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Influence of Particle Size and Surface Treatment on Mechanical Properties of Bambara Nut Shell and Cowpea Husk-Polyester Composites

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ABSTRACT

Bambara nut (Vigna subterranea) is a grain legume native to sub-Saharan Africa, with its shell remaining an underutilised agricultural waste. Similarly, cowpea (Vigna unguiculata L. Walp), a widely cultivated legume crop in Africa, has husks with untapped potential for composite applications. Despite their availability, using Bambara nut shell and cowpea husk as fillers in polyester resin composites has been minimally explored. This study aims to develop natural particulate-based composites using Bambara nut shell and cowpea husk fillers of varying particle sizes and loadings within an unsaturated polyester resin matrix. The physical, mechanical, structural, and morphological properties of these composites were investigated. The study revealed that optimal composite properties were achieved at a filler loading of 10 wt% for both Bambara nut shell and cowpea husk. Treatment of the fillers with a 5% alkaline solution significantly enhanced the composite performance. Bambara nut shell-filled composites exhibited superior hardness, whereas cowpea husk-filled composites demonstrated better tensile and flexural properties, higher crystallinity index, lower density, and more pronounced peak intensities. These results suggest that the developed composites are suitable for non-load-bearing applications, particularly in packaging and indoor partition board systems.

Keywords: *Bambara nut shell, Cowpea husk, Polyester resin, Composite, Surface treatment, Mechanical properties*

Introduction

There is a global problem of plastic recycling, as millions of tons of plastic are manufactured every year, and only a small percentage is recycled annually. The remaining waste is deposited in landfills or dispersed in the oceans (Matkó *et al*., 2005). Such disposal affects wildlife, the food chain, groundwater, and air quality in a negative manner (Rajmohan *et al*., 2019). As a result of this, the world is becoming more conscious of environmental conservation and its safety.

The objective of composite materials is to combine diverse component elements to achieve desired characteristics. As a result, a portion or structural component is formed that outperforms each of the components separately and potentially even better than just adding the attributes of each element (Sun *et al*., 2019). Composites derived from natural fibres have been extensively studied, and waste fibre composites are assumed to be environmentally friendly since the use of such fibres reduces pollution in the environment (Wong *et al*., 2010; Alsaeed *et al*., 2013). Besides, the bio-degradation advantage of natural fibre, composites of natural fibres are characterised by low weight, improved mechanical properties, low production costs, longer life of the processing equipment, and minimal health hazards (Mohammed *et al*., 2015; Elsabbagh *et al*., 2009).

Bambara nuts (Vigna subterranean (L.) Verdc.) are grain legumes indigenous to sub-Saharan Africa. its potential as filler materials in epoxy nano-structured composites was carried out by Eze *et al*. (2022). It has been used in the development of composites for structural board applications (Gital and Raji, 2022). Bambara nut shell was also observed to improve the mechanical properties of polyurethane foam (Uzoma *et al*., 2015) and polyethylene (Azeez *et al*., 2013), but its influence on the properties of polyester resins has not been fully explored.

Cowpea (Vigna unguiculata L. Walp) is a common legume crop grown mostly in Africa and is consumed worldwide by humans and animals (Abebe *et al*., 2022; Obembe *et al*., 2020). Its application in composite formulations is still developing. Anosike-Francis *et al*. (2022a) discussed the utilisation of cowpea husk and recycled polypropylene, in the production of wood-based composites. The results indicated that the composites met the required standards for particleboard and exhibited potential for applications in wall panelling and flooring.

The chemical modification of natural fibres has been extensively studied for application in polymer composites (Ahmad *et al*., 2019; Jonoobi *et al*., 2009).

The modification aims to reduce the hydrophilic nature of the fibres to improve adhesion with the polymer matrix (Phiri *et al*., 2020). Based on the literature, it has been observed that chemical surface treatment provides better dispersion and strong adhesion characteristics to natural fibres when incorporated into a thermosetting polymer matrix (Rajan *et al*., 2023). This conclusion is based on the results of mechanical, thermal, and morphological studies to determine their reinforcing effects in thermosetting polymers.

Despite all the various literature, there is little or no research on the use of cowpea husk and bambara nut shells of different sizes and loadings bonded in polyester resin composite. The aim of this study is thus to develop natural fibre-based composites using bambara nut shell and cowpea husk (agricultural wastes) as fillers in unsaturated polyester resin with improved properties, to investigate their physical, mechanical, structural and morphological properties of the composite.

Materials and Methods

Materials

The chemicals used are of analytical grade and were supplied by Sigma Aldrich, Burlington, Massachusetts, United States: Polyester resin was supplied by Nycil Nig. Ltd, Ikeja, Lagos, Diethylenetriamine (hardener), Methyl ethyl ketone peroxide (MEKP) (catalyst), Cobalt naphthalene (accelerator), Sodium hydroxide pellets, Aluminium foil (mould releasing agent), Acetic acid. Bambara nut shell (Plate X) and cowpea husk (Plate XI) used as fillers for the research were sourced from the Institute of Agricultural Research (IAR), Ahmadu Bello University, Zaria, Nigeria.

