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Special Issue
A Themed Issue in Honour of Professor Onukwuli Okechukwu Dominic (FAS).

This special issue is dedicated to Professor Onukwuli Okechukwu Dominic (FAS), marking his retirement and celebrating a remarkable career. His legacy of exemplary scholarship, mentorship, and commitment to advancing knowledge is commemorated in this collection of works.

Edited by
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Nzelu Chukwudubem, Taiwo Oluwaseyi, Nwoye Chukwuka, Ojukwu Pauline, Nwambu Chidume

Department of Metallurgical and Materials Engineering, Nnamdi Azikwe University Awka.
*Corresponding Author's E-mail: oo.taiwo@unizik.edu.ng

Abstract
The integration of agricultural waste into polymer matrices offers sustainable pathways for developing eco-friendly composites. This study focuses on the mechanical behaviour of epoxy composites reinforced with corn cob ash (CCA) for sustainable applications. Using the hand lay-up method, the composites were fabricated with varying weight percentages of CCA (0%, 3%, 5%, and 10%). Microstructural, mechanical and physical properties were analysed, including tensile strength, hardness, impact strength, flexural strength, water absorption, and density. Results show that adding CCA influences the tensile and flexural properties with increase in the hardness and impact strength of epoxy. Presence of CCA in epoxy also reduced it water absorption property while increase in density was reported. The study shows the potential of CCA-reinforced epoxy composites for lightweight, sustainable applications thereby contributing to efficient waste utilization in engineering materials.

Keywords: Epoxy composites, corn cob ash, mechanical properties, sustainability, agricultural waste.

1. Introduction

The increasing global focus on sustainability has intensified efforts to integrate agricultural by-products into composite materials, leading to the production of eco-friendly and cost-effective alternatives to synthetic reinforcements. Among these by-products, corn cob ash (CCA), derived from the calcination of corn cobs, offers significant promise due to its lightweight, abundant availability, and high silica content (Abdullah et al., 2022; Zhang et al., 2021). According to Choi et al., (2022), global corn production is more than a billion tonnes, an increase of over 40% in the last decade. The corn cob itself is about 15 - 18% by weight fraction of an ear of corn highlighting its contribution to wastes globally when not utilized or disposed off properly (Enrica-Santolini et al., 2024; Shinners et al., 2012). One of the emerging ways to add value to corn is by utilizing it for production of composite especially polymer composites. Polymer composites, more than other types of composites are increasingly produced using agricultural by-products owing to their unique attributes such as low cost, flexibility, easy production, and light-weight that enable them to be swapped with traditional engineering materials (Swami et al., 2022). Polymer composites like the ones reinforced with non-polymeric fillers with epoxy are one of the most widely used matrices. Epoxy, a thermosetting polymer, is known for its excellent adhesion, chemical resistance, and mechanical strength (Fouly, 2021). However, its brittleness and environmental concerns necessitate reinforcement with materials such as agricultural ash to improve its mechanical and physical properties while addressing sustainability challenges (Ibrahim et al., 2023). This study investigates the mechanical behaviour of epoxy composites reinforced with varying weight percentages of CCA to evaluate their suitability for sustainable engineering applications.

Several studies have looked into enhancing the properties of epoxy by adding ash additives as a reinforcement and investigating the different characteristics of epoxy composites, especially its mechanical properties (Mostovoy et al., 2020). Sim et al., (2020) studied the effect of reinforcing epoxy by fly ash on the mechanical properties of epoxy composites. The study shows an initial improvement of tensile strength before a later decline, the compression

strength was consistently improved with increasing weight fraction. Mulenga et al., (2023) investigated the mechanical behaviour of sisal fiber/bio-epoxy/fly-ash reinforced hybrid composites. The tensile strength shows a notable enhancement of up to 6.3%, flexural strength experiences an impressive increase of 68%, impact strength demonstrates a remarkable boost of 28%, and scratch hardness elevates by 17% which were all achieved at 20% fibre weight fraction and 5% fly ash weight fraction. Vivek and Kanthavel (2018) investigated the mechanical properties of bagasse ash-filled epoxy composites reinforced with hybrid plant. The bagasse ash was milled to 350 nm and incorporated with weight fractions of 1, 3 and 5 wt% in the composites. There was significant improvement in the tensile, flexural, and impact strength for the composite filled up to 3wt.%.

