing mechanically robust and biodegradable materials at reduced cost. Thus, making the addition of low cost and readily available natural fibers vital in material production and also as potential substitute for expensive synthetic fibers. [15]carried out research to develop a bio-derived hydroxyapatite composite for adhesive biomedical applications using epoxy resins as matrix and snail shell based HAp as reinforcements. This was done in order to develop sustainable adhesive biomaterials with ease of use without impairing the function of the surrounding tissue.

Advanced composites majorly of biodegradable sources are considered as a better alternative to conventional materials due to their significant enhancement in mechanical, structural, and tribological properties [10; 20; 18]. Natural fibers when used as reinforcements particularly in the nanometer range have been found to drastically improve the properties of the materials such as the strength, stiffness, fracture toughness, thermal stability, electrical conductivity, and wear resistance of materials making them highly suitable for application in diverse fields like construction, automobile, biomedical, marine, aerospace, and military [8; 17].

[3] investigated the properties of particulate snail shell reinforced polyester matrix composite where it was revealed that better results can be obtained within 5-20 wt% reinforcement addition since 25-30 wt% tends to give weak results in all. Snail shell/polyester composite can be used in place of pure polyester depending on the filler content and areas of application of interest like aerospace, maritime, auto parts and many more[3]. [13] 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 toughness.[19] worked on design and analysis of industrial safety helmet using natural fibers where it was revealed that wearing appropriate safety helmet significantly reduces the risk of injury or even death. Hence, the aim of this work is to improve the properties of industrial helmet. The helmet model is analyzed using Creo simulate 2.0 software to test whether the helmet can withstand high impact load or not and compared the results of both the natural fiber helmet and polypropylene helmet**.**

- **Materials and Method**

The main materials that were used for this work are cow bone (CB) and snail shell (SS) obtained from farmland in Akure, Ondo State while epoxy resin and hardener were gotten from GZ Industrial supplies, Lagos State, Nigeria. The cow bone and snail shells were washed and sundried for 5 days after which they were crushed with hammer and finally pulverized using laboratory ball mill. The particles were sieved with a sieve shaker to obtain particle size of <38 μm. Figure 1 (a-d) showed the snail shell, snail shell particulate, cow bone and cow bone particulate, respectively.

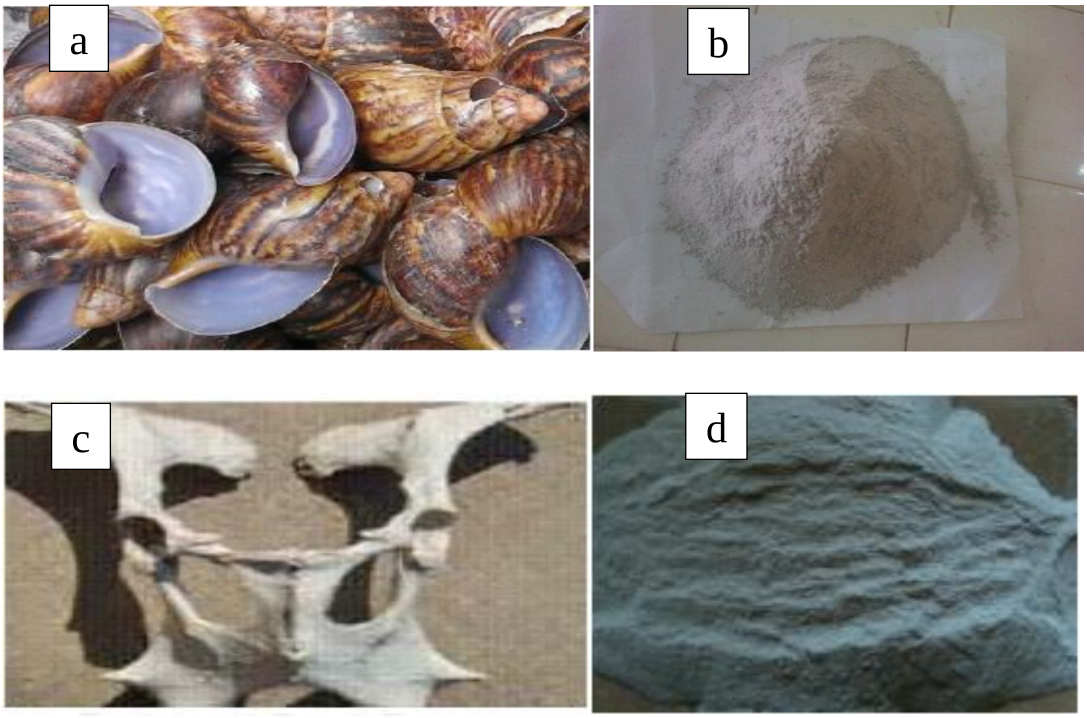

Figure 1: Animal wastes used: (a). Snail shell (b). Snail shell particulate (c). Cow bone (d). Cow bone particulate

