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Effect of Filler Content and Particle Sizes on the Mechanical and End-Use Properties of Eggshell Powder Filled Epoxy Polymer Composite

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Abstract

In pursuit of environmental sustainability, polymers filled with bio-particulates are replacing traditional plastic components. Epoxy Polymer composites of eggshell powder were prepared at filler contents of 10 wt%, 20 wt%, 30 wt%. the particle size of the eggshell powder was 75 µm, 180 µm, and 250 µm. The composite samples were prepared using the Carver Inc. Hydraulic Press (3851-0) by compression molding and the resulting composites were produced in sheets. Some mechanical properties of the composite were determined. the result showed that the eggshell powder improved the tensile modulus, flexural strength and impact strength of the polymer these properties increased with increase in the filler content and decrease in the filler size. But at 30 wt% a decrease in these properties were observed which can be attributed to agglomeration and stress concentration effects. Highlighting the fact that the dispersion and distribution of filler particles within the matrix play a crucial role in determining the mechanical properties. Inhomogeneities in mixing or particle clustering could lead to variations in the measured properties.

Keywords: Polymer composites; filler; particle size; mechanical properties.

I. INTRODUCTION

n recent years, the use of agricultural waste and renewable Iresources has grown in significance for sustainable development and the mitigation of environmental damage. The usage of natural materials for industrial applications is expanding due to environmental challenges such as waste management and the depletion of natural resources. The use of fillers in the polymer industry has drawn more attention over the past few decades. When added to polymers, fillers significantly improve their dimensional stability, impact resistance, tensile and compressive strength, abrasion resistance, thermal stability, and generally tailor material characteristics and contribute to sustainable practices. Agricultural waste was used as the reinforcing material for composites to attain the objective of recyclable and biodegradable composites [1-3].

In the middle of the 20th century, composite materials developed as a promising type of engineering material that offered fresh opportunities for contemporary technology [4]. Composites are materials made up of two or more parts that have distinctly different physical or chemical characteristics and continue to be distinct and recognizable in the final product [5]. A matrix of high-strength, low-stiffness fibers make up most composites. Typically, the objective is to produce a strong, stiff component that is frequently low density [6]. Any multiphase material that demonstrates a sizable amount of the traits of its two component phases such that a greater synergy of traits is created is referred to as a composite material [7-10].

The food sector produces a lot of eggshells, which is a major waste product. According to [11], eggshell is a biomaterial made up of 5% organic compounds such (Al_2O_3, SiO_2, S, Cl, P, and Cr_2O_3, MnO) and 95% calcium carbonate in the form of calcite. The generic eggshell structure is a protein lining covered with mineral crystals, often made of a calcium compound like calcium carbonate. These qualities make eggshell a strong contender for applications requiring cheap, lightweight composite materials in large quantities, such as those found in the automobile sector, vehicles, houses, offices, and factories. While they are vital materials for various applications, some businesses spend a significant amount of money on their disposal. According to [12], heavy metal from polluted water was removed using Ca^{2+} from an eggshell solution. It has been proven that eggshells may be used in place of cement in some situations [13].

Epoxy resins, thermosetting polymers, offer outstanding mechanical, chemical, and adhesive properties after curing. When combined with reinforcing components, epoxy resins create composites with greater strength, stiffness, and other desirable properties [14-15]. Epoxy resins are thermosetting polymers with a wide range of applications that serve as the matrix for epoxy-based composites. Epoxy resins are compatible with a range of reinforcing elements and can easily adjusted or changed to obtain specific qualities. Depending on the required qualities of the composite, the reinforcing elements employed in epoxy-based composites might be fibers, particles, or flakes. Epoxy-based polymer composites are ideal for a variety of demanding applications because they combine great mechanical performance with outstanding chemical resistance and design flexibility. By choosing the suitable reinforcing materials, fibre orientations, and manufacturing procedures, epoxy composites' unique qualities may be customized to fulfil certain performance requirements [16-17].

When assessing a composite material's structural integrity and functional performance, its mechanical qualities are of utmost significance. A thorough grasp of the interaction between these factors and their influence on mechanical characteristics will be attained by deliberately adjusting the concentration of eggshell filling and the particle sizes. The main goal of this study is to determine how the amount of filler and the size of the particles affect the mechanical and end-use characteristics of epoxy polymer composites supplemented with eggshell powder.

II. MATERIALS AND METHODS

A. Eggshell Sample Preparation

The eggshells were sourced locally from a pastry vendor in Zaria, Kaduna state and washed with hot water to remove them from membrane. Afterward, they were washed with Acetone and then methanol and dried again for two (2) days. The eggshell (EGS) was milled using a top-down method approach according to [18]. The EGS was milled using the pulverizing machine, then the powder was sieved with a 75 µm,180 µm and 250 µm sieve respectively. Then taken for further analysis.