Khalil et al., (2013) studied the development and characterization of epoxy reinforced with oil palm ash fibres. The study reported that nano-structured oil palm ash increases the flexural and tensile strength of composite up to 3wt.% with further weight fraction resulting in reduced tensile strength. A study by Baheti et al., (2015) studied the mechanical properties of glass fabric/epoxy composites filled with fly ash. The study reported that the presence of fly ash in glass fibre/epoxy composites caused a reduction in the compressive strength of the epoxy composite. In a bid to determine the maximum processing parameters that give optimum property combination, Sokolova et al (2024) studied the temperature regime, optimum content and particle size of filler epoxy reinforced with rice husk ash. The influence of rice husk ash on the hardness of the composite was also established. The result shows that the best compatibility with polymer epoxy matrix is possessed by rice husk ash obtained at the combustion temperature of 500oC introduced in the amount of 10wt.% rice husk ash – epoxy composite. Mestry et al., (2022) studied the wear and frictional behaviour of epoxy-based composites reinforced with corn cob ash of 2, 6 and 10%. The presence of corn cob ash was reported to influence the wear and frictional properties of the epoxy matrix. Swami and Raka, (2024) investigated the wear and frictional properties of graphite-reinforced corn cob ash-epoxy composites. The hybrid presence of corn cob ash and graphite improved on the wear and frictional behaviour of epoxy.

A review of the literature shows that waste ash products such as fly ash and other calcinated agricultural waste being used as reinforcement have influenced the mechanical properties of epoxy. While few studies found to have used corn cob ash as reinforcement for epoxy have focused on its wear application due to the high silica content of the corn cob ash. Consequently, this study aims to concentrate on the mechanical behaviour of the epoxy composites reinforced with corn cob ash. The tensile strength, hardness, impact strength, flexural strength, and young modulus will be investigated. Physical properties such as the water absorption and density while the microstructure of the epoxy composites will be evaluated.

2.0 Materials and methods

2.1 Materials

Epoxy resin (bisphenol-A) was used as the matrix material with a hardener (triethylenetetramine) for curing. Corn cob ash (CCA) was obtained through calcination of corn cobs at 600°C for 4 hours and sieved to particle sizes below 100 µm.

2.2 Composite Fabrication

The composite was prepared by a lay hand method. Prior to the resin mix, the control (i.e the epoxy) was poured only into the mould and allowed to dry, this is to serve as a base for composites performance evaluation. The predetermined weights percentage of corncob ash and epoxy resin are weighed using micro balance and mixed manually. Hardener was then weighed, with Epoxy to hardener ratio of (2:1) poured slowly in mixture and stirred uniformly and gently for 10 min to avoid the formation of bubbles Due to stirring, curing reaction starts, which can be experienced by exothermic reaction. The prepared mixture was gently poured inside the open wooden mould. The mould was covered with paper tape to aid easy removal of the composites after curing. The specimens were allowed to cure for 24 hours in the mould at room temperature before post-curing at 80°C for 2 hours. Once the solidification was completed, the test samples were removed from the pattern. The samples were further cut and polished as per ASTM standard and kept in plastic bags. Plastic bags are to prevent moisture absorption by samples. The procedure was carried out for 3wt.%, 5wt.%, and 10wt.%CCA reinforced epoxy composite samples. The selected weight fraction was based on reviews of literatures.

2.3 Test

2.3.1 Microstructural Test

2.3.1.1. The Fourier Transform Infrared (FTIR).
The Fourier Transform Infrared (FTIR) spectrum provides information about the functional groups present in the epoxy resin. Absorbance spectra were performed using a Thermo Scientific Nicolet™ model IR200 spectrometer, in the region from 4000 to 400 cm-1, with a spectral resolution of 4 cm-1 and 32 scans. The pellets were made by blending each dried sample in an oven at 105 °C with KBr in a 3:1 (w/w) ratio.

2.3.1.2 Scanning Electron Microscopy (SEM).
Scanning electron microscopy examinations of produced samples were carried out using a scanning electron microscope (SEM) (with model number TESCAN VEGAN III.) The composite samples were prepared and placed in the SEM chamber with a vacuum pressure of 70Pa. The accelerating voltage is 20 kV for achieving the desired resolution. The working distance is 9.5mm to optimize focus and image quality while the magnification level targeted is 9000x.

2.3.2 Mechanical Property Tests

2.3.2.1 Tensile and Flexural Strength: The Instron-Series 3369 Universal testing Machine was used for tensile and flexural test as per ASTM standards. ASTM D3039 and ASTM D7264 standard procedure was used for determining the tensile and flexural strength properties respectively of the composites.