Equipment and Tools

Table 1: list of equipment and their location

S/N	Equipment	Location
1	Scanning Electron Microscope (SEM) (JOEL-JSM 7600F, Japan)	Rolab Research and Diagnostic Laboratory, Ibadan
2	Benchtop X-ray Diffractometer	NLNG Laboratory, Ahmadu Bello University, Zaria
3	Electronic balance (Type: LP313, UK)	Polymer and Textile Engineering Department, Ahmadu Bello University, Zaria
4	Tensile Strength Test machine (Model EM2101-T7, China)	Polymer and Textile Engineering Department, Ahmadu Bello University, Zaria
5	Universal/Electronic flexural test machine (model number HD B6I5A-S, China)	Rolab Research and Diagnostic Laboratory, Ibadan
6	Charpy Impact testing machine (Resil Impactor Junior Series, Ceast Torino, Italy)	Nigeria Institute for Leather Science and Technology (NILEST), Zaria
7	Microvickers Hardness Tester (Model: MV-PC, Serial No.: 07/2012-1329, China)	Shell Professorial Chair Laboratory, Ahmadu Bello University, Zaria
8	Thermogravimetric Analyser (TGA) (Perkin Elmer TGA4000, Perkin Elmer Inc., USA).	Centre for Genetic Engineering and Biotechnology, Federal University of Technology, Minna

Materials Preparation and Moulding

Preparation of the fillers

The Bambara nut husk was separated from the nut manually; thereafter, it was washed and cleaned to remove contaminants. While the cowpea husk was obtained from IAR, Zaria, it was carefully cleaned, and all contaminant was removed. However, both fillers were then dried and ground with a hammer mill to obtain filler powder. The fillers were made to pass through a wire mesh screen to obtain different particle sizes of 150 µm and 300 µm. However, some parts of both particulate fillers were modified by alkali treatment (5% sodium hydroxide) to obtain treated and untreated fillers.

Alkali Treatment of the Fillers

The fillers were surface-treated with solutions of NaOH at a concentration of 5% for 4 hours at room temperature (27 ±2 ºC), followed by neutralising the basic solution with 1% Acetic acid, washing and soaping in distilled water till neutral to litmus paper and then drying. The filler was then oven-dried for 24 hours at about 70 °C before use to reduce the moisture content. Samples were thereafter stored in a sealed container before compounding.

Compounding of the Fillers with Resins

The formulation for the development of Bambara nut shell and Cowpea husk thermoset polymer Composites is presented in Table 2.

Composite Preparation

The unsaturated polyester resin was mixed with the MEKP catalyst and naphthalene cobalt accelerator in the ratio of 100:1:1 while the epoxy resin and the hardener were mixed in the ratio of 2 to 1 based on the supplier's instructions. The resins were filled with the predetermined weight of the Bambara nut shell and cowpea husk fillers for both the sodium hydroxide-treated and untreated samples, thoroughly mixed and transferred into the glass mould using the casting technique. A filler weight fraction of 0-50 wt% at 10 wt% intervals was used during the compounding process, as shown in Table 2. This was obtained using a mould with dimensions of 120 mm x 120 mm x 4 mm for length, width, and thickness, respectively. It was left to cure for 24 hours at an ambient temperature of 27 ºC. Post-curing was done for 1 hour at 60 ºC before they were cut into different shapes according to the required ASTM standard for analysis, as shown in Fig. 1.

Table 2: Formulation of Bambara nut shell and Cowpea husk thermoset polymer Composites

Bambara nut shell filler loadings (wt%)		**Cowpea husk filler loadings (wt%)**	
UP/Treated and Untreated Bambara (150 µm)	UP/Treated and Untreated Bambara (300 µm)	UP/Treated and Untreated Cowpea (150 µm)	UP/Treated and Untreated Cowpea (300 µm)
0	0	0	0
10	10	10	10
20	20	20	20
30	30	30	30
40	40	40	40
50	50	50	50

Key: UP = Unsaturated Polyester

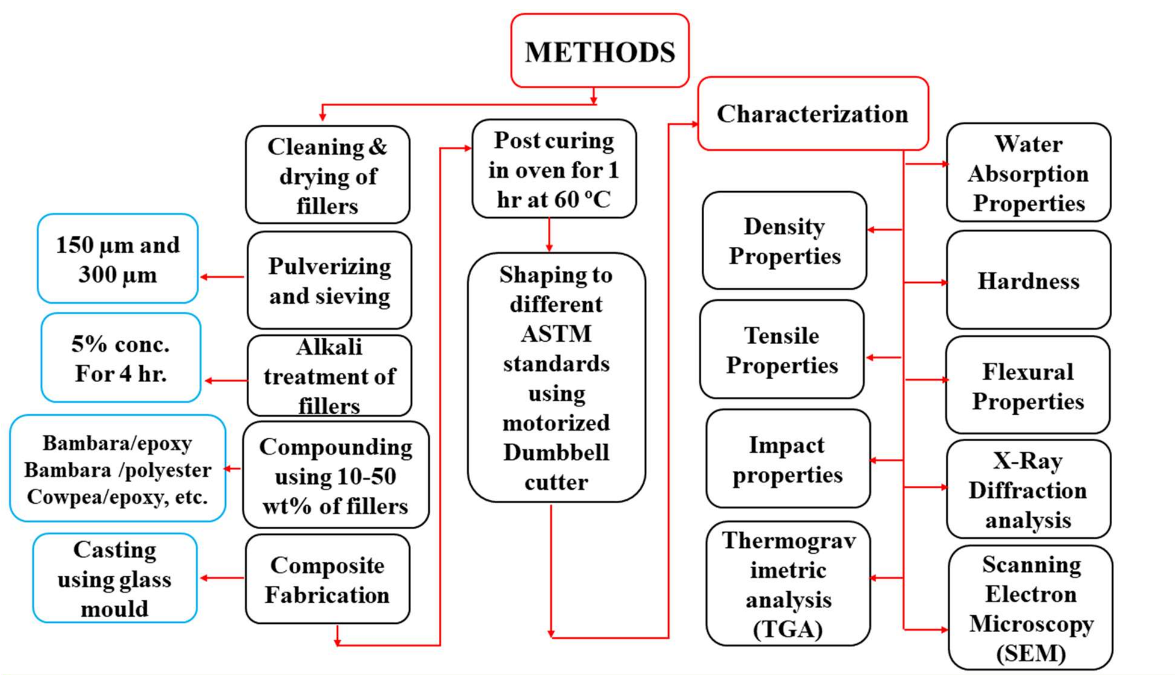

Figure 1: Flowchart Representation of the Composite Fabrication Process and Analysis