Development of animal waste particulate reinforced epoxy based bio-composites

Development of the composites was formed using the open mould technique that was engraved to the shape of the tests to be carried out. The pulverized CB/SS micro particles were integrated into the epoxy matrix in the range of 5 to 25 wt. % CB/SS. Respective particulate CB/SS reinforced epoxy bio-composites were formed by casting compounded materials (epoxy resins, hardener and CB/SS particles) into test sample moulds and extracted after curing. The samples were tested in accordance to appropriate ASTM standards. Table 1 shows the various sample formulations for the bio-composites based on Epoxy, Hardener and CB/SS while Figure 2 showed the developed composite samples after curing.

Table 1: Formulation of the bio-composites

Filler (%)	Epoxy	Hardener	Cow bone/Snail Shell
Control	50.00	25.00	-
5	47.50	23.75	2.50
10	45.00	22.50	5.00
15	42.50	21.25	7.50
20	40.00	20.00	10.00
25	37.50	18.75	12.50

Figure 2: Developed composite samples

- **Material Testing**

Tensile mould of dimension 90 x 5 x 3 mm shape was used for the production of tensile samples. Testing procedures were conducted in accordance with the ASTM D412. The test was carried out by placing the tensile samples between two fixtures called “grip” which clamp the sample. The samples are subjected to load that keeps increasing while at the same time experiencing change in length. Three (3) samples each were tested and the average values were obtained and used as the representative values.

Flexural test was carried out on three points bending test on the samples using Testometric Testing Machine with serial number 25257 and capacity M500-25KN. The flexural test was carried according to ASTM D7264 at a cross-head speed of 20 mm/min maintaining a span of 100 mm. The flexural test specimens were of 120 x 50 x 10 mm in dimension. The test was performed by placing the samples under a three point bend fixture. Three (3) samples each were subjected to bending by supporting them at both ends and a midpoint load applied until failure as recommended in ASTM D7264.

The impact energy test of the composite samples was determined using Charpy impact testing machine with model no 412-07-15269C. The procedure used was in accordance with ASTM D-256. Samples were sectioned to 100 x 10 x 3 mm dimensions and notched at the middle after which 3 samples were each prepared, mounted on the machine, and a swinging pendulum released, under gravity, to hit the sample(s). The energy at impact was read directly from a dial indicator of the machine and the average values of the results obtained were recorded.

Hardness test was carried out in accordance with ISO R 868, using micro hardness tester ASTM E384 with maximum load of 4000 N. The test was carried out by impressing the sample with the tip of the indenter for five seconds before taken the readings from the calibrated scale. Ten readings were taking for each sample and the average value was used as the representative value for the mechanical tests carried out.

X- Ray Fluorescence

X-ray fluorescence test was carried out on cow bone and snail shell particulates to determine the elemental composition of the materials. The chemical analysis was performed with EDXRF Spectrometer (EDX3600B). About 0.5 g of the sample material was pressed using lithium tetraborate (flux) which allowed an evenly dispersed solid solution. The X-rays beamed on thesample and excited some of the atomic nuclei (inside the atoms of the elements present), to jump on the higher energy orbitals and the elements in the sample were shown and identified.

Scanning Electron Microscope (SEM) Characterization

The EVO MA 15, Carl Zeiss SMT operated at 15 kV was used for the SEM morphological study of the fractured surfaces of the developed composite samples. The samples were gold coated with Quorum coating machine (Q150RES) to make them conductive before SEM observation.

- **Results and Discussion**

The results from the XRF analysis of snail shell carried out revealed the information on Table 3. From the table, calcium oxide had the highest composition of 91.63%.The next element with highest composition was sodium oxide with a value of 1.20%. Others were in minor composition. From Table 4, calcium oxide (principal mineral) had the highest composition of 53.00%. This was followed by phosphorus oxide with a value of 38.19%. Alumina and silica have the values of 2.40% and 2.34% respectively while other element oxides were in small quantities.

