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Innovative Use of Agricultural Byproducts: *Theobroma cacao* Shell Powder in Polypropylene Composites

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ABSTRACT

Some agricultural byproducts, considered waste, offer significant potential for sustainable composites. This study analyzed composites made from *Theobroma cacao* shell powder (CFS) and polypropylene (PP), and examined their structural, mechanical, and thermal properties. Characterization methods included water absorption, flammability, hardness, tensile strength, scanning electron microscopy (SEM), and X-ray diffraction (XRD) analyses. Results showed that water absorption increased with higher CFS content due to the hydrophilic nature of cocoa shell particles. Flammability tests indicated shorter ignition time and faster flame propagation with increased filler, which were linked to enhanced oxygen permeability in the composite's porous structure. Tensile strength and hardness decreased as CFS content rose, though treated composites exhibited improved performance over untreated ones. SEM revealed weak interfacial bonding and particle clustering, while XRD demonstrated greater crystallinity in treated composites, reflected in sharper peaks and better structural order. These findings highlight the potential of CFS/PP composites for domestic applications, emphasizing the importance of optimizing filler content and treatment methods. The study underscores the value of agricultural byproducts in advancing sustainable and practical composite materials.

Keywords: Cocoa shell powder, polypropylene composites, mechanical properties, X-ray diffraction

INTRODUCTION

Cocoa (*Theobroma cacao* L.) is an important global crop, producing around 5 million tons of dry cocoa beans each year [1]. The *Theobroma cacao* tree, a tropical perennial from the *Malvacea*e family, is grown for its edible seeds. Cocoa varieties fall into four main categories: Criollo, Trinitario, Forastero, and Nacional, each defined by their geographical origins and the characteristics of their beans. Among these, the Forastero variety is the most widely produced, making up about 70% of the global market [2]. With the increasing demand for construction materials and growing environmental concerns, eco-friendly materials are becoming more critical [3].

Recently, *Theobroma cacao,* or cocoa, has emerged as a promising filler for composite materials due to its availability, low density, and ability to reinforce structures. Cocoa bean shells, a byproduct of cocoa processing, have been investigated as natural fillers in polymer composites. For instance, bioactive poly(lactic acid)-cocoa bean shell composites have shown potential in biomaterial applications [4]. Likewise, modified cocoa pod husk fillers in polypropylene composites indicate a pathway toward sustainable material production [5]. Composite materials, which consist of a matrix and reinforcement, enhance properties by merging different phases.

Plate 1 shows the image of a dried cocoa shell. By incorporating cocoa shell powder with polypropylene, it is possible to create eco-friendly composites with improved mechanical properties and durability. These advancements offer sustainable alternatives to conventional materials like plywood, helping to address environmental issues and lessen reliance on limited resources [6, 7].

This study emphasizes the importance of utilizing agricultural byproducts such as cocoa shells for the production of sustainable composites, contributing to advancements in material science and promoting environmental sustainability.

Plate 1: *Theobroma cacao* Shell

This work is therefore aimed at the preparation and characterization of composite material produce from polypropylene waste and Cocoa Fruit-shells. The objectives of the research are as follows: to collect and pretreat cocoa seed shells; to produce and characterize composites from unused and waste polypropylene reinforced with the prepared *c*ocoa seed shells; to characterize the two composites produced; to determine the composite morphology using SEM; and to determine the solid-state structure of the composites produced using X-ray diffraction.

MATERIALS AND METHODS

Sample Collection and Preparation

The *Theobroma cacao* shells were obtained from Boki cocoa plantation, Boki Local Government Area of Cross River State, Nigeria. These shells underwent the process of grinding into powder. This procedure was carried out at the Chemistry laboratory of the Federal University Wukari in Taraba State, Nigeria. Polypropylene plastic, sourced from around the neighborhood and some bought from the Wukari market in Taraba State, Nigeria, was utilized for the study. Some of these polyethylene films were intentionally exposed to sunlight for a duration of four weeks to induce photo-degradation.

Alkaline Treatment

Alkaline treatment or mercerization is one of the best-used chemical treatments for natural fillers. Due to alkali treatment, there is an increase in the amount of amorphous cellulose at the expense of crystalline cellulose. By this treatment, there is a removal of hydrogen bonding in the network structure.