Composite Characterization

Physical Properties Characterisation

1. Density

ASTM D792 (08.01) standard procedure was used to determine the densities of the composite samples with 30 mm x 30 mm x 4 mm dimensions. Each specimen was weighed using a balance to get the individual weights in grams. The volume of each plastic sample was determined using the displacement method in the water medium. The bulk densities of the composites were finally calculated using Equation 1 for the average of five (5) specimens per sample.

$$\rho = \frac{m}{v} \quad \ldots\ldots.. (1)$$

where: ρ = density (g/cm^3), m = mass of sample (g), v = volume of sample (cm^3)

2. Hardness test

Hardness values were determined using Microvickers Hardness Tester (model: MV1-PC (Serial No.: 07/2012-1329) using the HV scale according to ASTM E384 (03.01) test method for Vickers Hardness of materials. The sample was placed on a flat surface, and a load of 0.3 kgf was applied, with maximum and minimum limits of 100 HV and 010 HV, respectively.

The readings were digitally collected from three (3) different points on the sample with 1/16″Steel ball indenter before manual mapping to obtain the hardness. Rectangular-shaped samples with dimensions of 4 mm in thickness, 30 mm in width, and 30 mm in length were used. The test was repeated about three times and the average values were obtained.

3. Water Absorption test

The water absorption test was carried out according to ASTM 2842 standard procedure. 2mm x 20mm x 3 mm samples were completely immersed in a 100 ml beaker. The composite samples were left in the water for 24-hour intervals, after which the samples were removed, cleaned with a clean cloth and reweighed.

The same procedure was repeated until the samples attained saturation, reweighing after 48 hrs and the percentage water absorption was calculated using Equation 2.

$$\text{Water absorption (\%)} = \frac{\text{final weight} - \text{initial weight}}{\text{initial weight}} \times 100 \ \ldots\ldots\ldots\ldots \ 2$$

Mechanical Properties Characterisation

1. Tensile Test

The tensile properties were carried out using the Tensile Strength Test Machine (Model: EM2101-T7, China) following ASTM D638. A 10-kN load cell and cross-head speed of 5 mm.min^{-1} were used to determine the ultimate tensile strength, elastic modulus, and elongation at the break of bambara nut shell and cowpea husk composites. The uniaxial load was applied to each end of the respective samples until failure using sample dimensions of 4 mm thickness, 10 mm width and 40 mm gauge length. The test was repeated with five specimens for each sample and the average value was recorded along with its 95% confidence interval. Before the mechanical tests, the specimens were conditioned at 65 °C for 1 h to eliminate the internal stress caused by the process and possible moisture.

2. Impact Test

Charpy Impact test was performed on the Bambara nut shell and cowpea husk reinforced composites according to ASTM D256 on a pendulum impact tester (Resil Impactor Junior Series, manufactured by Ceast Torino, Italy), with a speed range of 2.9 – 3.7 m/s. A sample with dimensions of 100 mm x 13 mm and 4 mm thickness, having a 0.5 mm v-notch, was used with a 2.75 J impact hammer (expandable to 25 J). The reported absorbed energy and impact strength values represent the average of five different specimens along with a 95% confidence interval.

3. Flexural test

Three-point bending test was performed to measure flexural properties on the Bambara nut shell and cowpea husk composites using a Universal/Electronic flexural test machine (model number HD B6I5A-S, China) to determine the flexural modulus and strength according to ASTM D-790 (ISO 4049.13) standard procedure with a cross-head speed of 2 mm/min. Five specimens were prepared for each of the samples. The specimens were cut into rectangular sizes with dimensions of 4 mm thickness, 30 mm width and 40 mm gauge length and the deformation in mm and load in kN were recorded.

4. SEM fractography

To distinguish the different damage mechanisms, the specimen fractured surfaces for the Bambara nut shell and cowpea husk composites were examined using SEM, model JOEL-JSM 7600F operated at 20 kV and magnifications of 7000x and 9000x using ASTM E986-04(2017). About 13 mg of the samples were fitted in the specimen chamber and mounted rigidly on a specimen holder. Before observation, the samples were coated with electrically conducting material and deposited on the sample under high vacuum conditions using a gold-palladium alloy target for sputtering in an argon atmosphere.

Results and Discussions

Filler Processing and Composite Fabrication

This work has resulted in the successful fabrication of cowpea husk and bambara nut shell composites as fillers in unsaturated polyester resin. The developed sheets are shown below before they were cut into different shapes according to ASTM standards using a motorised Dumbbell cutter.