B. Composite Sample Preparation

The composite samples were prepared in the polymer laboratory in NILEST Zaria using the Carver Inc. Hydraulic Press (3851-0) by compression molding. This was done by mixing epoxy polymer and hardener in a ratio of 2:1 in a beaker. Upon achieving a homogenous mixture, the filler was added accordingly and further mixed for 3 minutes until a uniform filler distribution was obtained. The composite mixture was poured into a mold measuring 240 mm by 180 mm by 3.2 mm, which had already been prepared with aluminium foil and released oil for easy removal of the composite after formation. The sample was cured on a hot hydraulic press at 130°C and a pressure of 2.5 MPa. The curing times of 90 minutes were used based on the experimental design for each sample. At the end of the curing time, the mold was opened, the sample was carefully removed from the mold, placed on a flat surface, and allowed to stand for 24 hours to completely cure at room temperature. The samples produced were labelled in preparation for further analysis. The prepared samples are described in Table I.

Table 1. Epoxy + Eggshell particles sample preparation description.

Sample no	Sample description	Sample description	Sample description
1	$Epoxy + 10\ wt\% + 75\ \mu m$	$Epoxy + 10\ wt\% + 150\ \mu m$	$Epoxy + 10\ wt\% + 250\ \mu m$
2	$Epoxy + 20\ wt\% + 75\ \mu m$	$Epoxy + 20\ wt\% + 150\ \mu m$	$Epoxy + 20\ wt\% + 250\ \mu m$
3	$Epoxy + 30\ wt\% + 75\ \mu m$	$Epoxy + 30\ wt\% + 150\ \mu m$	$Epoxy + 30\ wt\% + 250\ \mu m$

C. Spectroscopic characterization of eggshell powder

1) Fourier transform infra-red (FTIR) analysis.

This was carried out on the eggshell powder in the multipurpose laboratory in the department of chemistry in ABU Zaria to determine the organic components and chemical bond in the filler. A sample specimen of approximately 2 g was placed in the Pelkin Elmer frontier FTIR machine. The result gives the FTIR spectra of the sample from 700 to 3700 cm^{-1}.

2) X-ray fluorescence (XRF) analysis.

XRF analysis of the eggshell powder was carried out. The sample quantity of approximately 2 g was formed into pellets with hydraulic press and placed into the chamber of the

machine and sealed [19]. The machine was allowed to run for 1000 s at a voltage and current of 30 KV and 50 µA respectively.

3) Scanning electron microscopy (SEM-EDX)

The surface morphology of the eggshell particles was studied using the scanning electron microscope (SEM-EDX) this will allow for the identification and quantification of the elemental composition of the filler material. The composite sample specimen of dimension 20x20x3mm was also produced for the test. The samples were polished and firmly held with the aid of a sample holder in the analysis chamber of the SEM. The scanning was performed for three different magnifications.

D. Mechanical Characterization

1) Density.

The Density of the composites was determined by measuring their respective mass and volume. Sample specimens of dimension $20 \times 20 \times 5\ mm^3$ were produced for the test. The mass was determined with the aid of the weight balance to an accuracy of four decimal places. The volume was found using Archimedes' principle. The mass divided by the volume thus determined gives a measure of the average density of the sample [6].

The density of each sample was determined using (1).

$$D = \frac{m}{V}\left(\frac{g}{cm^2}\right) \quad (1)$$

Where m is the mass of the composite sample, V, the volume of the sample.

2) Flexural strength analysis.

The samples specimens were produced for three specimens of each composite with dimensions of 100 mm by 30 mm for the test (Fig. 1). This was carried out using the universal material testing machine Enerpac 100 kW capacity, cat.no 21 (Plate 1). The test sample was placed between three-point bending rollers with a gauge length of 80mm and force (hydraulic handle) was applied until the sample ruptures.

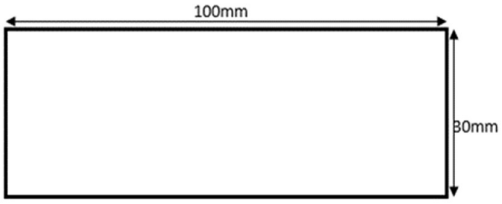

Fig. 1 Flexural test sample dimension

Flexural strength is the ability of the composite to withstand bending. This was evaluated by determining the modulus of rupture (MOR) and modulus of elasticity (MOE) using (2) and (3).

$$MOR(\sigma_f) = \frac{3F}{2bd^2}(MPa) \quad (2)$$

$$MOE(E_f) = \frac{FL^3}{4bd^3D}(MPa) \quad (3)$$

Where F is the force (load) (N), L, the gauge length (mm), b, the width (mm), d, the thickness (mm) and D, the deflection (mm).