Table 3: XRF analysis of the Snail Shell

Constituent	Description	Amount (%)
SiO_2	Silica	0.60
Al_2O_3	Alumina	0.51
Fe_2O_3	Ferrous oxide	0.56
CaO	Calcium oxide	91.63`
MgO	Magnesium oxide	0.69
SO_3	Sulphur trioxide	0.19
Na_2O	Sodium oxide	1.20
K_2O	Potassium oxide	0.12
TiO_2	Titanium oxide	0.03
P_2O_5	Phosphorus Pentoxide	0.21
MnO_3	Manganese oxide	0.02
Cl	Chlorine	0.03

Table 4: XRF Analysis of the Cow Bone

Constituent	Description	Amount (%)
SiO_2	Silica	2.40
Al_2O_3	Alumina	2.34
Fe_2O_3	Ferrous oxide	0.14
CaO	Calcium oxide	53.00`
MgO	Magnesium oxide	1.52
SO_3	Sulphur trioxide	0.30
K_2O	Potassium oxide	0.15
P_2O_5	Phosphorus Pentoxide	38.19
BaO	Barium oxide	0.14

Figure 3 shows the variation of young modulus against filler content. The Young's modulus 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 result revealed that, Cow bone gave better enhancement in most of the weight fractions used compared to other reinforcements. However, best enhancement was achieved with 20 wt.% Snail shell reinforcement with a value of about 374.05 MPa. The hybrid reinforced sample showed that, the modulus decreases as the reinforcement content increases. The best enhancement obtained from Cow bone was achieved with 20 wt.% reinforcement which has a value of about 288.08 MPa while the one from hybrid was achieved with 5 wt.% reinforcement having a value of about 275.61MPa. All these were higher than the control value of 226.04 MPa.

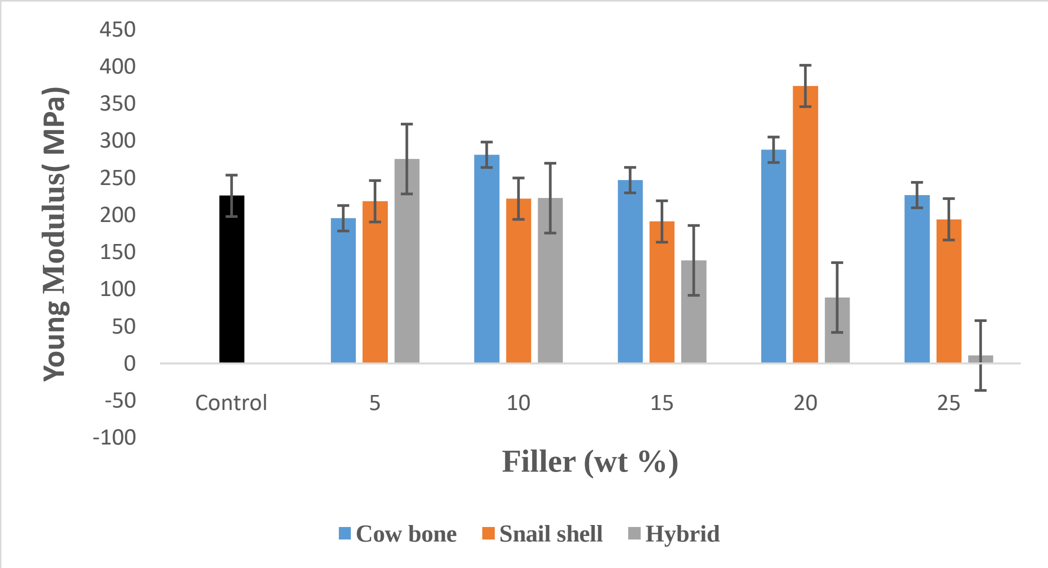

Figure 3: Variation of Young Modulus against Filler Content

Figure 4 shows the variation of ultimate tensile strength against filler content. Ultimate tensilestrength is the maximum stress that a material can withstand while being stretched or pulled before necking. Developed hybrid composites in all the variation have better ultimate tensile strength compared to other reinforcements and control. The presence of elements like Calcium (Ca) and Titanium (Ti) in Snail shell coupled with that of Cow bone induced strength into the hybrid composites making it to have highest ultimate tensile strength. It exhibited the highest ultimate tensile strength at hybrid 20 wt.% filler content with 48. 06 MPa compared with the control sample with 10.40 MPa.