2.3.2.2 Hardness**:** Vickers hardness tester will be used to measure the hardness of composite based on the Vickers hardness test method. A load of 500g was applied to a pyramid-shaped industrial indenter and the size of the indentation on the surface of the material was measured three times.

2.3.2.3 Impact Strength: Evaluated using Charpy impact tests (ASTM D256). The Charpy impact test (Charpy V-notch test) will be used to measure the toughness of materials under impact load at different temperatures. The Charpy impact test (Charpy V-notch test) will be used to measure the toughness of materials under impact load at different temperatures

2.3.3 Physical Property Tests

2.3.3.1.Water Absorption: The water absorption of the composites was measured according to ASTM D570. Composite specimens were cut precisely according to the test method specifications. Then, the specimens were dried in an oven for 4 hours and 50°C and placed in a desiccator to cool. Immediately upon cooling, the dried specimens are weighed with a high degree of accuracy using a scale. The weighed samples were then immersed in treated water at 23°C for a predetermined duration, often 24 hours. After the composites remain underwater for the specified period, the specimens are removed and carefully caressed dry with a lint-free cloth to remove surface water. Then, it is measured again. The water absorption percentage was calculated based on the weight change of the specimens before and after immersion. The following formula was used to determine the percentages of water absorption.

$$\text{Water Absorption (\%)} = \left(\frac{w_2 - w_1}{w_1}\right) x\ 100 \qquad (1)$$

Where:
W1 = Initial Weight of the dry specimen
W2 = Weight of the specimen after immersion

2.3.3.2 Density: The density of the composite is evaluated as per ASTM D 792. The density of the composite is calculated using the formula:
ρc =
Where:
is the weight of the sample in air.
is the weight of the sample in liquid.
is the density of the immersion liquid (for distilled water at 23°C, =1 g/cm3).second level heading.

3.0 Result and Discussion

3.1 FTIR

The FTIR spectrum confirms the presence of characteristic epoxy functional groups such as hydroxyl (-OH), ether (C-O-C), aromatic C=C, and possibly unreacted epoxy rings (González et al., 2012). Also, peaks around 1600 cm^{-1} and 1250 cm^{-1} are strong evidence of aromatic and ether groups, confirming the base structure of bisphenol – an epoxy resin. If curing is complete, the intensity of the 910–830 cm^{-1} peak (epoxy group) will diminish, while hydroxyl peaks (3400–3500 cm^{-1}) will increase, indicating successful crosslinking. It is important to note that the decrease in the epoxy ring peak intensity is used to monitor the progress of the curing reaction. Also, peaks associated with carbonyl (1700 cm^{-1}) or hydroxyl groups may indicate the presence of curing agents or fillers. Peaks around 1700 cm^{-1} (C=O) and changes in hydroxyl content provide insights into the thermal or oxidative stability of the resin.