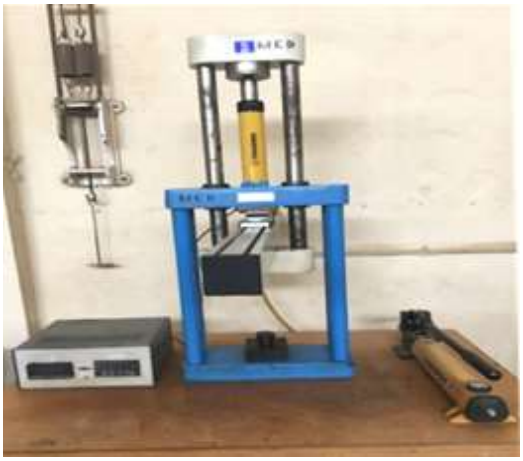

Plate 1 Universal Material testing Machine.

3) Tensile strength Analysis.

Three dumbbell shaped samples of the specimen were produced for each composite combination with the dimensions as shown in Fig 2. The Hounsfield tensiometer type 'W'S/No 3179, 20 kN capacity (Plate 2) was used for this test.

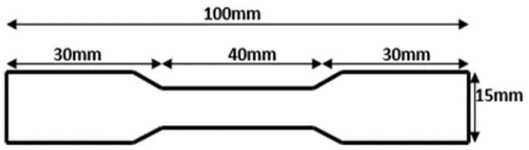

Fig. 2 Tensile test sample dimension

Plate 2 Hounsfield Tensiometer.

The dumbbell part was clamped to jaws of the machine and the extension was produced within the gauge span of the specimen (40 mm). From the plot of the force-extension from the tensiometer the ultimate tensile strength (UTS), strain, percentage elongation and modulus of elasticity was determined using (4) to (7) respectively [21].

$$UTS(\sigma) = \frac{Force(N)}{cross-sectio \quad area\ (mm^2)}(MPa) \tag{4}$$

$$strain(\xi) = \frac{\Delta L}{L} \tag{5}$$

$$\%\ elongation = \frac{\Delta L}{L} \times 100\% \tag{6}$$

$$modulus\ of\ elasticity\ E = \frac{UTS}{\xi}(MPa) \tag{7}$$

Where, F is the force, L, the length and ΔL the extension.

III. RESULT AND DISCUSSION

A. Spectroscopic characterization of filler

1) FTIR Analysis.

Fig. 3 illustrates the FTIR of the eggshell powder in transmittance mode, which is used to determine the chemical composition of the eggshell from the characteristic frequency of the spectra. The acquired spectra are consistent with those reported by [22-24], demonstrating the reproducibility of the eggshells' chemical composition. The C=O bonds from carbonate are represented by the weak bands at 1796 cm-1 and 1643 cm^{-1}. The C-O stretching and bending modes of calcium carbonate can be distinguished by two distinct infrared bands at 1405.2 cm^{-1} and 872.2 cm^{-1} [24]. A peak at 2512 cm^{-1} at the fingerprint region is caused by OH in $Ca(OH)_2$ that forms when CaO absorbs water, and a sharp band at 712 cm^{-1} is related to Ca-O bonds [22].

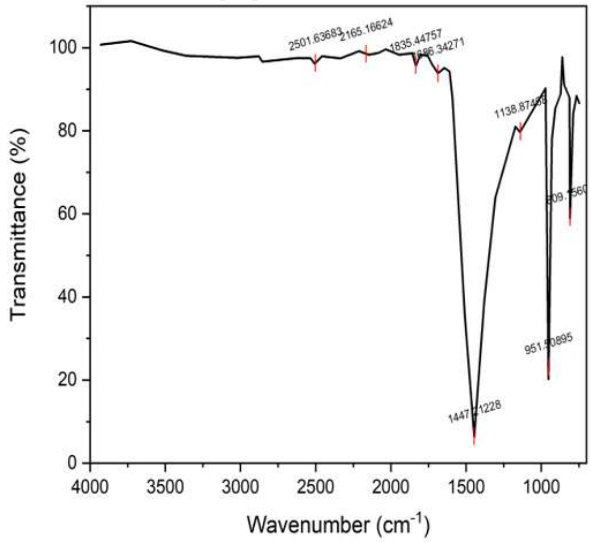

Fig. 3 FTIR spectrum of eggshell powder.

2) SEM-EDX Analysis.