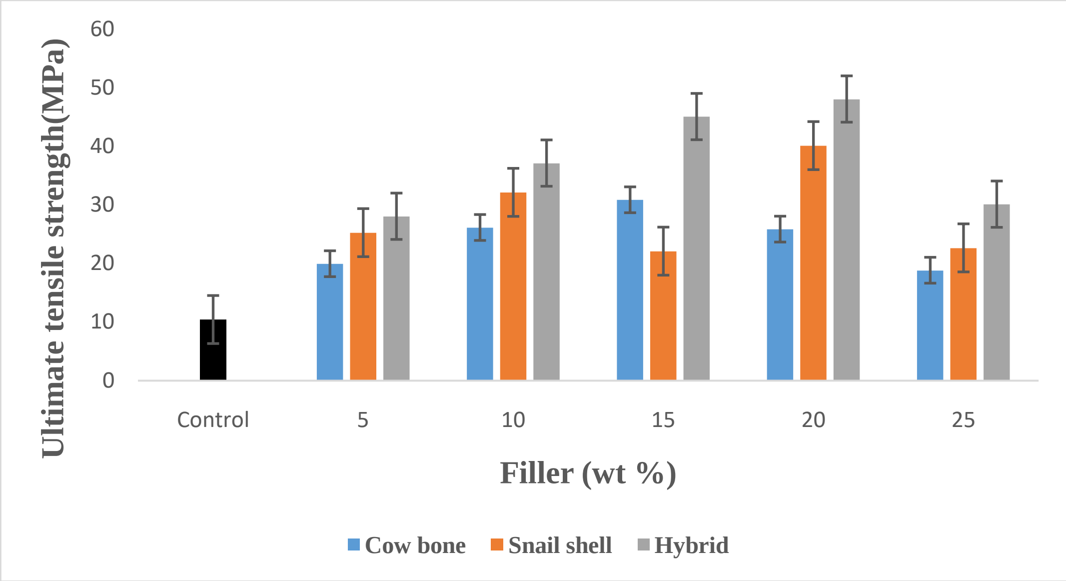

Figure 4: Variation of Ultimate Tensile Strength against Filler Content

Figure 5 shows the variation of tensile strain against filler content. Tensile Strain at fracture is the strain strength of the material at the point of rupture. Here, it is seen that the 5 wt.% cow bone reinforcement has the highest strain (0.0382 mm/mm) and also the 15 wt.% hybrid reinforcement showing the least strain (0.0082 mm/mm), while the snail shell showed its maximum strain at 15wt.% filler concentration with value of 0.027 mm/mm compared with control with value of 0.0385 mm/mm. This graph shows that additional filler concentration to the 5wt.% snail shell reinforcement and the 15 wt.% cow bone reinforcement will not improve the strain. The result showed that all the samples displayed low tensile strain at fracture compared to the control. This was envisaged based on the properties expected on particulate reinforced epoxy composite. The addition of these particles induced stiffness into the material.