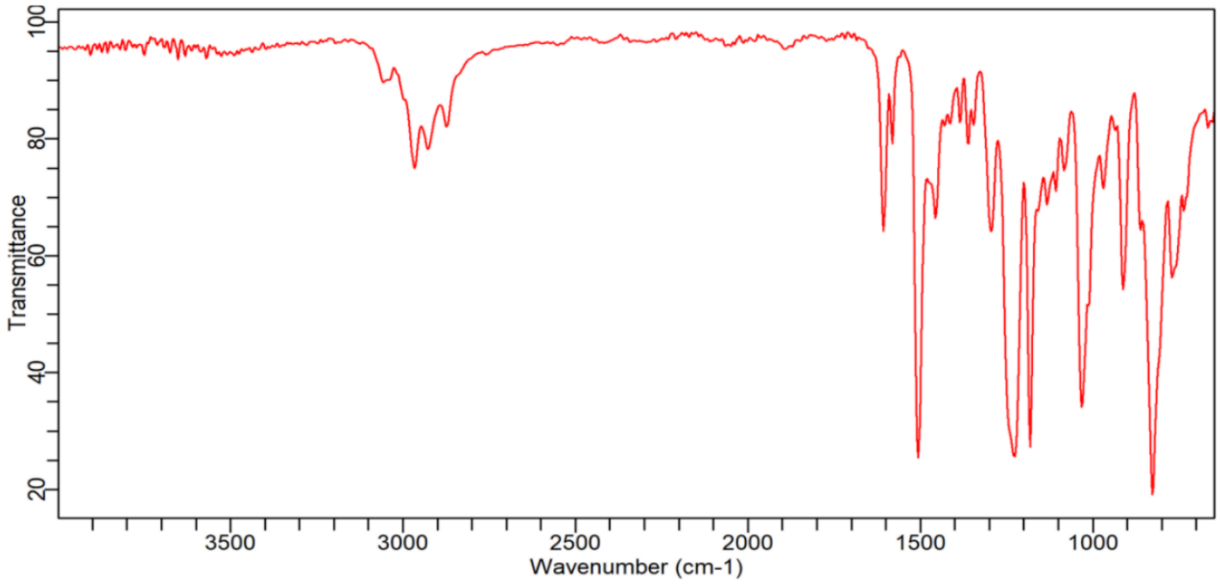


Figure 1: FTIR of Epoxy

Figure 2 shows the FTIR spectrum of corn cob ash. The broad peak around 3500–3200 cm^{-1} is generally attributed to hydroxyl groups' O–H stretching vibration, which may come from adsorbed water or hydroxyl groups in the ash. Also, the peaks around 2900–2800 cm^{-1} are typically associated with C–H stretching vibrations from aliphatic hydrocarbons. However, in ash, these peaks may not be prominent due to combustion removing organic content. The peaks around 1650–1600 cm^{-1} is associated with the C=C stretching of aromatic compounds or the bending vibration of water molecules.

Peaks near 1450–1400 cm^{-1} as observed are often due to symmetric and asymmetric bending vibrations of CH_2 or CH_3 groups, which may arise from residual organic material or lignin degradation products. Prominent peak around 1100–1000 cm^{-1} corresponds to the Si–O stretching vibrations, indicating the presence of silica, which is a significant component in corn cob ash. Other peaks in the 900–400 cm^{-1} region are associated with metal-oxygen bonds (e.g., Si–O, Al–O) or the crystalline forms of minerals like quartz or other silicates (Padilla et al., 2019).

Corn cob ash primarily contains silica (SiO_2) and other inorganic minerals formed during the combustion of biomass (Wardhani et al., 2017). In the FTIR spectrum, the O–H stretching and bending vibrations indicate moisture or hydroxyl groups in the ash. The intense peak around 1100 cm^{-1} confirms the presence of silica, either in amorphous

or crystalline form. Finally, peaks in the fingerprint region may correspond to metal oxides or carbonates, depending on the combustion conditions (Padilla et al., 2019).