Plate 3 (a), (b), and (c) display the observable morphology of the eggshell powder analyzed using SEM-EDX at various particle sizes. The images were captured at magnifications of 3000x, 4000x, and 5000x, for each particle size with working distances of 10.0 mm, 10.5 mm, and 9.6 mm, respectively. The horizontal field widths for the corresponding images were 122 µm, 122 µm, and 130 µm. A voltage of 20 kV was applied during the imaging process. The micrograph depicts a surface consisting of particles of varying sizes, as observed from the image. The eggshell powder exhibits an average particle length of 27 µm at a magnification of 3000x. To verify the elemental distribution within the eggshell powder, additional EDX measurements were conducted. Various regions of interest were selected during the measurement process, and the resulting peaks are presented in Fig. 4. The measured atomic percentages of C, O, Si, Fe, Mg, Ca, and P are 3.0, 20.0, 42.0, 3.0, 4.0, 22.6, and 1.30, respectively.

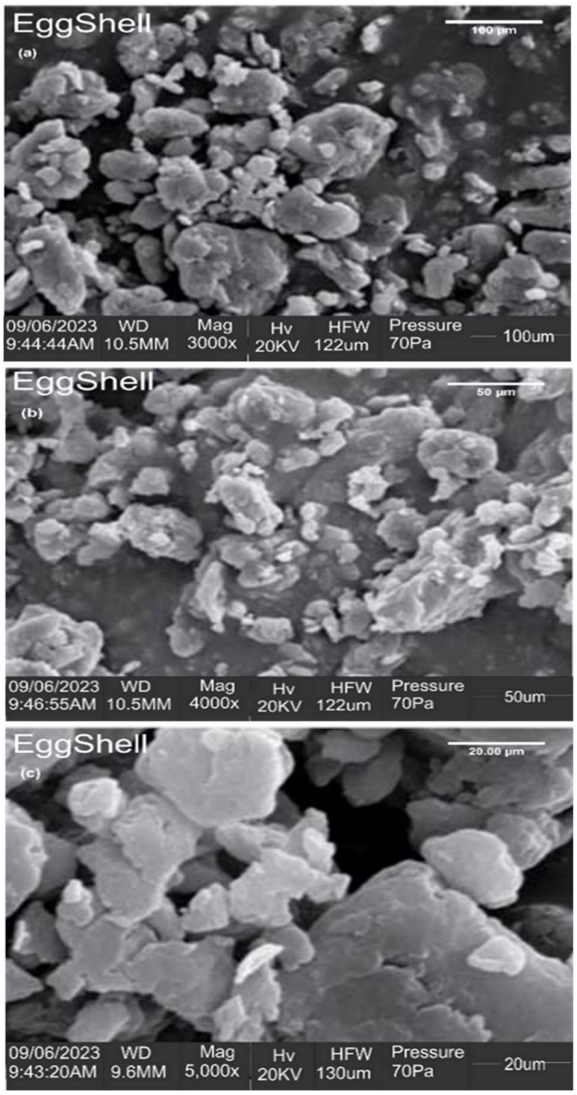

Plate 3 (a) Microstructure of eggshell powder of 75µm at 3000 magnification (b) Microstructure of eggshell powder of 150 µm at 4000 magnification (c) Microstructure of eggshell powder of 250 µm at 5000 magnification.

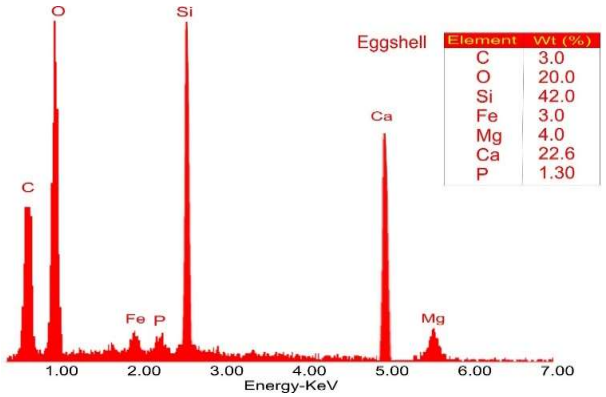

Fig. 4 EDX microstructure of eggshell powder.

3) XRF Analysis

XRF analysis of the eggshell carried out gave the elemental configuration of the eggshell given in Table II. the major compounds of the eggshell are calcium oxides, magnesium oxides and aluminium oxides of about 95%.

Table II XRF of Eggshell powder

Chemical composition	Percentage (wt.%)	Chemical composition	Percentage (wt.%)
SiO_2	0.641	CuO	0.038
V_2O_5	0.010	SrO	0.191
MnO	0.008	WO_3	0.002
Fe_2O_3	0.090	SO_3	0.598
Co_3O_4	0.012	ZnO	0.003
Ta_2O_5	0.003	CaO	92.932
NiO	0.020	MgO	2.589
K_2O	0.041	Ag_2O	0.003
BaO	0.025	Cl	0.531
Al_2O_3	2.035	SnO_2	0.225

B. Mechanical characterization of composite

The developed composites are shown in Plate 4. Plate 5 and 6 show the dump bell shapes carved out of the composite for the tensile strength test and flexural test respectively.