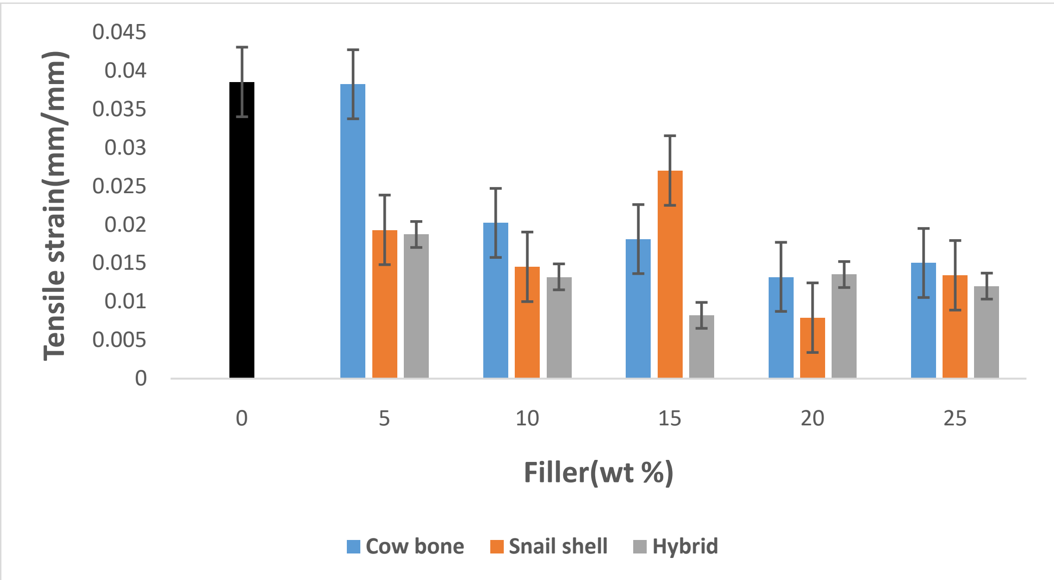

Figure 5: Variation of Tensile Strain against Filler Content

Figure 6 shows the variation of bending strength at peak against filler content. Bending strength at peak is a material's ability to resist deformation under load. It represents the highest stress experienced within the material before rupture. From Figure 4.4, it was observed that bending strength at peak for all the three reinforcements gave different responses. There is an increase from 5–10 wt. %for Snail shell samples before a sharp decrease while initial decrease from 5–10 wt. % for hybrid was followed by an increase from 15 – 20 wt.% followed by slight decrease at 25 wt.%. These two reinforcements (hybrid and Snail shell) possess some values that are higher than the control at different wt.%. However, for Cow bone, none of the samples gave any enhancement compared to the control. What was noticed was that, initial increase from 5–10 wt. % reinforcement. Best result was obtained from hybrid composite with 20 wt. % reinforcement having a value of 34.02 MPa followed by sample from Snail shell reinforced composite at 10 wt.% with a value of 33.64 MPa.

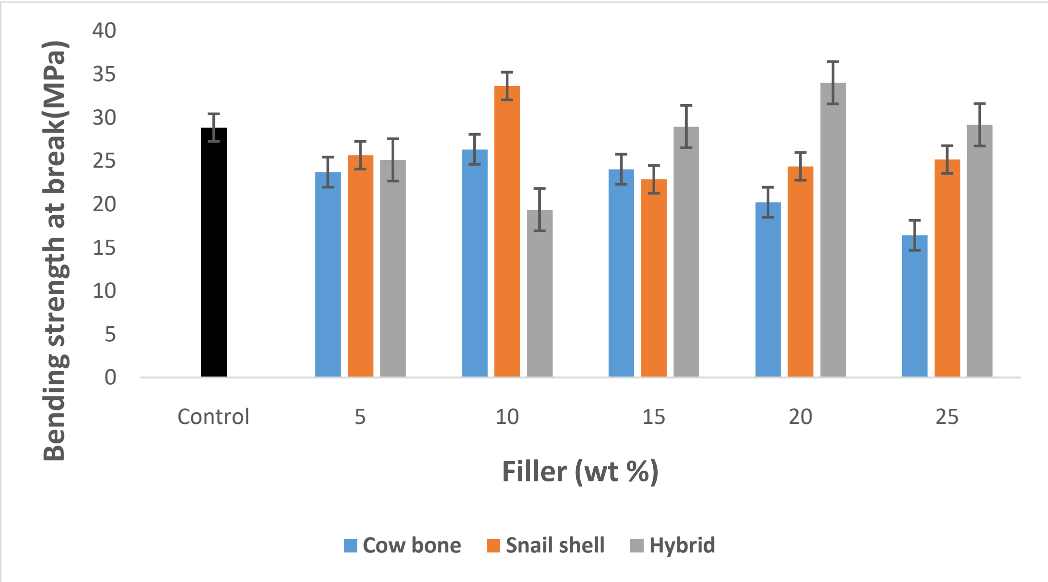

Figure 6: Variation of Bending Strength at Peak against Filler Content

Figure 7 shows the variation of bending modulus against filler content. Bending modulus is the ratio of maximum fiber stress to maximum strain within elastic limit of stress-strain diagram obtained in flexure test. Figure 4.5 shows the bending modulus of all the three reinforcements from where it was observed that, the modulus tend to increase as the reinforcement content increases. The resultrevealed that hybrid reinforced sample hadbetter enhancement compared to that of cow bone and snail shell particle reinforced samples.The best result was obtained when 20 wt .% hybrid was used with an optimum value of 1720.60 MPa followed by sample with 25 wt. % cow bone reinforced sample with a value of 1648.25MPa compared to the control sample which had a value of 926.10 MPa.