The reaction which takes place during this treatment is shown in Equation 1[8]

$$\text{Fibre-OH} + \text{NaOH} \longrightarrow \text{Filler-O}^{-}\text{Na}^{+} + \text{H}_2\text{O} \quad (1)$$

To undergo alkali treatment, *Theobroma cacao* shell fillers were immersed in a 5% NaOH solution at room temperature, maintaining a liquor ratio of 15:1. The fillers remained in the alkali solution for the duration of 4 hours. Subsequently, the fillers underwent thorough washing with fresh water to eliminate any NaOH adhering to the filler surface. Neutralization was achieved using dilute acetic acid, and a final wash with distilled water was performed to maintain a pH of 7. The treated fillers were then subjected to air drying at room temperature for 48 hours, followed by additional oven drying at 100 °C for 6 hours.

Preparation of Composite

Theobroma cacao shell was dried in an oven at 100 °C for 5 hours to remove moisture and cooled. A given amount of the *Theobroma cacao* shell was measured and added to the solvent-resin mixture, stirred continuously for five minutes, and was then cast in an aluminum mold with a diameter of 2m thickness by 8 m width by 10 m length. Composite was made to set at a temperature of 23±2 °C and relative humidity of 50±5% for 40 hours.

Characterization of Composite

Water Absorption

To determine the water absorption of the composites, a distinct assessment was made at 24 hours. This involved immersing five specimens of each formulation in distilled water at a temperature of 24 °C. Before immersion, specimens were dried in an oven for 24 hours and weighed. Subsequently, the specimens were placed in distilled water, and after the designated immersion periods, they were removed. Surface water was wiped off using a dry cloth, and the wet weight was measured [9].

The percentage of water absorption was then calculated using Equation 2

$$M\ (\%) = \frac{m_t - m_0}{m_0}\ X\ 100 \quad (2)$$

Where m_0 and m_t denote the oven-dry weight and weight after time of composite respectively.

Flammability Test

A 20 mm mark was precisely measured and delineated on each specimen. Subsequently, each specimen was horizontally clamped in a retort stand, with the marked 20 mm distance extending out of the clamp. The free end of the sample was ignited, and the time taken for the sample to ignite was meticulously recorded as the ignition time (I_t). The sample was then permitted to burn until reaching the 20 mm mark (Dp).

The relative rates of burning for the various samples were determined using Equation 3 [3].

$$Flame\ Propagation\ Rate\ (mms^{-1}) = \frac{Dp\ (mm)}{P_{t(\sec)} - I_{t(\sec)}} \quad (3)$$

Where Dp = Propagation distance measured in mm, P_t = Flame propagation time measured in seconds I_t = Ignition time measured in seconds.

Hardness Test

Hardness is referred to as the resistance of a material to indentation, the higher this resistance the harder the material, and vice-versa. The hardness test was carried out using the modified meyer hardness tester (ASTM D-2240) as modified [10]. The hardness for the samples was determined using Equation 4.

$$BHN = \frac{F}{\pi/_2(D - \sqrt{D^2 - D_i}} \quad (4)$$

Where F = Imposed load D =Diameter of the indenter D_i = Diameter of the indentation

Tensile Strength test

The dimensions of tensile and flexural test specimens were in accordance with ASTM D 638-14 and ASTM D 790-15, respectively. In the tensile tests, five specimens of composites were analyzed, with dimensions in agreement with the ASTM D 638 standard for each of the composites [11].

Scanning Electron Microscopy

A morphology study was carried out using scanning electron microscopy to evaluate the fractured surface of samples. The changes in morphology are important for predicting filler interaction with the matrix in composites [12].

X-ray diffraction

Powdered samples were pelletized and sieved to 0.074 mm. These were taken in an aluminum alloy grid (35 mm x 50 mm) on a flat glass plate and covered with a paper. Wearing hand gloves, the samples were compacted by gently pressing them with the hand. Each sample was run through the Rigaku D/Max-lllC X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan and set to produce diffractions at scanning rate of 2 0/min in the 2 to 500 at room temperature with a CuKa radiation set at 40 kV and 20mA. The diffraction data (d value and relative intensity) obtained was compared to that of the standard data of minerals from the mineral powder diffraction file, ICDD which contained and includes the standard data of more than 3000 minerals. Similar diffraction data means the same minerals to standard minerals which exist in the soil sample.

RESULTS AND DISCUSSION

Plate 2 shows the composites produced from cocoa shell powder and polypropylene.