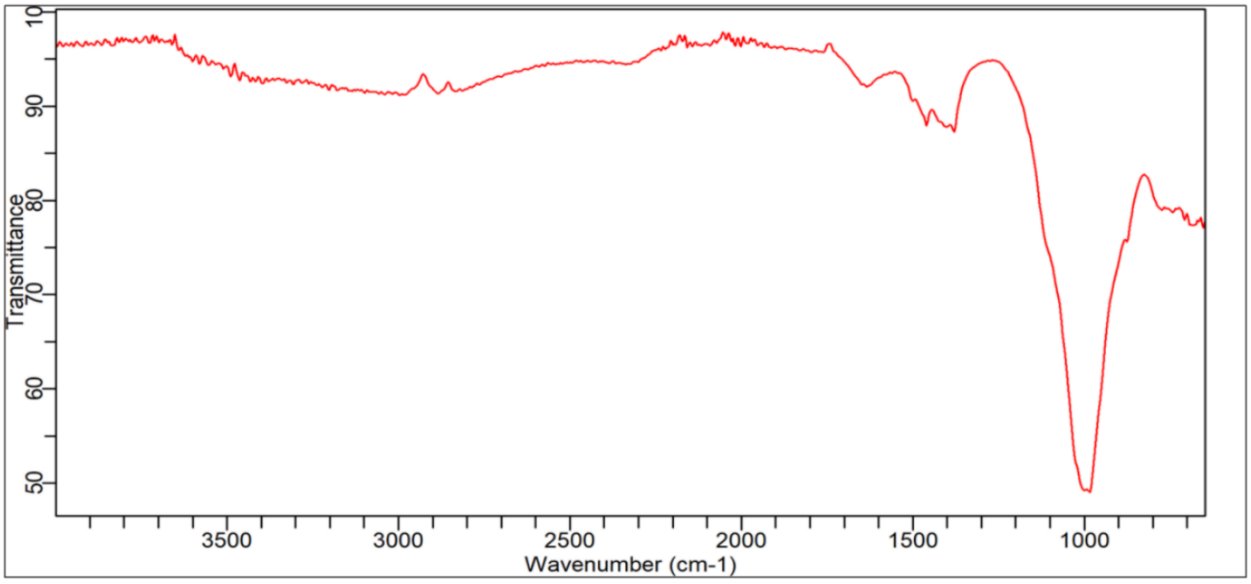


Figure 2: FTIR for Corn hub

3.2 Microstructure of Epoxy – CCA Composite

Figure 3 shows the SEM image of a 3 wt.% CCA reinforced epoxy composite provides critical insights into the microstructure, surface morphology, and particle-matrix interactions of the material. The SEM image shows a highly irregular, rough morphology, typical of composites with bio-based fillers. This structure suggests the presence of filler-matrix interfaces and agglomerated filler particles. The corn cob particles appear to be unevenly distributed within the epoxy matrix, showing some clustering or agglomeration. This clustering could affect mechanical properties, such as reducing tensile strength or impact resistance.The large void-like regions (dark areas) suggest poor interfacial bonding between the epoxy matrix and corn cob fillers (Kumar et al., 2020). There is also possible shrinkage during curing of the epoxy resin. The fillers appear to have fractured edges and porous structures, contributing to the roughness of the composite.

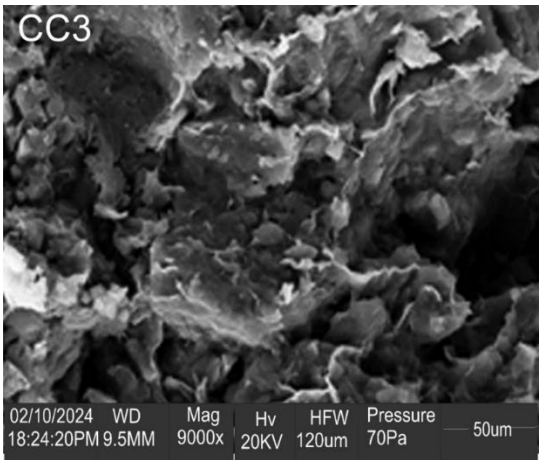


Figure 3: SEM Image of 3wt.% CCA reinforced Epoxy Composite

3.3 Tensile Strength of Epoxy – CCA Composite

The tensile strength of epoxy composites reinforced with CCA at weight fractions of 3 wt.%, 5 wt.%, and 10 wt.% is presented in Figure 4. The results reveal a consistent decline in tensile strength as the filler content increases, which aligns with findings from previous studies (Aigbodion et al., 2010; Singh et al., 2018). At 3 wt.% corn cob ash, the tensile strength of the composite is 19.4 MPa, indicating a slight decrease from pure epoxy (24.5 MPa). This suggests that a small amount of filler marginally reduces the tensile properties of the composite owing to the introduction of fillers within the matrix (Alhassan et al., 2021). At 5 wt.%, the tensile strength further declines to around 13.9 MPa, showing a more noticeable reduction. This can be attributed to the potential agglomeration of the filler particles or inadequate stress transfer between the filler and matrix (Olowu et al., 2020). At 10 wt.%, the tensile strength significantly drops to 9.4 MPa, indicating that excessive filler content deteriorates the composite's mechanical performance. This is consistent with findings that poor dispersion and interfacial bonding at higher filler concentrations weaken the material (Ighalo & Adeniyi, 2019). The downward trend in tensile strength with increasing filler content demonstrates an inverse relationship between tensile strength and the amount of corn cob ash reinforcement (Sharma & Pathak, 2020).