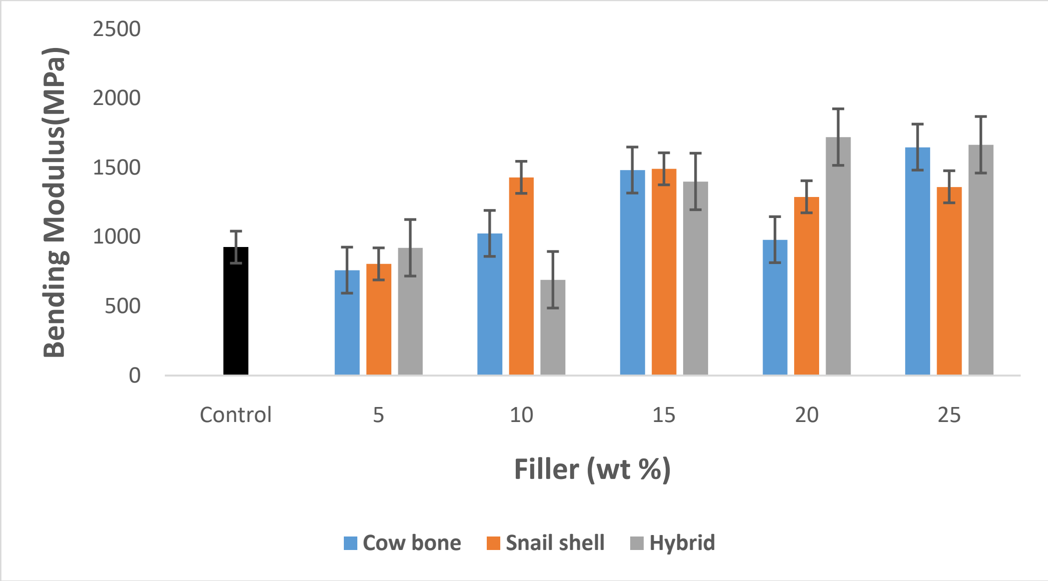

Figure 7: Variation of Bending Modulus against Filler Content

Figure 8 shows variation of impact strength against filler content. The impact energy at fracture is the energy that the specimen has absorbed up to the point of failure. Hybrid composite gave best results with respect to the wt% fractions used with the value at 15 wt. % having a value of about 12.92 J. For single reinforcement, Cow bone samples tends to decrease as the wt% fraction increases while for Snail shell, initial decrease from 5-10 wt.% reinforcement was followed by increase from 15-25 wt.%. The best was obtained at 25 wt. % for Snail shell. 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 that the impact strength decreases.

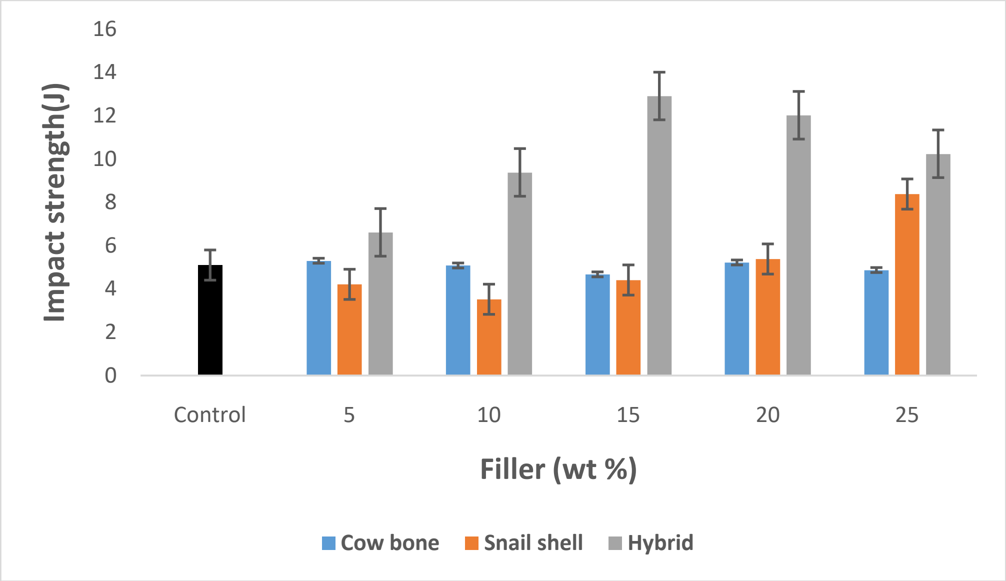

Figure 8: Variation of Impact Strength against Filler Content

Figure 9 shows the variation of Brinell hardness against filler concentration. Hardness property is a measure of the resistance of the materials to surface indentation and wear. The hardness tends to increase as the reinforcement content increases except in 20 and 25 wt.%, respectively. It was noticed that Hybrid and Snail shell reinforced samples tends to be the best in entire fraction content considered. It was noticed that the highest hardness was exhibited by hybrid at 15 wt.% with a value of 80.25 BHN compared with the control with value of 28.70 BHN. This may due to the high percentage of CaO presence in the Snail shell and later complement that of Cow bone (hybrid) which tends to improve the hardness of the materials.