Plate 2: Composites produced from cocoa shell powder and polypropylene

Water Absorption (%) of the Composites

The impact of filler content on the water absorption of treated and untreated cocoa fruit shell (CFS) combined with both unused and waste polypropylene-based composites as shown inn Plate 2, after 24 hours of immersion in water, is illustrated in Figure 1. An increase in CFS content, while keeping the PP matrix constant, resulted in a notable rise in water uptake. This phenomenon can be explained by the presence of polar hydroxyl groups in the cocoa fruit shell particles, which enhance water absorption. The hydrophilic characteristics of these particles, along with the increased interfacial area between the particles and the matrix, contribute to the

higher water uptake as the particle content in the matrix rises [13]. A significant increase in water absorption was noted, ranging from 58.8% to 80.1% for treated CFS/unused PP, and from 61.9 to 83% for treated CFS/waste PP. This pattern aligns with the findings of Kocaman and Ahmetli [14] which indicated that alkaline-treated shells absorb less water than their untreated counterparts. The highest water absorption rates were observed in untreated CFS/waste PP (62.6 to 88%) and untreated CFS/unused PP (61.9 to 83%). Typically, natural fillers in reinforced composites exhibit considerable water absorption due to micro-voids at the filler-matrix interface, which arise from the matrix's incomplete wetting of the filler [15].

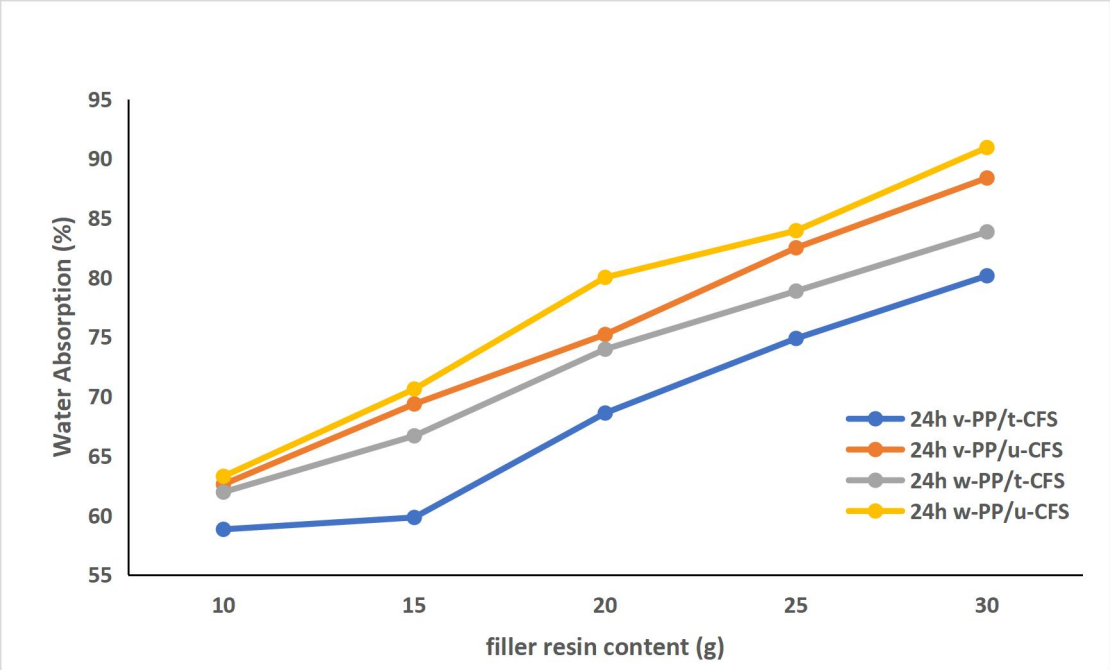


Figure 1: Effect of filler content on water absorption of treated and untreated cocoa fruit shell with unused and waste polypropylene-based composites at constant polypropylene after 24 h water immersion

Flammability Propagation Rate (mm/s) of the Composites

Figure 2 illustrates the flammability of both unused and waste polypropylene/CFS wood dust particle composites with a constant polypropylene film. As the content of wood dust particles rises, the ignition time decreases, resulting in an increase in the flame propagation rate from 0.28 to 0.08 mm/s for the unused polypropylene composite and from 0.36 to 0.09 mm/s for the waste

polypropylene composite. A gradual decline in the flammability of the composite was noted as the amounts of CFS wood particles and polypropylene films increased. These pores are interconnected, forming a network of void spaces that facilitates the passage of oxygen, thereby enhancing ignition. Generally, materials with a higher cellulose filler content exhibit greater density and resistance to oxidation. Consequently, reducing the porosity of the composite limits oxidation [16]. A higher oxygen index indicates that more oxygen is required to ignite the sample, complicating the ignition process. As the ratio of wood flour to polymer matrix increases, the oxygen index also rises, aligning with findings in existing literature [17].