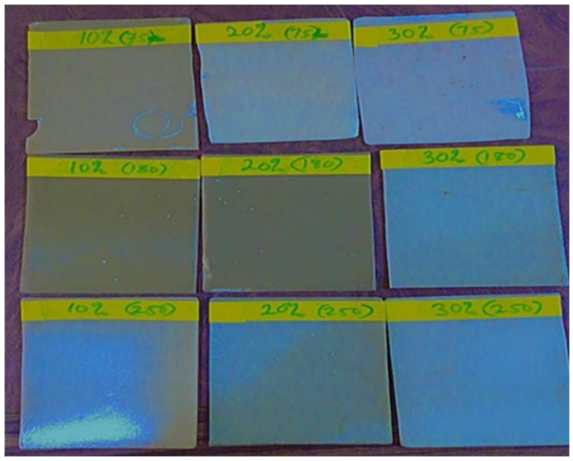

Plate 4 Developed Composite Samples.

Plate 5 Dumbbell sample for Tensile strength analysis.

Plate 6 Rectangular shaped sample for flexural test.

The physical and mechanical properties of the produced composite with varying filler content and particle size were analysed in the following subsections.

1) Density

The result of the density measurements for the composite samples are presented in Fig. 5, depicting data for particle sizes of 75 µm, 180 µm, and 250 µm, each at filler contents of 10 wt.%, 20 wt.%, and 30 wt.%, respectively. The figure illustrates a consistent trend: as the filler content increases from 10 wt.% to 30 wt.% while keeping the filler size constant, there is a noticeable decrease in density. This suggests that the overall density decreases with the addition of more filler to the composite material. This phenomenon can be attributed to the fact that the filler material is typically less dense than the matrix material, leading to a reduction in overall density as it occupies more space within the composite. When examining the same filler content, it becomes apparent that particles with larger sizes tend to exhibit lower density values, possibly due

to increased void spaces between the larger particles. This observation aligns with the findings of [20] and [25].

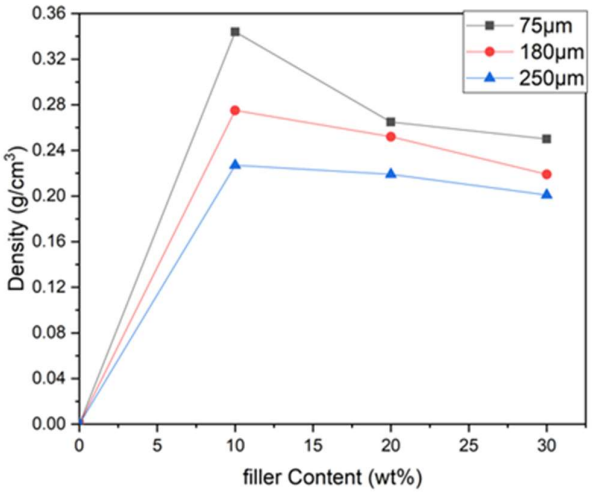

Fig. 5 Density versus filler content for composites at different filler particle size.

2) Flexural Strength Analysis

Flexural testing was carried out on the specimens to determine the bending strength of the composite materials under three-point loading condition, which indicates the materials stiffness when being flexed, according to the ASTM D790 test method. The mold used for this specimen is based on the flexural bar dimension and thickness outlined by ASTM international with dimensions of 100 mm by 30 mm (Plate 6). The flexural testing was conducted using the universal material testing machine Enerpac 100kW capacity, cat.no 21 (Plate 1).

- *Modulus of Rupture (MOR)*

Fig. 6 depicts the influence of varying filler content and particle size on the Modulus of Rupture (MOR) in eggshell/epoxy composites. The graph reveals that MOR increases linearly as filler content rises from 10 wt.% to 30 wt.%. The MOR values range from 30-35 MPa at 10 wt.% filler content, reaching their highest values of 44-50 MPa at 30 wt.% filler content. This trend aligns with previous studies by [26] and [27]. A decrease in MOR is observed with increasing particle size. For instance, at 10 wt.% filler content, the 250 µm particle size exhibits higher MOR than the 75 µm particle size. Similarly, at 20 wt.% and 30 wt.% filler contents, there is a decrease in MOR associated with smaller particle sizes. Comparing MOR values at the same filler content, larger particle sizes, such as 250 µm, consistently result in higher MOR. For example, at 10 wt.%, MOR increases from 30.63 MPa (70 µm) to 35.83 MPa (250 µm), indicating that larger filler particles contribute to greater mechanical strength in the composite. These findings align with the observations of [28], emphasizing the role of filler content and particle size in influencing the MOR of composite materials. The data provides valuable insights for optimizing composite formulations to meet specific mechanical strength requirements for various applications.