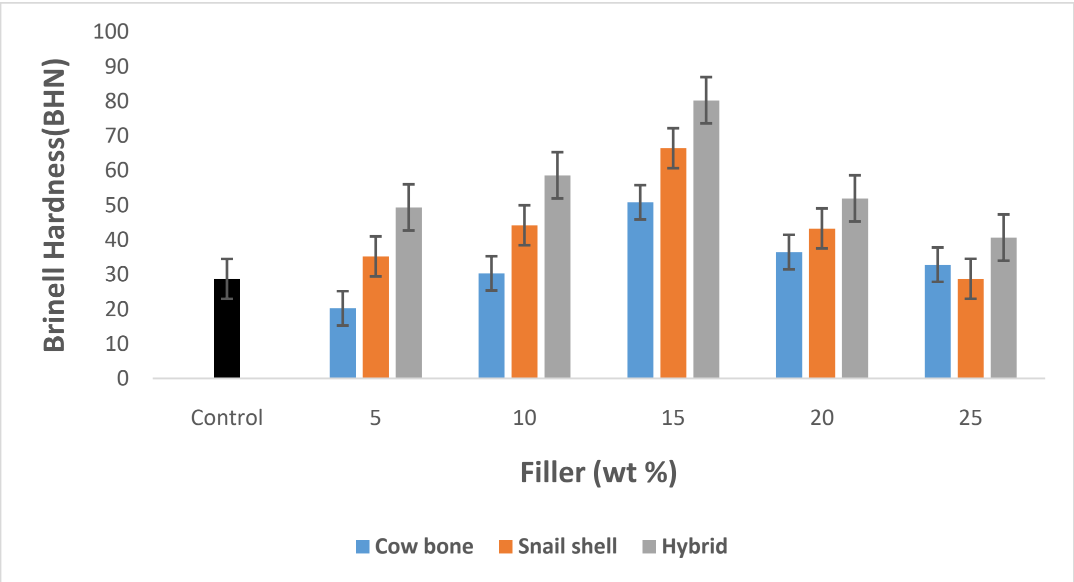

Figure 9: Variation of Brinell Hardness against Filler Content

Scanning Electron Microscope Image of Fractured Hybrid Composite

The extent of dispersion of particles within the matrix is observed using SEM where it was observed that hybrid reinforced epoxy composites revealed irregular distribution of CB/SS particles in epoxy matrix as shown in Plate 1. This sample was selected because it falls within the range of composition with the optimum properties that was recommended for the development of helmet as expected. This observed distribution may likely contribute to good interfacial adhesion between the particles and epoxy which invariable aid improved mechanical properties as observed from the results. This is in agreement with the findings of Oladele et al. (2022) where it was discovered that the adhesive property of epoxy resin was part of the reasons for proper interfacial adhesion at the HAp/epoxy resin interphase that produced improved properties at various weight fractions.

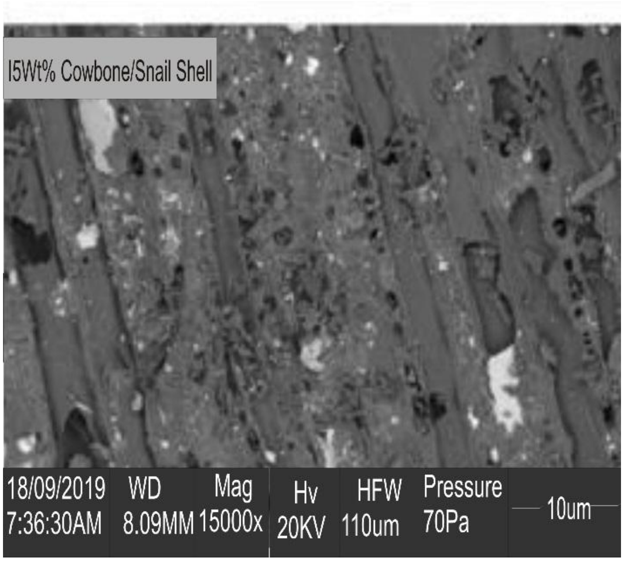

Plate 1: SEM Micrograph of 15 wt. % SS/CB composite

Development of helmet prototype

Helmet prototype was produced with 15 wt. % CB/SS hybrid reinforced epoxy composites following the design shown in Figure 10. The epoxy resin with hardener was thoroughly mixed with required reinforcements before pouring into the mould. The reinforcements/resin was allowed to spread uniformly up to required thickness and allow curing before de-moulding. The develop helmet prototype was removed by uncoupling the male and female moulds. The moulds and the developed helmet prototype are shown in Figures 11-12.