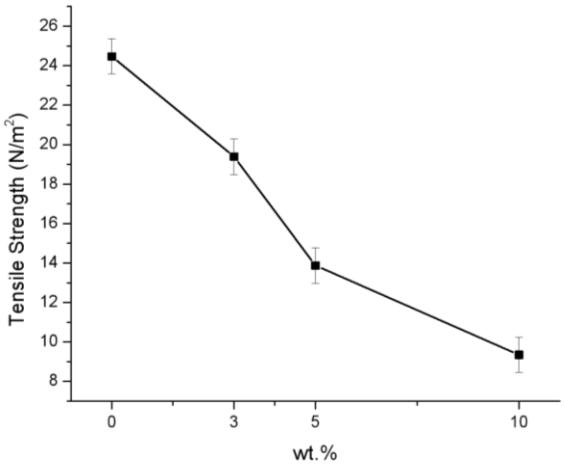


Figure 4: Tensile Strength of Epoxy reinforced Corn Cob Ash

3.4 Hardness of Epoxy - CCA Composites.

The hardness properties of epoxy composites reinforced with 3 wt.%, 5 wt.%, and 10 wt.% corn cob ash, as shown in Figure 5 below indicate a progressive increase in hardness with the addition of corn cob ash as a reinforcing filler. (Aigbodion et al., 2010; Alhassan et al., 2021). At 3 wt.% filler content, the hardness increases to 33.43, representing an approximate 21% improvement over pure epoxy. This enhancement can be attributed to the dispersion of the rigid corn cob ash particles, which impede localized plastic deformation and enhance surface hardness (Ighalo & Adeniyi, 2019). The hardness further rises to 34.80, showing that additional filler continues to improve the composite's ability to resist deformation. This result suggests that the filler particles are well-distributed at this content level, contributing to a more uniform reinforcement effect (Olowu et al., 2020). At 10 wt.%, the hardness reaches a maximum of 39.03, representing a 41.6% improvement over the baseline. The significant increase is due to the cumulative effect of more filler particles, which restrict the movement of the polymer matrix under applied force. However, excessively high filler content might lead to diminishing returns in some cases due to potential agglomeration, but this is not observed here (Singh et al., 2018). The data demonstrate a direct correlation between the filler content and the hardness of the composite. As the corn cob ash content increases, the composite's hardness improves. This is consistent with the theory that adding hard, rigid particles to a softer matrix enhances its surface resistance to indentation or localized deformation (Aigbodion et al., 2010; Alhassan et al., 2021).

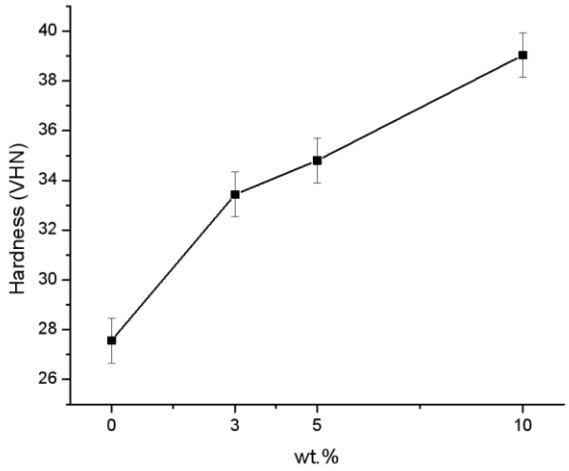


Figure 5: Hardness of Epoxy reinforced with Corn Cob Ash.

3.5 Impact Strength of Epoxy – CCA Composites.

The impact strength of epoxy composites reinforced with 3 wt.%, 5 wt.%, and 10 wt.% corn cob ash, as shown in Figure 6 below. The impact strength increases from 5.42 J to 6.33 J at 3 wt.% filler content, an improvement of 16.8%. This enhancement can be attributed to the dispersion of corn cob ash particles, which improve energy dissipation mechanisms during impact. The rigid filler particles help in transferring the stress, thereby enhancing the composite's ability to withstand impact (Ighalo & Adeniyi, 2019). At 5 wt.% filler content, the impact strength decreases slightly to 5.65 J. This decline suggests that the corn cob ash dispersion might be less uniform, leading to stress concentrations that reduce the composite's toughness. Studies indicate that improper filler dispersion can adversely affect the energy-absorbing capacity of composites (Olowu et al., 2020). The impact strength further drops to 4.77 J at 10 wt.% filler content, a reduction of 12% compared to pure epoxy. This significant decline is likely due to filler agglomeration, which creates weak points in the composite matrix. Such agglomerates act as stress concentrators, reducing the material's ability to absorb and dissipate energy under impact loading (Alhassan et al., 2021).

