

## Effect of particle size and weight percentage variation on the mechanical properties of periwinkle shell reinforced polymer (epoxy resin) matrix composite

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### ABSTRACT

Polymers are very interesting and useful materials that have many applications in various areas of engineering. Composites formed with these materials are known to exhibit outstanding mechanical, electrical, and thermal properties. In this work, a polymer, epoxy resin, was reinforced with a biodegradable material, periwinkle shell (PWS) particles, using the hand lay-up method. The PWS was pulverized using a ball mill and three sieve sizes of the PWS (75, 150, and 300  $\mu\text{m}$ ) were sieved out. Various samples of the composite were produced by reinforcing the epoxy resin matrix with 10, 20, 30, 40, and 50 wt% of each of the PWS particle sieve sizes. The samples so formed were subjected to the following mechanical tests: hardness, tensile, compressive, and impact tests. It was found out that the samples of composites showed higher values of the parameters tested for than ordinary epoxy resin showed. In the samples of composites, it was found that the samples with a higher weight percentage of the PWS reinforcement recorded higher values of those mechanical properties tested for. The higher the weight percentage of the PWS in the composite, the greater the value of the mechanical property tested for.

**Keywords:** Composites, Epoxy resin, Periwinkle shell, Mechanical properties.

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### 1. INTRODUCTION

In recent times composite making has become a major method of developing new materials with superior quality for service in various areas of engineering. The application of composite materials can be seen in construction, civil, electrical, automobile, aeronautics, and mechanical engineering. Generally, a composite is a material made from two or more materials with distinctly different properties but amalgamated and engineered to have specific superior properties different from those of the constituent materials (Mishra et al., 2002; Landesmann et al., 2015). In a composite material, one of the constituent materials will necessarily be in a continuous phase known as the matrix while the other constituent is in a discontinuous phase known as the reinforcement. While the matrix is relatively softer and accommodates the reinforcement phase and shares the applied load with it, the reinforcement is harder and majorly carries the applied load to the composite. Many authors have shown that composite forming leads to the improvement of various mechanical, electrical, thermal, and corrosion resistance properties of the material being reinforced (Iyasele, 2018; Ofem and Umar, 2012; Loto and Udo, 2019; Singh et al., 2020; Rajak et al., 2019; Babalola et al., 2015).

Polymers such as epoxy resins are among the materials that have been successfully used as matrix components in developing composite materials (Oladele et al., 2016; Saba et al., 2016; Yang et al., 2017; Rana et al., 2020). Epoxy resins constitute cross-related compounds, many of which have the same properties or form of a reactive set of functions. Epoxy resin is a versatile thermosetting resin produced by copolymerizing an epoxy with another compound containing two classes of hydroxyl (Adams et al., 2015). It is used primarily in coatings and adhesives. The nomenclature epoxy resin applies to both untreated and cured resin. The commercial interest and usage of such resins lie mostly in its useful properties such as strong adhesion to other substrates, relatively high durability, strong tolerance to the atmosphere, high electrical resistance, low shrinkage, etc. Epoxy resin is generally known for its adhesive qualities and good corrosion resistance. It is one of the toughest polymeric products known to man with many other compelling properties such as corrosion tolerance, environmental deterioration resistance, capacity to bind firmly to a number of substances, and appreciable thermal decomposition tolerance compared to other polymers (Barcia et al., 2003).

The reinforcement used in this work is the periwinkle shells (PWS), a shell got after the edible part of a marine gastropod mollusk, *territella communis*, has been removed. The shell which acts as a protective coat for the exoskeletal mollusk against mechanical damage and predators is regarded as waste after the edible part has been removed and more often is discarded and stock-piled only to constitute a source of environmental pollution. Periwinkle shell is constituted of several layers of an organic matrix (conchiolin) which is bonded with calcium carbonate precipitate which is impervious to water (Njoku et al., 2011). Calcium carbonate is a natural mineral filler used for the production of composites. Studies show that particulate extracts from PSW can be used to boost some inherent engineering properties of polymer materials (Banoriya et al., 2020; Karami et al., 2019; Hongwei et al., 2013). Onuoha et al. (2017) in their study of the effect of filler loading and particle size on mechanical properties of periwinkle shell filler recycled polypropylene composite, found that periwinkle shell powder improves tensile strength, flexural strength, and hardness of polypropylene composite. Umunakwe et al. (2017), who worked on the assessment of some mechanical properties and microstructure of particulate periwinkle shell aluminum 6063 metal matrix composite showed that the addition of periwinkle shell particulate resulted in improved strength, ductility, toughness, and porosity of the composite. Previously, most of the reinforcement materials were synthetic material like silicon carbide, SiC, titanium oxide, TiO, etc but recently researchers have found out that some biodegradable materials like periwinkle shells, eggshells, rice husks, coconut shell are composed of minerals like calcium carbonate, silicon dioxide which can act as fillers to reinforce composites and these biodegradable materials