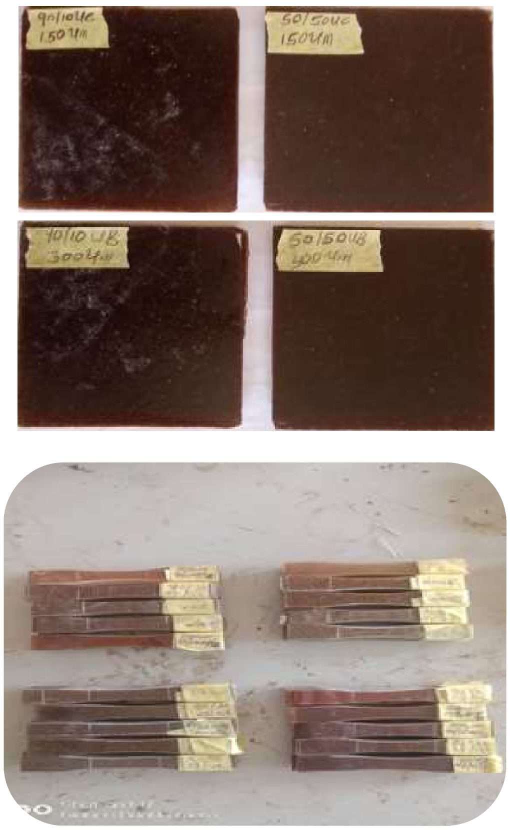

Plate 1: Fabricated Composite Sheets and Cut Tensile Samples for Analyses

Effect of Filler Loadings and Treatment on the Physical Properties of the Composite

The density of Bambara Nut Shell and Cowpea Husk in Polyester Resin Matrix

The densities of treated/untreated bambara nut shell and cowpea husk of unsaturated polyester composites are shown in Figures 2 and 3. The densities of the filled composite decrease with an increase in filler. Treated cowpea husk/unsaturated polyester composite at 10 wt% and 150 µm had a value of 1.12 g/cm^3 (Figure 2), which decreases to 1.08 g/cm^3 at 50 wt% while treated bambara nut shell composite as the same leading and filler size had values of 1.2 g/cm^3 and 1.16 g/cm^3 respectively. The importance of this analysis is that the introduction of fillers into the polymers in various applications has shown the improvement of stiffness to density ratio and has even further boosted their advancement (Evens *et al.,* 2019). Depending on the specific application, an increase in density may be undesirable, especially if lightweight characteristics are a priority.

The hardness of Bambara nut shell and Cowpea husk in Polyester resin matrix

The hardness of bambara nut shell and Cowpea husk composites in polyester resin polymer is shown in Figure 4. The composites resistance to indentation is also seen to improve with increasing filler loading and particle size as shown in Figure 4-5. The 50 wt% untreated bambara nut shell fillers had the strongest resistance to penetration, with hardness values of 72.2 HV compared to the cowpea husk fillers with values of 70.2 HV. The unfilled polyester resin had a hardness value of 28.5 HV which had a minimum of 24% increment after the introduction of fillers at 10 wt% for the treated bambara nut shell (40.6 HV) and treated cowpea husk (37.3 HV).

These values progressively increased with every addition of either bambara nut shell and cowpea filler. The type and size of fillers used in resin composites have a significant impact on their hardness. Larger fillers tend to increase the hardness of the material due to their greater resistance to deformation, while smaller fillers provide a smoother surface and may improve the polishability of the material if needed.

Considering the resin type, polyester resin presented higher hardness values when used as the matrix system for agricultural wastes such as bambara nut shell and cowpea husk. Even while the hardness of the various fillers and their contents varies, this is still not the only indicator for product design and cannot be used as a benchmark to determine which performs best for product development (Sagir, 2019).

Mechanical characterisation

Tensile Properties of Bambara nut Shell and Cowpea husk in Polyester Resin Matrix

The effect of polyester/bambara nut shell and polyester/cowpea husk fillers on tensile properties are well presented in Figures 6-7. These analyses were necessary to compare the effect of resin on the performance properties of these agricultural fillers. The tensile strength (Figure 6) decreased linearly with increasing filler loading; this shows that the fillers are non-reinforcing fillers (Kolawole, 2016). The tensile strength of the untreated cowpea husk/unsaturated polyester composite for maximum and minimum values with 28.06 Mpa and 18.13Mpa respectively, while that of the treated cowpea husk/unsaturated polyester composite is 34.57Mpa and 22.66Mpa respectively. The decrease in tensile strength with an increase in filler loading was not surprising since other studies such as Raju *et al.* (2012) had also indicated that the incorporation of filler into the matrix might not necessarily increase the tensile strength of the composites. The 10 wt% of the filler loadings recorded the best composites for the different filler types.

The difference between the unfilled polyester resin and the best composites (34.57 MPa) was 2% for the treated cowpea at 150 µm (Figure 6). Gital and Raji (2022) also reported that the ultimate tensile strength (UTS) of their polyester composites decreased from 20.90 MPa to 13.76 MPa when the percentage weight of the Bambara nut shell particles (BSP) increased within the matrix of the composites. The polyester composite with the lowest weight fraction of the filler (10 wt%), had the highest strength. Bambara nut husk fillers like other lignocellulosic fillers are non-reinforcing fillers hence the mechanical properties decrease in proportion to the filler content.

The larger filler sizes (Figure 7) have been seen to result in inadequate bonding or adhesion between the filler particles and the polyester matrix material. This led to poor interfacial bonding that weakened the overall composite, limiting the transfer of load between the matrix and the filler. The tensile strength was 34.57 MPa at 150 µm (Figure 6) and was reduced to 30.28 MPa at 300 µm (Figure 7) for the optimum loading of 10 wt% for treated cowpea.