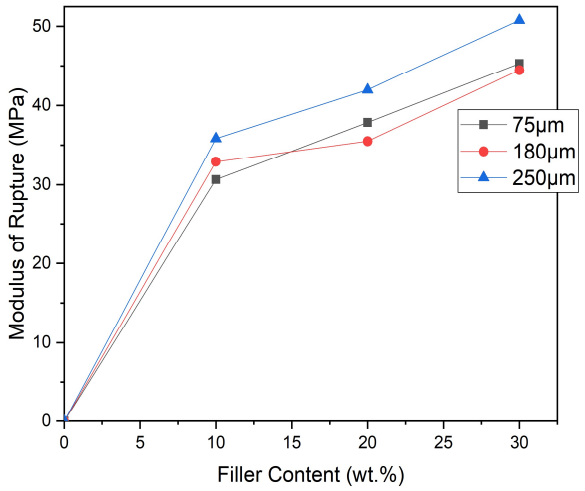

Fig. 6 Variation of Modulus of rupture with filler content

- *Modulus of Elasticity (MOE)*

Fig. 7 depicts the Modulus of Elasticity, concerning the increase in weight percentage of the reinforcement. Unlike the previous analysis for flexural strength and modulus of rupture, a clear trend emerges in the MOE values concerning filler content. Increasing filler content consistently leads to an increase in MOE, a trend that holds true across all particle sizes. For example, at a particle size of 75 µm, the MOE values increase from 1447.1 MPa (10 wt.%) to 2268.7 MPa (30 wt.%). Similar trends are observed for other particle sizes. This indicates that higher filler content contributes to increased stiffness and elasticity in the composite material.

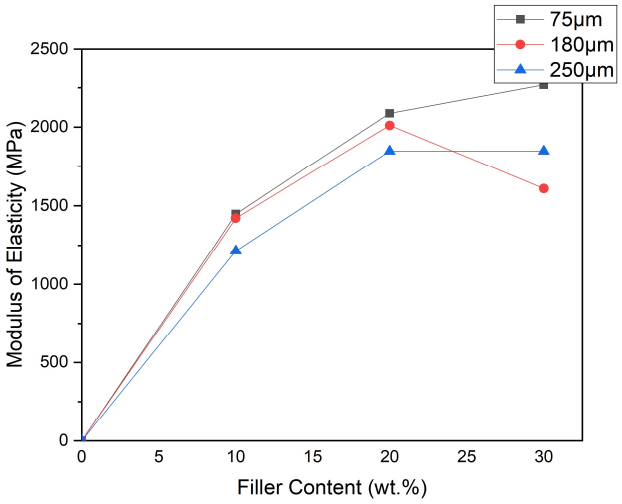

Fig. 7 Variation of Modulus of Elasticity with filler content.

The effect of particle size on MOE varies depending on the filler content. At 10 wt.% filler content, there is a trend of decreasing MOE with increasing particle size. For instance, the MOE decreases from 1447.1 MPa (75 µm) to 1213.7 MPa (250 µm). However, at 20 wt.% and 30 wt.% filler contents, no consistent trend with particle size emerges, resulting in varying MOE values. This sudden decrease in MOE indicates that the saturation level of the filler matrix composition is determined by filler agglomeration, as reported in previous works by [29] and [30] these observations suggest that the influence of particle size on MOE is more pronounced at lower filler contents.

3) Tensile strength test

Three dumbbell shaped samples of the specimen were produced for each composite combination with the dimensions as shown in Plate 5. The Hounsfield tensiometer type 'W'S/No 3179,20kN capacity was used for this test.

- *Ultimate Tensile strength (UTS)*

The ultimate tensile strength of the eggshell/Epoxy composite is depicted in Fig. 8 as it varies with filler content and particle size. The Ultimate Tensile Strength (UTS) values illustrate a linear increase in UTS as filler content increases, ranging from 10 wt.% to 30 wt.%. A similar trend to the present study can be observed in studies conducted by [31] and [26].