have been used as reinforcement in form of particulate or fiber in the composite making. Most of these biodegradable materials are regarded and treated as waste materials that can only be discarded or stockpiled away as a nuisance and source of pollution of the environment. In this work, a polymer, epoxy resin is reinforced with particulates of the periwinkle shell using a hand lay-up method to produce composites from different grain sizes and volume fractions of the pulverized periwinkle shell. The composites so produced were subjected to a mechanical properties test to see whether periwinkle shell can be used as reinforcement material for epoxy resin, and to find out whether this reinforcement will lead to the improvement of mechanical properties of the epoxy resin.

## 2. MATERIALS AND METHOD

### 2.1 Materials

The main materials used in this research include (a) periwinkle shells, (b) epoxy resin, (c) epoxy resin hardener. Periwinkle shells were procured from Ota open market in Ogun State, Nigeria while epoxy resin and the hardener were procured from a chemical supply company in Lagos, Nigeria.

#### 2.1.1 Materials Preparation

The periwinkle shells (see Fig. 1a) from the open market were purchased and thoroughly cleaned to remove all dirt from them. The cleaning involved soaking the PWS in detergent water for 6 hours, washing them thoroughly with a wire brush, drying them under the sun for 36 hours to remove all moisture. The thoroughly washed and dried PWS were pulverized (See Fig. 1b) using a ball mill and then sieved or classified using a sieving machine to get the required grain sizes for the research work. The grain size used were 75, 150, and 300  $\mu\text{m}$ . On the other hand, the epoxy resin was mixed with an epoxy hardener and stirred properly for uniform mixing.



**Fig. 1.** (a) Periwinkle shell (b) Pulverized periwinkle shell

The different samples of the composite were produced using the 'Hand Lay-up' method. The process started with pattern making, which this time involved cutting out a 60 mm length from a mild steel pipe of 30 mm inner diameter to serve as the mould. The inside of the mould was then coated with release gel using a brush so that easy removal

of the composite will be facilitated after the composite would have set. Then, the weight fractions of the matrix material – the epoxy resin and the reinforcement - the PWS were carefully meted out, mixed together, and vigorously stirred to ensure even dispersion of the particulates of the PWS in the epoxy resin. The mixture was poured into the mould and left to cure. Five sets of the composite samples were produced from each grain size of the reinforcement. That is, for PWS particles of 75 µm grain size, composite samples having 10, 20, 30, 40, and 50 wt% were produced. This was repeated for PWS particles of 150 and 300 µm grain sizes. The samples from the 3 sieve sizes of PWS, ie 75, 150, and 300 µm were labeled as shown in Table 1.

**Table 1.** Composite sample labelling

Samples	PWS/Polymer wt%
A	0/100
B	10/90
C	20/80
D	30/70
E	40/60
F	50/50

**2.2 Mechanical Tests**

Each of these samples was subjected to Brinell hardness, tensile strength, compressive strength, and impact tests. The results of these tests were recorded.

**2.2.1 Brinell Hardness Test**

This test was carried out on a 10 x 10 mm cut specimens and it was conducted using a TQ SM 1000 Universal Testing machine having a steel ball of 10 mm depth indenter. The samples for the test were prepared according to ASTM E10 standard and with a Brinell scale relationship given in equation (1). The Brinell hardness values were calculated for the various samples.

$$HBS = 0.102 \cdot \frac{2F}{\pi \cdot D (D - \sqrt{D^2 - d^2})} \tag{1}$$

where HBS =Hardness Brinell Steel, F = Applied force, D = Indenter diameter, d = Indentation diameter

**2.2.2 Tensile Strength Test**

The samples for this test were prepared in accordance with ASTM D638 standard test. The samples were sectioned having 3.2 mm thickness, 19.5 mm wide and 165 mm long and the tensile test was conducted on Universal Testing Machine – UTM (Instron5567). A test sample was gripped at both ends and the machine slowly pulls the sample until it fractured.