The smaller the particle size, the larger the surface area, hence the interfacial interaction is better. Hence, it is recommended that a lower particle size should be used so that uniform dispersion of the small particles within the matrix will be easy to achieve, leading to even mechanical properties and performance recorded in this study. At higher loadings, the surface wetting of the filler by the matrix is reduced and filler-filler interaction increases. This leads to a decrease in mechanical properties. In summary, while fillers play a crucial role in enhancing certain properties of composite materials, using larger filler sizes can have drawbacks, particularly with respect to tensile strength. Achieving a balance between filler content, size, and distribution is essential for optimising the overall performance of composite materials in various applications.

Elongation at Break

The elongation at break for the different filler sizes is shown in Figures 8 and 9. The addition of higher filler sizes reduced the elongation of the polyester resin, which was progressive for the different weight percentages of the fillers (10 to 50 wt%). At 10 wt%, the elongation at break for treated bambara nut shell was 2.45% (Figure 8) for 150 µm, which reduced to 2.43% for 300 µm (Figure 9).

This pattern is also seen to repeat for other filler loading percentages. The fillers are seen to restrict the movement of the polymer chains within the matrix, leading to decreased flexibility and elongation at break. The results also show that lower particle sizes (Figure 8) improved the elongation at break better than larger particle sizes (Figure 9).

At 10 wt% and 150 µm, the composite with untreated bambara nut shell had the elongation at break increased from 2.37% to 2.45% after treatment, while that of cowpea husk increased from 2.60% to 2.63% after treatment. Emphasis here is placed on the 10 wt% for the ease of comparison as it presented the composites with optimum properties.

The treatment of the fillers with an alkali solution is seen to improve the elongation at break for all the composites, irrespective of the filler. Cowpea husk composites performed better than the Bambara nut shell composites in all cases. Larger filler particles can be said to have acted as stress concentrators, causing localised areas of high stress within the composite. This concentration of stress-initiated crack formation and propagation reduces the material's ability to elongate before failure. It may also result in inadequate bonding or adhesion between the filler particles and the matrix and poor interfacial bonding can lead to weak points in the material, negatively impacting its elongation at break.

Inhomogeneous distribution may also result in areas with different mechanical properties, affecting the overall elongation at the break of the composite. From the results it shows that the composites are brittle materials

Tensile Modulus

The tensile modulus of the cowpea husk and bambara nut shell polyester composites are shown in Figures 10 and 11, for the 150 and 300 µm, respectively. It is a measure of the samples' stiffness (rigidity) for the different fillers. With respect to treatment, there were reductions in the composites' modulus properties when they were filled with treated fillers.

As seen in Figure 10 (for 10 wt% loadings), there was a drop from 1.39 GPa to 1.38 GPa for untreated and treated bambara nut shell composites, respectively. This became pronounced for the cowpea husk composites, which dropped from 1.47 GPa to 1.37 GPa. When filler loadings were considered, it can be seen that there was a progressive increase in the modulus of elasticity as the filler loadings were increased from 10 to 50 wt% for both the 150 and 300 µm. Concerning filler size, composites filled with 300 µm particle sizes had a higher tensile modulus (Figure 11) compared to the tensile modulus of 150 µm (Figure 10).

The study has shown that the type of filler used can influence the modulus. As the filler content increases, the modulus of the composites increases for higher-modulus fillers, even for non-reinforcing fillers. It has been observed by many researchers that the modulus of lignocellulosic fillers increases with an increase in filler loading. (Raju *et al.,* 2012)

Impact Properties of Bambara nut Shell and Cowpea husk in Polyester Resin Matrix

The impact strength of the bamboo nut shell and cowpea husk in polyester resin is shown in Figure 4.12. Cowpea composites were better in performance than the Bambara nut shell composites with a value of 0.48 kJ/m^2 for the treated filler (Figure 12) and 0.43 kJ/m^2 for the untreated filler (Figure 12) at 10 wt% loading for 150 µm. These values were further reduced at a filler particle size of 300 µm (Figure 13) for the different loadings that are higher than 10 wt%. (Kolawole, 2016).

Smaller particles are seen to give a more homogeneous distribution within the composite material. This uniform distribution helped minimise the formation of voids or gaps between particles, which acted as stress concentration points during impact loading. Reduced void formation improves the overall structural integrity of the composite and enhances its resistance to impact-induced damage. Also, the presence of smaller particles can promote toughening mechanisms within the resin matrix as they act as crack deflection sites, hindering crack propagation and dissipating energy during impact events. This increased flexibility can help the composite absorb impact energy more effectively, reducing the likelihood of brittle failure and enhancing its impact resistance.

Flexural Properties of Bambara nut shell and Cowpea husk in Polyester Resin Matrix

The flexural properties of bambara nut shell and cowpea husk in polyester resin are shown in Figures 14 – 15. The treated fillers exhibit improved flexural strengths compared to their untreated counterparts. Untreated cowpea husk composites have a flexural strength of 34.22 MPa, while untreated bambara nut shell composites demonstrate even lower strength at 28.56 MPa.

Alkali treatment yields marginal improvements in flexural strength for both fillers, with alkali-treated cowpea husk composites showing the highest strength at 35.56 MPa. Overall, these results underscore the superior performance of cowpea husk over bambara nut shell as a filler in the composites, especially when considering mechanical properties like flexural strength.