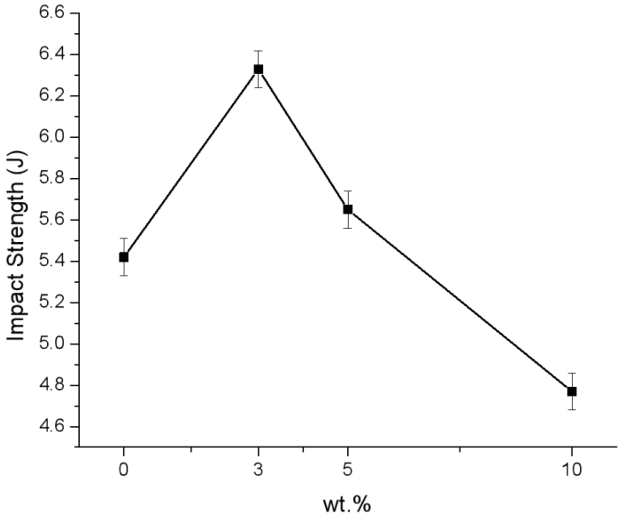


Figure 6: Impact Strength of Epoxy reinforced with Corn Cob Ash

3.6 Flexural Strength of Epoxy - CCA Composites.

Figure 7 shows the flexural strength of CCA reinforced epoxy composites. The baseline flexural strength of pure epoxy is 15.38 MPa. This high value reflects the inherent rigidity and strong bonding of the unreinforced polymer matrix. At 3 wt.% filler content, the flexural strength slightly decreases to 15.36 MPa, representing a marginal reduction. This slight decline may be due to the initial disruption of the epoxy matrix caused by the addition of fillers, which can interfere with the matrix's structural integrity (Ighalo & Adeniyi, 2019). A noticeable drop in flexural strength occurs at 5 wt.% filler content, where the value reduces to 10.81 MPa. This represents a significant decrease of approximately 29.7% compared to pure epoxy. At 10 wt.% filler content, the flexural strength slightly recovers to 12.26 MPa, indicating an improvement compared to 5 wt.% but still lower than the baseline value. The flexural strength follows a non-linear trend, decreasing significantly at 5 wt.% filler content and partially recovering at 10 wt.%. This behavior highlights the critical role of filler dispersion and interfacial bonding in determining the mechanical properties of epoxy composites. Similar trends have been reported in studies on natural fillers, where poor dispersion and weak bonding lead to a reduction in flexural properties (Sharma & Pathak, 2020). Previous studies have shown that the addition of natural fillers such as rice husk, palm kernel shell ash, and corn cob ash often results in an initial decline in flexural strength due to poor compatibility and filler agglomeration.

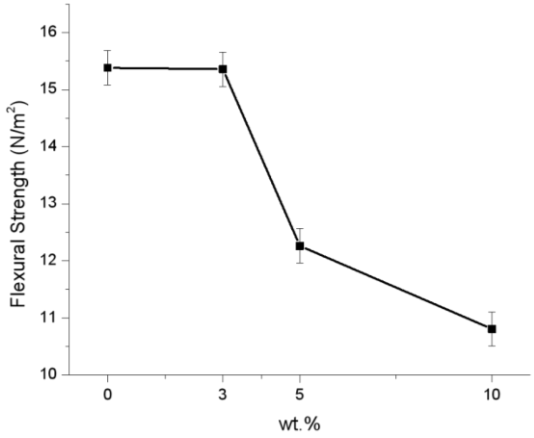


Figure 7: Flexural Strength of Epoxy reinforced with Corn Cob Ash

3.7 Water Absorption of Epoxy – CCA Composites.

The water absorption values of epoxy composites reinforced with 3 wt.%, 5 wt.%, and 10 wt.% corn cob ash, as presented in Figure 8 below, shows the effect of filler content on the composite's hydrophilic behaviour. Water absorption is a critical property that affects the durability and performance of composites, particularly in applications where moisture exposure is prevalent (Sharma & Pathak, 2020; Singh et al., 2018). The water absorption for pure epoxy is 0.9434%, reflecting the low moisture uptake typical of epoxy resins due to their hydrophobic nature and tightly crosslinked polymer structure. Epoxy matrices are known for their excellent resistance to water absorption, which contributes to their high durability (Aigbodion et al., 2010). The addition of 3 wt.% corn cob ash reduces water absorption to 0.88496%. This decrease may be attributed to the partial filling of voids and micro-cracks within the matrix by the ash particles, which can limit the pathways for water penetration (Olowu et al., 2020). A reduction in water absorption is observed at 5 wt.% filler content, with the value dropping to 0.75758%. This substantial decrease indicates improved filler dispersion and a possible barrier effect created by the ash particles, which inhibit water ingress. Studies suggest that at certain filler loadings, the distribution of fillers can effectively block moisture pathways, enhancing water resistance (Ighalo & Adeniyi, 2019). At 10 wt.% filler content, water absorption decreases significantly to 0.17153%. The water absorption behaviour demonstrates a linear trend. The reduction in water absorption at 10 wt.% filler content is the most notable, highlighting the optimal level of filler addition for enhancing water resistance. Similar studies on natural fillers like rice husk ash and palm kernel shell ash have reported non-linear water absorption trends. The improved water resistance at optimal filler content is often attributed to enhanced filler-matrix interaction, while higher filler loadings introduce defects that facilitate water uptake (Singh et al., 2018; Aigbodion et al., 2010). The water absorption results suggest that all

composites are suitable for applications requiring high moisture resistance, such as marine or outdoor environments (Olowu et al., 2020).