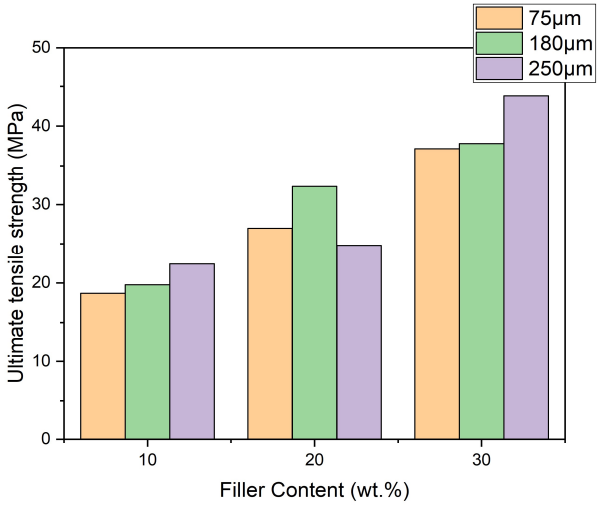

Fig. 8 Variation of Ultimate Tensile strength with filler content.

From this, we can deduce that the increase in tensile strength with increasing filler content may be attributed to an increased packing fraction and reduced void content in the composite, as suggested by [32]. These results indicate that higher filler content contributes to greater stiffness and elasticity in the composite material. The influence of particle size on MOE varies with both filler content and particle size. At 10 wt.% filler content, there is a general trend of increasing MOE with increasing particle size. For instance, the MOE values rise from 18.68 MPa (70 µm) to 22.39 MPa (250 µm). However, at 20 wt.% and 30 wt.% filler contents, the trend becomes less consistent, and the MOE values fluctuate. This inconsistency could be attributed to poor surface adhesion of the reinforcing fillers with the epoxy matrix. Additionally, aggregations and agglomerations of the reinforcing fillers, resulting from the increased interfacial area within the epoxy matrix, may disrupt the filler-matrix bonding, as noted in studies by [33] and [34].

- *Strain (ξ)*

Fig. 9 depicts the relationship between strain and the filler content. There doesn't seem to be a consistent trend in the strain values with respect to filler content. In some cases, increasing filler content leads to an increase in strain (e.g., 10 wt.% at 75 µm and 180 µm), while in other cases, the relationship is not clear (e.g., 20 wt.% at 75µm and 180 µm). This suggests that the relationship between filler content and strain is complex and might be influenced by multiple factors.

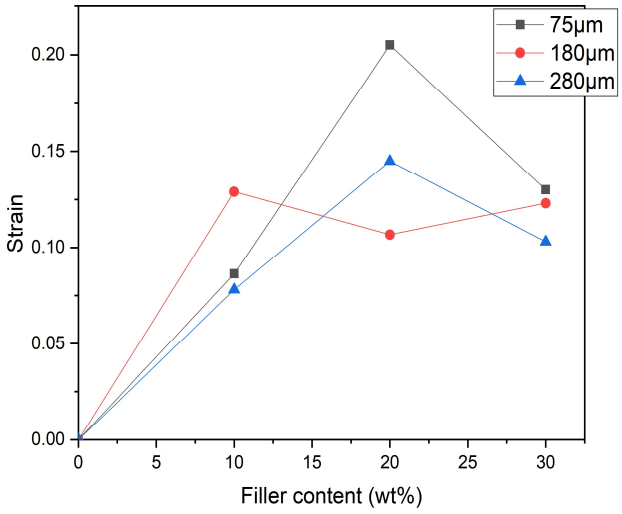

Fig. 9 Variation of Strain with filler content

Similar to filler content, there is no clear consistent trend in the strain values with changing filler size. The strain values do not consistently increase or decrease with filler size changes for a given filler content. This indicates that filler size alone may not be the primary determinant of strain behaviour. When considering the interaction between filler content and size, again, there isn't a straightforward pattern. The strains for a specific filler size vary with different filler contents. This complexity suggests that multiple factors, including the mechanical properties of the matrix and filler materials, their interaction, and stress distribution, play a role in determining the strain behaviour. The strain values are crucial for understanding how a material deforms under applied loads. The variations in strain with filler content and size highlight

the importance of characterizing the material's deformation behaviour in relation to its intended application.

Percentage Elongation

Fig. 10 depicts the relationship between Percentage elongation, the filler content and particle size. The percentage elongation values show variations with changing filler content. The tensile elongation shows illustrates that, as filler content increase, percentage elongation shows a decrease for all composites from 10 wt.% to 30 wt.%. Based on the observations in Fig. 9, it's apparent that the stiffness of the samples has an impact on the percentage of elongation. Therefore, one can hypothesize that the stiffer the specimen, the lower the percentage of elongation at the point of breakage. Additionally, it can be inferred that the decrease in tensile elongation with increasing filler content may be attributed to the formation of agglomerations, leading to poor adhesion. The influence of particle size on percentage elongation is evident across different filler contents. Larger particle sizes, such as 250 µm, tend to result in lower percentage elongation compared to smaller sizes, especially at higher filler contents (20 wt.% and 30 wt.%). For instance, at 10 wt.% filler content, the percentage elongation decreases from 20.5% (75 µm) to 15.4% (250 µm). This suggests that larger particles may limit the material's ability to deform plastically before fracture [34].