Flexural Modulus

For the flexural modulus of polyester composites shown in Figures 16 and 17 for the 150 and 300 µm, respectively, there are also reductions in the polyester composites' stiffness.

The increase in flexural modulus of the composites when the filler loadings were increased from 10 to 50 wt% can be attributed to several factors. First, with higher filler loadings, there is a greater volume fraction of the filler material within the composite, leading to a stiffer and more rigid structure. This increased filler content enhances the composite's resistance to bending and deformation under applied load, resulting in a higher flexural modulus. Second, the higher density of filler particles at increased loadings can lead to more efficient load transfer between the filler and the matrix, improving overall stiffness and modulus.

Additionally, at higher filler loadings, there is a decrease in the amount of matrix material present, which may lead to a reduction in matrix-dominated deformation and contribute to the observed increase in flexural modulus. However, it's essential to note that beyond a certain filler loading threshold, excessive filler content can lead to agglomeration, increased porosity, and a reduction in mechanical properties (Rueda *et al*., 2017). Therefore, while increasing filler loadings can enhance the flexural modulus, there is a balance to be maintained to avoid detrimental effects on other mechanical properties of the composite.

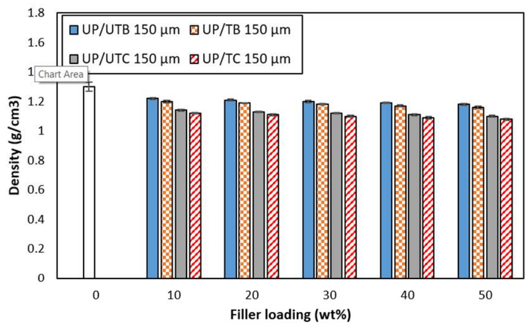

Figure 2: Effect of Filler Loading and Treatment on the Density of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 150 µm

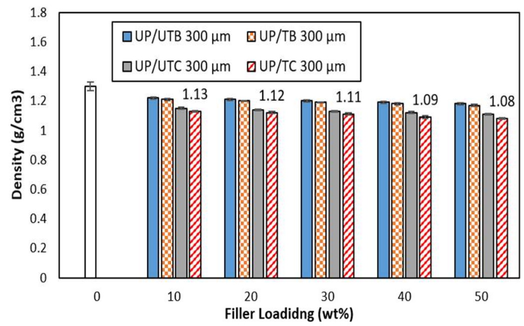

Figure 3: Effect of Filler Loading and Treatment on the Density of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 300 µm

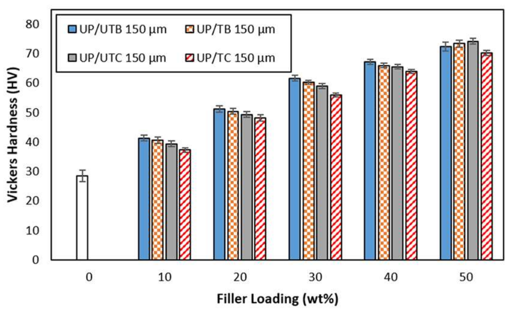

Figure 4: Effect of Filler Loading and treatment on the Hardness of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 150 µm

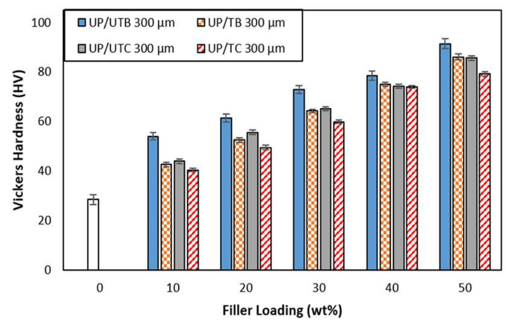

Figure 5: Effect of Filler Loading and Treatment on the Hardness of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 300 µm

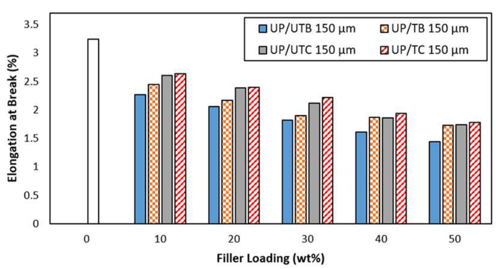

Figure 8: Effect of Filler Loading and Treatment on the Elongation at Break of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 150 µm

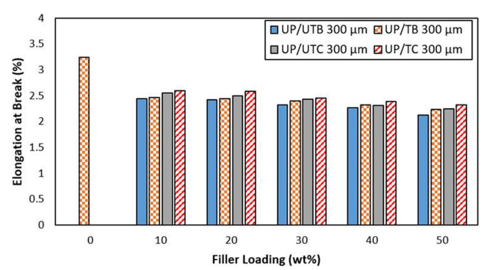

Figure 9: Effect of Filler Loading and Treatment on the Elongation at Break of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 300 µm

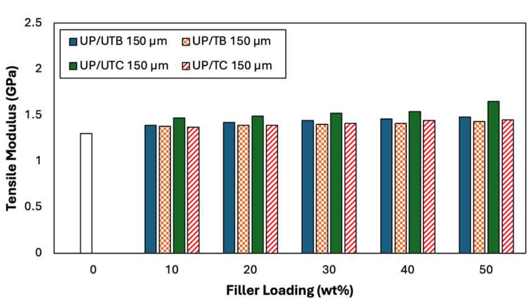

Figure 10: Effect of Filler Loading and Treatment on the Tensile Modulus of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 150 µm