**2.2.3 Impact Test**

Impact strength is the measure of a material’s toughness. While toughness is the material’s ability to absorb energy without rupturing. The samples for this test were prepared

according to the ASTM D256 standard with a sample size of 12 mm x 64 mm and the test was carried out in Izod impact testing machine.

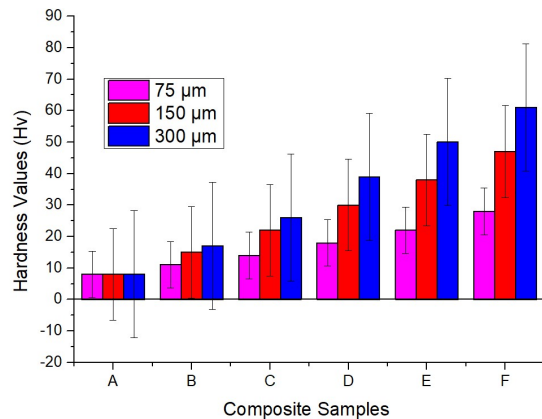
**2.2.4 Compressive Strength Test**

The compressive strength of construction material is a measure of its ability to withstand loads tending to reduce its size. Compression strength test was carried out on Universal Testing Machine using sample size of 15 x 15 x 15 mm in accordance with ASTM C39 test standard (Mulaba-Kapinga et al., 2020). Each sample of the composite was placed between two compression platens to introduce compressive load to the material. The compression continues until the material fails.

**3. RESULTS AND DISCUSSION**

**3.1 Brinell Hardness**

The result of the Brinell hardness test conducted on the samples of the composite is represented graphically in Fig. 2.



**Fig. 2.** Graph of variation of Brinell Hardness with wt% of PWS

The result shows that the hardness of the composite is greater than that of the epoxy resin without any reinforcement. Fig. 2 indicates that 300 µm was the highest in all tested composite samples with hardness values (Hv) of 61, 50, 39, 26 and 17 Hv for samples F, E, D, C and B respectively followed by 150 µm with hardness values (Hv) of 47, 38, 30, 22 and 15 Hv for samples F, E, D, C and B respectively while 75 µm recorded the least values of hardness in all tested composites samples with 28, 22, 18, 14, and 11 Hv for samples F, E, D, C and B respectively. Nevertheless, at Sample A which is pure polymer resin, all sizes (i.e. 75, 150 and 300 µm) were observed to have the same hardness values of 8 Hv. Standard deviation for hardness values at different microns was taken at a scaling factor of 1. It was noted that the standard deviation of 7.38693 was recorded when 75 µm was used, and 14.55564

standard deviation was recorded at 150  $\mu\text{m}$  while 20.18663 standard deviation was computed at 300  $\mu\text{m}$  for hardness values as reflected in Fig. 2. The addition of reinforcement greatly after the hardness properties which is also proportional to the particle sizes of the PWS. This result is in line with other earlier findings on the effect of periwinkle shell particles on polymers (Barcia et al., 2003; Njoku et al., 2011; Onuoha et al., 2017). The presence of hard particles of calcium carbonate ( $\text{CaCO}_3$ ) from PWS in the matrix of epoxy resin imparts hardness to the composite. The result also reveals that the more the quantity of the PWS particles in the composite, the higher the hardness value. Also, within the limits of this research, hardness values increased as the grain size of the filler PWS increases. The composite samples with 300  $\mu\text{m}$  grain size and 50 wt% of PWS recorded the highest values of 61.

### 3.2 Tensile Strength

Fig. 3 shows the result of the tensile tests conducted on the samples of the PWS reinforced epoxy resin matrix composite. Within the limits of this research, it is found out that the result of the tensile test on the composite is almost following the pattern of the result of the hardness test. That is all samples of the composite show a better tensile strength value than that of unreinforced epoxy resin. This reveals that periwinkle shell reinforcement enhances and improves the tensile strength of epoxy resin. Also, within the limit of this research work, the tensile strength value increases as the grain size of the periwinkle shell increases. Other research works on a similar has shown that higher grain sizes of PWS lead to a lower tensile strength of composites, but this always occurs where the gain size exceeds 300  $\mu\text{m}$  (Banoriya et al., 2020; Onyechi et al., 2015). The increase in tensile strength can be attributed to drastic reduction or absence of void or porosity in the composite matrix, good dispersion of the PWS in the epoxy resin matrix as well as good interfacial bond existing between the epoxy resin and PWS reinforcement. According to Fu et al. (2008), matrix/filler interfacial adhesion significantly affects the strength of particulate composites apart from the filler's particle size and weight fraction.