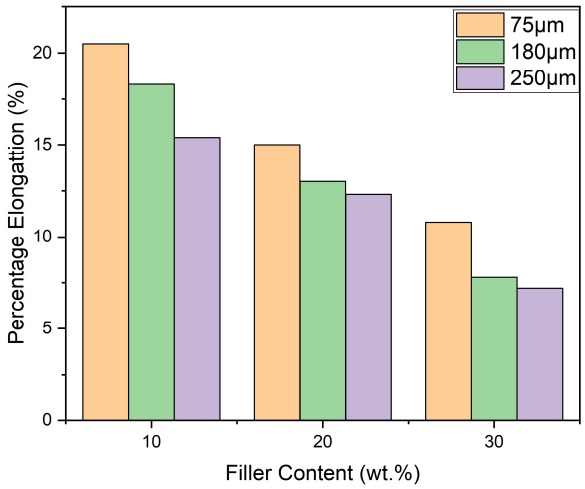

Fig. 10 Variation of Percentage elongation with filler content

Young's Modulus

Fig 11 depicts the Young's modulus of elasticity with variations in filler content across different particle sizes. Young's modulus exhibits a behaviour similar to that of the flexural strength graph. It can be observed that increasing the filler content from 10 wt.% to 30 wt.% results in a noticeable increase in Young's modulus for all particle sizes. For example, at a particle size of 75 µm, Young's modulus values increase from 181.2 MPa (10 wt.%) to 216.5 MPa (30 wt.%). Similar trends are observed for other particle sizes. This trend suggests that higher filler content contributes to greater stiffness and reduced deformation in the composite material, which is consistent with previous studies by [26], [27], and [35]. The impact of particle size on Young's modulus varies across different filler contents. Larger particle sizes, such as 250 µm, tend to result in higher Young's modulus values compared to smaller sizes, especially at higher filler contents (20 wt.% and 30 wt.%). For instance, at 10 wt.% filler content, Young's modulus increases from 181.2 MPa (75 µm) to 240.6 MPa (250 µm). This indicates that larger particles contribute to increased material stiffness and reduced deformation

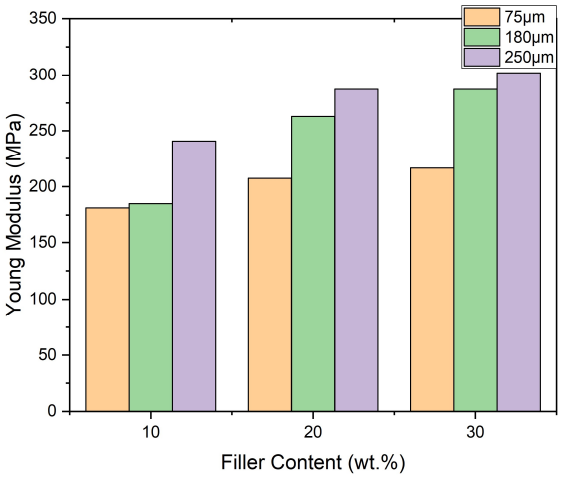

Fig. 11 Variation of Young Modulus with filler content

IV. CONCLUSION

This study has provided valuable insights into the mechanical properties of epoxy composites with varying particle sizes and filler contents. It also established the importance of filler quantity and particle size in controlling mechanical behavior of composite materials. The results obtained emphasized the complex relationships among these factors and their collective impact on the mechanical behavior of the material. The analysis of flexural and tensile behavior revealed noticeable changes with filler content increased from 10% to 30%. Higher filler content was shown to initially improve mechanical qualities, but there was a critical threshold (usually at 20 wt.%) beyond which the mechanical properties started to decrease. The consequences of agglomeration and stress concentration could be attributed for these occurrences. It's interesting to note that the best particle size and filler ratio combinations produced the best mechanical performance. For instance, various particle sizes produced the maximum UTS or MOR values at particular filler concentrations. These results highlight the significance of adjusting filler quantity and particle size to get the

necessary mechanical characteristics for certain applications. The study unveiled trade-offs between different mechanical properties, revealing that enhancing one property might come at the expense of another. Therefore, a balanced consideration of UTS, MOE, and MOR is essential for selecting the optimal composite configuration. A major factor in defining the mechanical characteristics of the matrix is the dispersion and distribution of filler particles. Various changes in the observed parameters might be caused by inhomogeneities in mixing or particle clustering.

DECLARATION OF COMPETING INTEREST

The authors declare that there are no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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