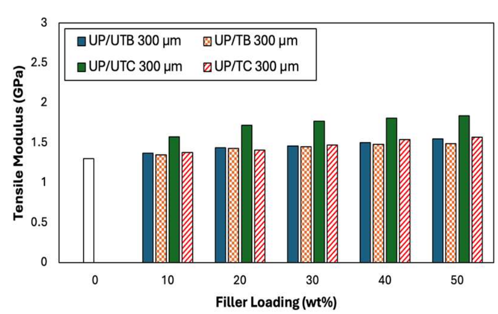

Figure 11: Effect of Filler Loading and Treatment on the Tensile Modulus of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 300 µm

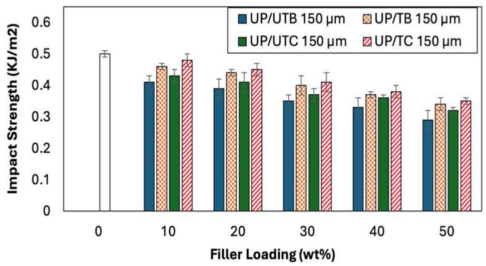

Figure 12: Effect of Filler Loading and Treatment on the Impact Strength of Polyester/Cowpea Husk and Polyester /Bambara nut shell Composites at 150 µm

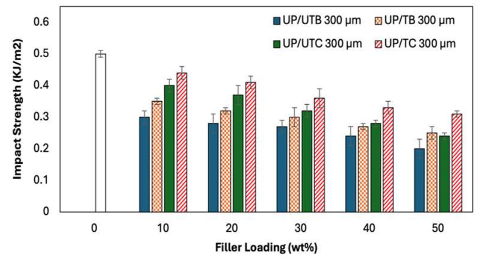

Figure 13: Effect of Filler Loading and Treatment on the Impact Strength of Polyester/Cowpea Husk and Polyester /Bambara nut shell Composites at 300 µm

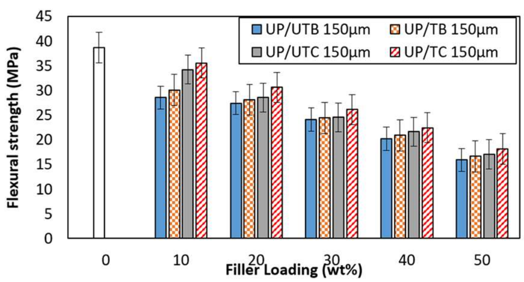

Figure 14: Effect of Filler Loading and treatment on the Flexural Strength of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 150 µm

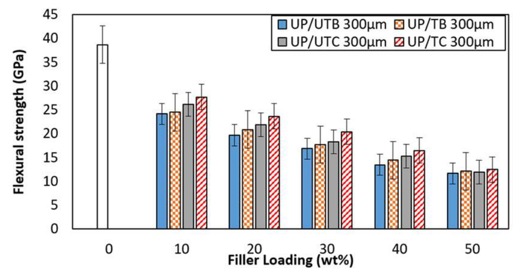

Figure 15: Effect of Filler Loading and Treatment on the Flexural Strength of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 300 µm

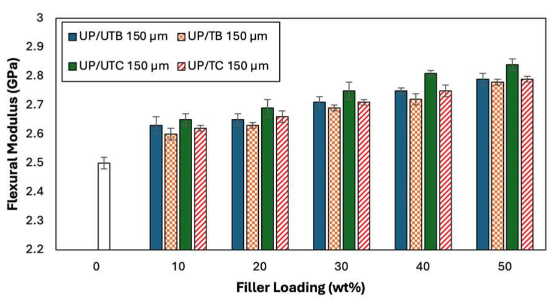

Figure 16: Effect of Filler Loading and Treatment on the Flexural Modulus of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 150 µm

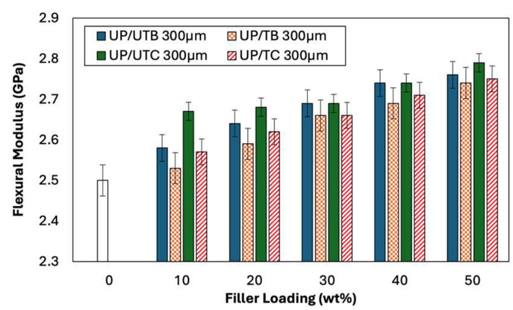

Figure 17: Effect of Filler Loading and Treatment on the Flexural Modulus of Polyester/Cowpea Husk and Polyester/Bambara nut shell Composites at 300 µm

Scanning Electron Microscopy

Effect of Filler Loading and Treatment

The scanning electron micrographs of the tensile fractured surfaces are shown in Figures 18-20 at magnifications of 7,000x and 8,000x, respectively. The different filler loadings are seen to affect the fracture behaviour of the composites. At lower filler loadings, such as 10 wt% and 150 µm (Figures 18a and b), the composites exhibited more ductile fracture behaviour, characterised by plastic deformation and energy absorption before failure. This is because the filler particles are dispersed within the matrix, allowing for some matrix deformation to occur before failure.

However, as filler loading increased (Figures 18c and d), the composite became more filled with filler particles, which acted as stress concentrators. This led to a transition from ductile to more brittle fracture behaviour, with less plastic deformation before failure. Additionally, at higher filler loadings, the increased presence of filler particles has also led to poor interfacial bonding with the matrix, promoting crack initiation and propagation along the filler-matrix interfaces.