Better matrix/filler interface adhesion improves the tensile strength of polymer matrix composite (Onyechi et al., 2015). The tensile strength was also found to increase as the weight percentage of the filler PWS increased in the composite. This result is in line with similar findings (Banoriya et al., 2020; Njoku et al., 2011). Numerically speaking, it was noticed that at 300  $\mu\text{m}$ , tensile strength recorded the highest values for all samples reinforced with PWS in which sample F was 364.9 MPa while sample E, D, C and E recorded 326.4, 287.4, 248.8 and 210.3 MPa respectively. There was no significant difference in the results when difference particle sizes were used on sample A with no reinforcement (172 MPa for all microns used). Nearly uniform results were observed when 150  $\mu\text{m}$  PWS was used for reinforcement on all samples with 214.3, 206.5,

198.1, 188.9, and 180.7 MPa for composite samples F, E, D, C and B respectively. The same trend was noticed when 75  $\mu\text{m}$  PWS reinforcement was applied. Standard deviation for tensile strength values at different microns was taken at a scaling factor of 1. It was noted that the standard deviation of 8.7683 was recorded when 75  $\mu\text{m}$  was applied, and 15.93743 standard deviation was recorded at 150  $\mu\text{m}$  while 72.25158 standard deviation was computed at 300  $\mu\text{m}$  for tensile strength values as depicted in Fig. 3.

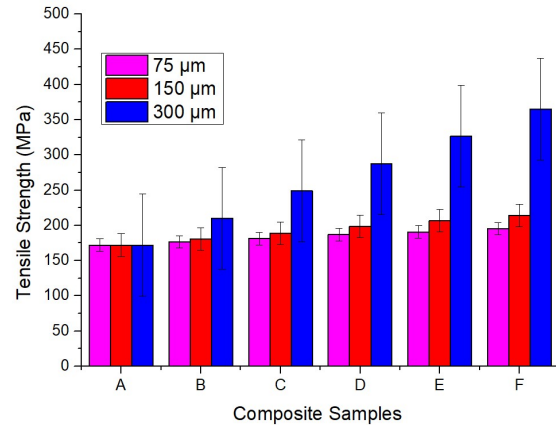


Fig. 3. Graph of variation of Tensile strength with wt% of PWS

### 3.3 Compressive Strength

The results of the compressive strength test on the composite are given in Fig. 4. From the result, it can be seen that the compressive strength of the composite increases as the weight fraction of PWS reinforcement increases. This is in line with findings in cases similar to this (Fu et al., 2008; Bhanu and Madhusudhan, 2015). The epoxy matrix is naturally brittle and therefore possesses good compressive properties, then when some amount of PWS particles containing  $\text{CaCO}_3$  are added to the matrix the compression is observed to be at an increase (Sahu et al., 2020). The grain size of the reinforcement also affected the compressive strength of the composite. Within the limits of the research work, the higher the grain size of the reinforcement particles, the greater the compressive strength of the ensuing composites. All the samples of the composite have a greater value of compressive strength than the ordinary epoxy resin without reinforcement.

From Fig. 4, It was noticed that the size of reinforcing particle has great impact on the compressive strength of the developed composites. The results obtained for sample E and F at 300  $\mu\text{m}$  are very close with 102.8 and 115.6 MPa respectively, while samples D, C and B recorded 87.3, 66.9 and 44.3 MPa respectively at 300  $\mu\text{m}$  whereas sample A with no reinforcement produced 14.3 MPa and this was uniform for all the microns used. Sample F at 150  $\mu\text{m}$  reinforcement produced 85.9 MPa compressive strength while samples E, D, C and B at the same 150  $\mu\text{m}$  produced



72.1, 59.4, 48 and 30.3 MPa respectively. The same trend was also noticed at 75 µm across all composite samples. Standard deviation for compressive strength values at different microns was taken at a scaling factor of 1. It was noted that the standard deviation of 21.38118 was recorded when 75 µm was applied, and 26.51601 standard deviation was recorded at 150 µm while 37.9636 standard deviation was computed at 300 µm for compressive strength values as depicted in Fig. 4.