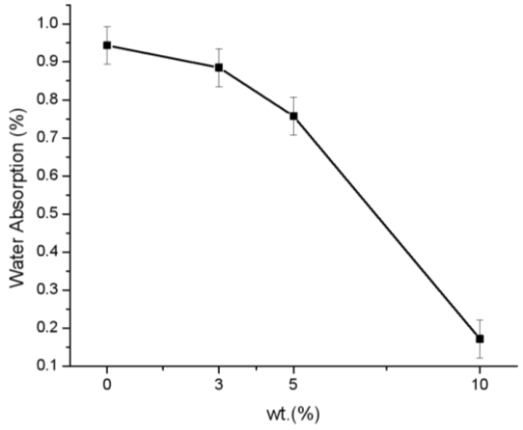


Figure 8: Water Absorption of Epoxy reinforced with Corn Cob Ash

3.8 Density of Epoxy - CCA Composites.

Figure 9 shows the density of epoxy composites reinforced with 3 wt.%, 5 wt.%, and 10 wt.% CCA, indicating the influence of CCA on the physical properties of epoxy. The density of pure epoxy is measured at 0.97 g/cm³. The density slightly increases to 0.99 g/cm³ with the addition of 3 wt.% corn cob ash. This minimal increase is attributed to the incorporation of the filler material, which likely has a higher density than the epoxy matrix (Ighalo & Adeniyi, 2019). At 5 wt.% filler content, the density rises to 1.01 g/cm³. This increase could be due to the higher filler content, as the density of the ash particles contributes more prominently to the composite's overall density. Effective bonding between the filler and the matrix could also reduce voids, contributing to the higher density (Sharma & Pathak, 2020). The density increases to 1.22 g/cm³ at 10 wt.% filler content.

Similar trends have been observed in studies involving other agricultural waste fillers, such as rice husk ash and palm kernel shell ash. However, these studies reported an initial increase in density with filler content due to the higher density of fillers, followed by a decrease at higher filler contents due to poor dispersion and void formation (Aigbodion et al., 2010; Ighalo and Adeniyi, 2019). The results suggest that the composites could be beneficial for applications requiring higher density and better mechanical performance, such as structural components.

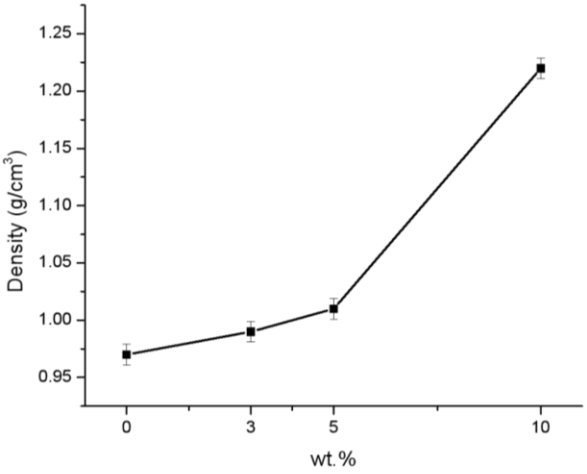


Figure 9: Density of Epoxy reinforced with Corn Cob Ash.

4.0. Conclusion

The present study advances research on the use of CCA as a reinforcement in epoxy composites by focusing on the mechanical and physical properties. This study demonstrates that epoxy composites reinforced with corn cob ash exhibit improved mechanical properties, particularly hardness and impact strength, at moderate reinforcement levels. The increased density and reduced water absorption further support their application in lightweight structural components. However, the decline in tensile and flexural strength highlight areas for optimization. The study proposes advanced technique treatment for CCA to enhance adhesion with epoxy matrix. It is also important to note that scaling up of the CCA reinforced epoxy composites is environmentally sustainable and economically viable due to its low cost of production. Furthermore, the findings in this study underscore the potential of CCA as a sustainable reinforcement for polymer composites, contributing to waste valorization and eco-friendly material development.

5.0 Recommendation

Further characterisation on the wear behaviour of CCA reinforced epoxy can be investigated.

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