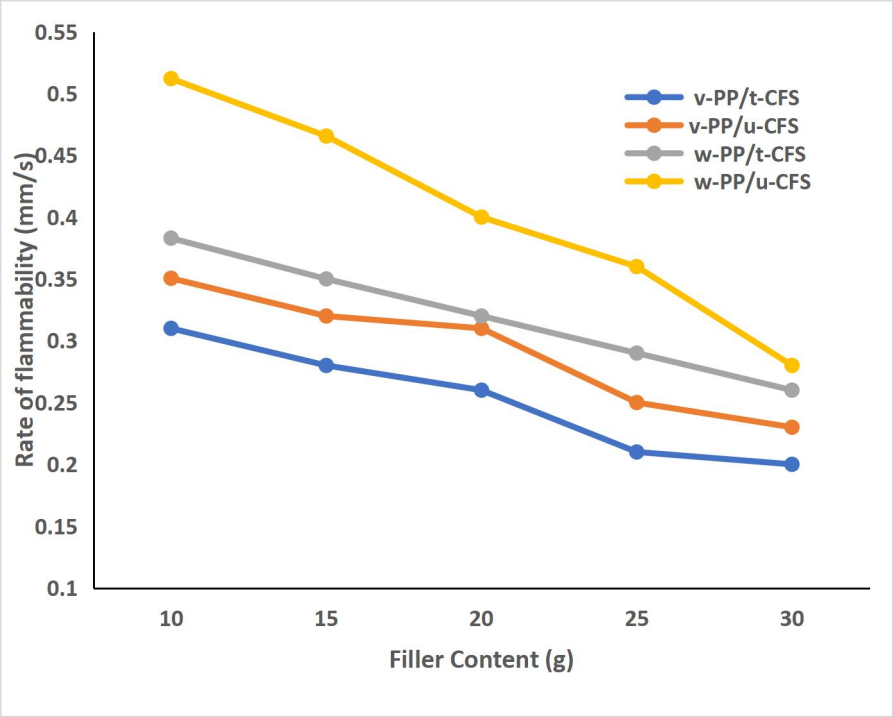


Figure 2: Effect of filler content on rate of flame propagation of treated and untreated cocoa fruit shell with unused and waste polypropylene-based composites at constant polypropylene.

Tensile Strength of CFS and polypropylene-based composites.

The results of the tensile properties for the composites developed with different filler loadings are shown in the Figure 3. It was noted that the tensile strength of the composite samples decreased as the filler content increased. A steady decline in tensile strength was observed as the filler loading rose from 10 g to 30 g. The addition of Cocoa shell filler to the polypropylene

matrix led to a reduction in tensile strength from 22.0 MPa to 14.17 MPa for unused/treated composites, from 18.67 MPa to 13.67 MPa for waste/treated composites, from 17.43 MPa to 11.67 MPa for unused/untreated composites, and from 16.17 MPa to 11.33 MPa for waste/untreated composites. It is evident that at filler loadings exceeding 15 g, up to 30 g, there was a significant drop in tensile strength. This can be attributed to the matrix's inability to adequately wet the filler at higher loadings. Consequently, the matrix struggles to effectively transfer the load to the filler. Additionally, at elevated filler loadings, agglomeration of the filler becomes almost unavoidable due to poor wettability during loading, which further impairs the composite's capacity to transfer stress efficiently.