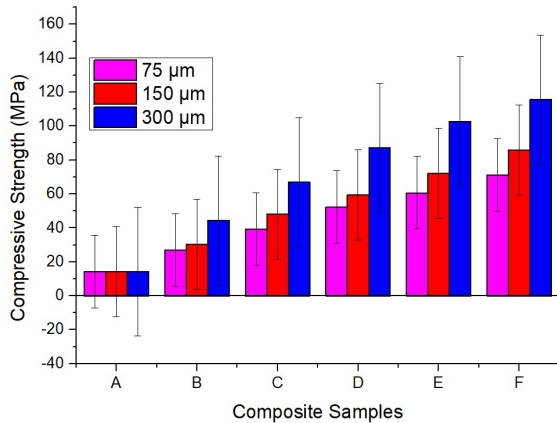


Fig. 4. Graph of variation of Compressive strength with wt% of PWS

### 3.4 Impact Strength

Fig. 5 shows the result of the impact strength test conducted on the Periwinkle shell reinforced epoxy resin composite. From the Fig. 5, it was revealed that fracture energy for sample F had 17.5 J at 300 µm which is far higher than the sample E of the same 300 µm which has 8.5 J while sample B recorded least value at 300 µm with 3.5 J. The same trend was noticed with 150 µm reinforcement, at sample F, it was 13.5 J impact strength which was lower than what was recorded at 300 µm. This can be attributed to the fineness of the particles and their compartments on the composite sample. The reduction in the impact strengths was noticed in all samples as the microns of the reinforcing particles decreases. The result shows that ordinary epoxy resin without reinforcement has 0 J impact strength but the impact strength increases directly proportional to both the grain size and weight fraction of the PWS reinforcement in the composite. Standard deviation for impact energy values at different microns was taken at a scaling factor of 1. It was noted that the standard deviation of 4.11603 was recorded when 75 µm was applied, and 4.7267 standard deviation was recorded at 150 µm while 5.96378 standard deviation was computed at 300 µm for impact energy values as illustrated in Fig. 5. In the study conducted by Bhanu and Madhusudhan (2015), it was established that good impact strength in polymer matrix composites can be attributed to good bonding strength between the filler and the matrix, as well as the flexibility of the interface molecular chain resulting in absorbing and dispersing of more energy and

effectively preventing crack initiators (Fatoba et al., 2021; Ikumapayi et al., 2020; Oladapo et al., 2020). The result, therefore, shows that PWS makes for good bonding strength with epoxy resin, and the strength increases as the wt% and grain sieve size of PWS increases within the limits of this work.

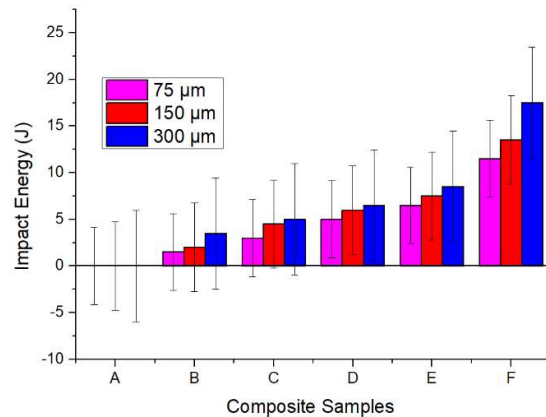


Fig. 5. Graph of variation of Impact strength with wt% of PWS

## 4. CONCLUSION

From the experiment carried out, results obtained and their discussions, the following findings can be established. The values of mechanical properties of hardness, tensile strength, compressive strength, and impact strength are greatly improved in the periwinkle shell (PWS) reinforced epoxy resin matrix composite compared to the values of those mechanical properties in ordinary epoxy resin without reinforcement. The values of mechanical properties investigated increased as the weight percentage and sieve size of the PWS increased within the limits of this research work. It was established that the highest values for hardness, tensile, compressive as well as impact strengths were obtained when reinforcing with 300 µm in all tested composites samples followed by 150 µm while 75 µm recorded the least values in all tested composites samples.

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