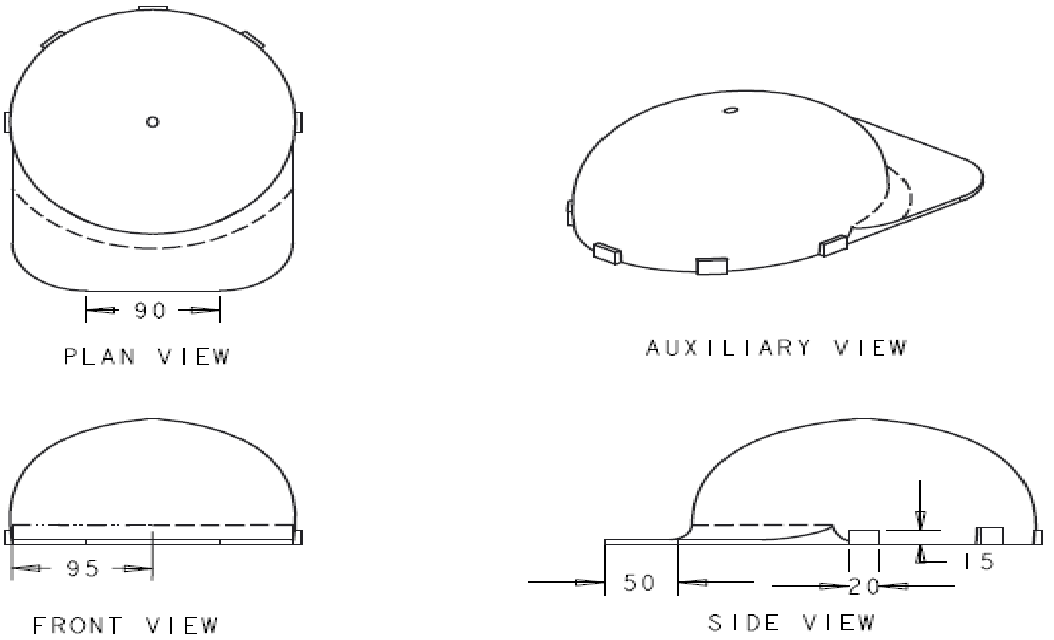

Figure 10: Mould drawn using PTC Creo 2.0

Figure 11: Mould used for the development of the helmet prototype (Cope and Drag)

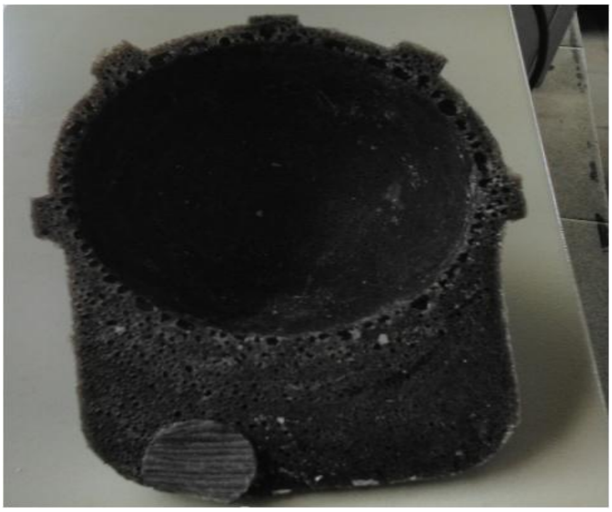

Figure 12: Developed helmet prototype

- **Conclusion**

This work revealed that helmet can be successfully produced using cow bone and snail shell particulates as filler in epoxy matrix based composite with improved mechanical properties.

X-ray fluorescence revealed that high content of CaO in snail shell and cow bone with other elemental compositions in low contents. The blend of these compounds aided the improved properties obtained in the hybrid reinforced composites compared to the single reinforced ones. Hybrid reinforced composites was the best in most of the properties examined except for tensile modulus where Snail shell reinforced samples gave the best result followed by the hybrid samples. The properties were mostly enhanced within 15-20 wt.% reinforcement in particular, for hybrid reinforced composites. Hence, helmet prototype was produced with 15 wt.% CB/SS hybrid reinforced epoxy composites.

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