On treatment, impurities, waxes, and other contaminants were removed from the Bambara nut shell filler surfaces, exposing more reactive sites and improving filler-matrix adhesion (Figure 18b), with decreased cracks and voids. This enhanced bonding at the interface led to better load transfer between the filler and matrix, resulting in improved mechanical properties and fracture toughness, as earlier recorded in previous results. Additionally, alkali treatment modified the surface chemistry of the fillers, making them more hydrophilic and promoting better wetting and dispersion within the matrix. As a result, composites containing alkali-treated fillers exhibited enhanced resistance to crack initiation and propagation, as well as improved energy dissipation mechanisms during fracture.

From Figure 18b, it is observed that the fracture surface of lower filler loadings had less obvious crack propagations running through the entire surface, which indicates that the material could withstand reasonable tensile stress. Hence, the resistance to crack propagation is more leading to fewer voids because of the homogeneity of the resin, as also previously reported (Visakh *et al*., 2012).

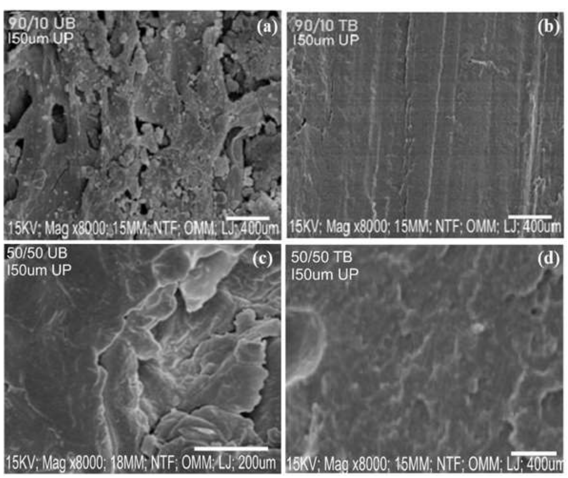

Figure 18: SEM Images for the Fractured Surfaces of (a) Untreated at 10 wt% (b) Treated at 10 wt% and (c) Untreated at 50 wt% (d) Treated at 50 wt% Bambara nut Shell Composites of Polyester Resin at 150 µm

Effect of Filler Particle Size

It is also seen that the formation of mechanical bonding at the surface is mainly dependent on the surface topology of the bamboo nut shell fillers in the matrix. The surface features of the fractured composites, such as contours, cracks, and voids, are observed in the micrographs containing fillers with higher particle sizes (Figure 19a and b). Larger particle sizes may exacerbate stress concentrations, hinder matrix flow, and decrease composite ductility, resulting in reduced fracture toughness and increased susceptibility to brittle fracture (Anosike-Francis *et al*., 2022b).

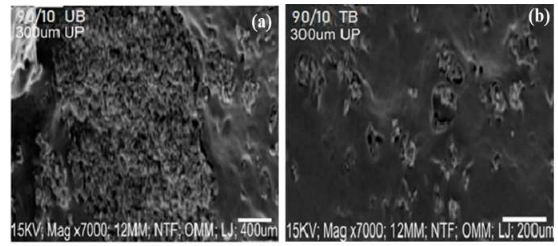

Figure 19: SEM Images for the Fractured Surfaces of (a) Untreated at 10 wt% (b) Treated at 10 wt% and Bambara nut Shell Composites of Polyester Resin at 300 µm

The fractured surfaces of the treated and untreated cowpea husk composites in polyester resin are shown in Figure 20. It can be seen that despite the low loading of the cowpea husks (10 wt%), the higher filler sizes of the cowpea husk can be visibly seen protruding out and showing their inability to form a homogenous system. Therefore, careful selection of filler particle size and loading is essential in optimising the mechanical properties and fracture behaviour of these composites for specific applications.

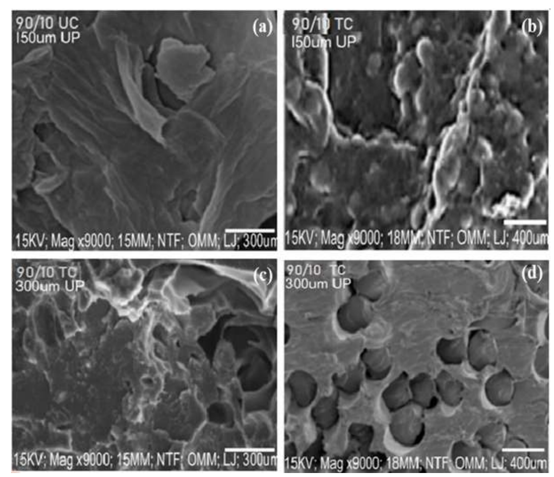

Figure 20: SEM Images for the Fractured Surfaces of (a) Untreated at 150 µm (b) Treated at 150 µm and (c) Untreated at 300 µm (d) Treated at 300 µm Cowpea Husk Composites of Polyester Resin

Conclusions

This work successfully studied the effect of surface treatment on the physical, mechanical, and morphological properties of bambara nut and cowpea husk composites of polyester polymer. It was observed that composites with moderate density were obtained at 10 wt% bambara nut shell and cowpea husk filler loadings for the polyester resins, which improved when the fillers were treated with a 5% alkaline solution. The Bambara nut shell-filled composites had a higher hardness value, while cowpea-husk-filled composites had better tensile properties, flexural properties, a higher crystallinity index, lower density and peak intensities. The developed composites have properties that make them suitable for non-load-bearing applications. Thus, they will have a performance advantage in packaging applications and also suitability for indoor, non-load-bearing partition board applications.

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