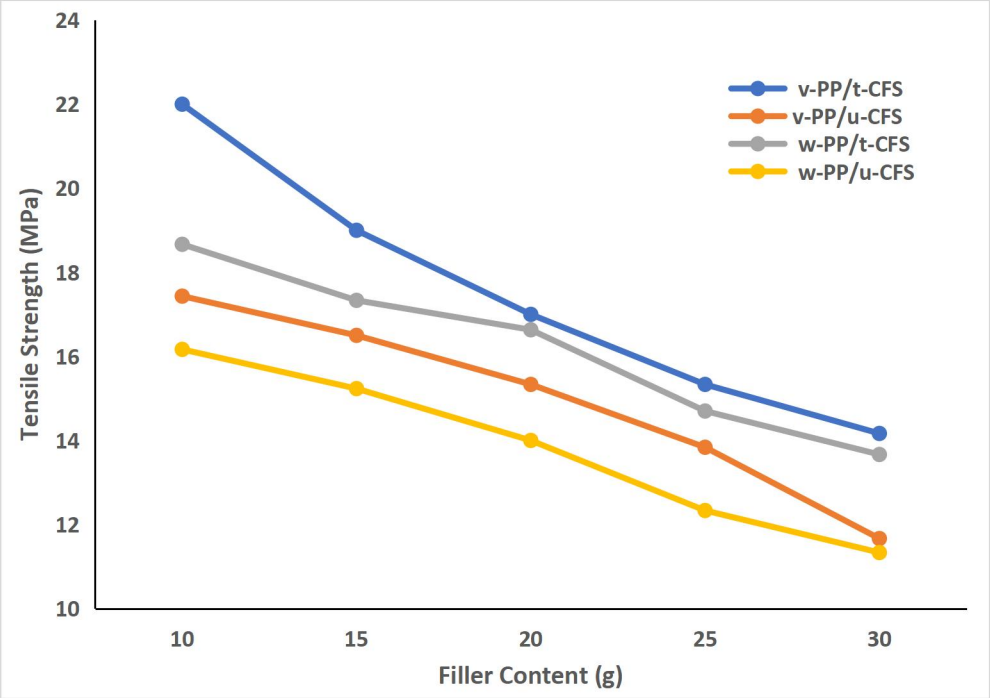


Figure 3: Effect of filler content on tensile strength of treated and untreated cocoa fruit shell with unused and waste polypropylene-based composites

Hardness of the Composites

In Figure 4, it was noted that the hardness decreased with an increase in filler content while keeping the polypropylene constant. For the composite made from unused polypropylene film, the hardness dropped from 59.4 to 22.8 MPa, whereas for the composite made from waste polypropylene film, it fell from 55.5 to 20.4 MPa. Interestingly, the unused polypropylene

composite showed higher hardness compared to the waste polypropylene composite. The results from the indentation tests indicated a linear increase in indentation values as the CFS wood particles in the composite increased, while there was a gradual decrease in indentation values with more polypropylene films in the composite. Figure 4 illustrates a decline in composite hardness as CFS wood particles increase, but an increase in hardness with more polypropylene films. This trend has been observed by other researchers studying natural filler-based composites. Obidiegwu & Ogbobe [18] noted a reduction in hardness values with higher flax filler content in high-density polyethylene/flax filler and polypropylene/flax filler composites. Similarly, Alomayri & Low [19] found that hardness values diminished with increased filler content in their research on polyurethane and empty fruit bunch blend composites. They explained this decrease as a result of the matrix's failure to properly encapsulate the filler strands, while the increase in hardness was attributed to the strong binding effect of the polymer resin. Notably, in this study, the composite made from unused polypropylene films and CFS wood dust particles demonstrated greater hardness than that made from waste polypropylene films and CFS wood dust particles. This difference may be due to the rubbery characteristics of the unused films. In contrast, the waste films may have undergone degradation from photo-degradation, leading to the formation of voids.

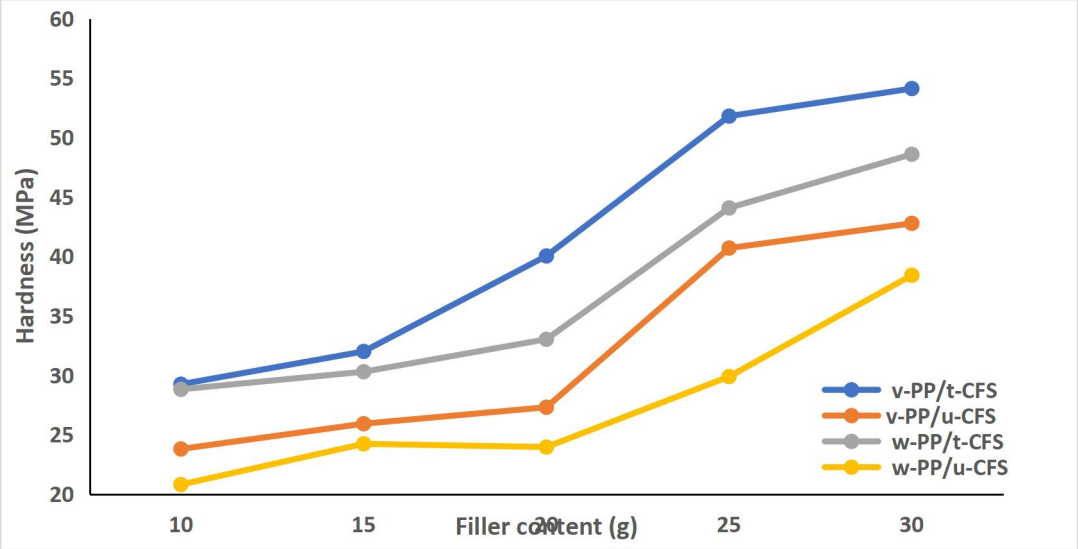
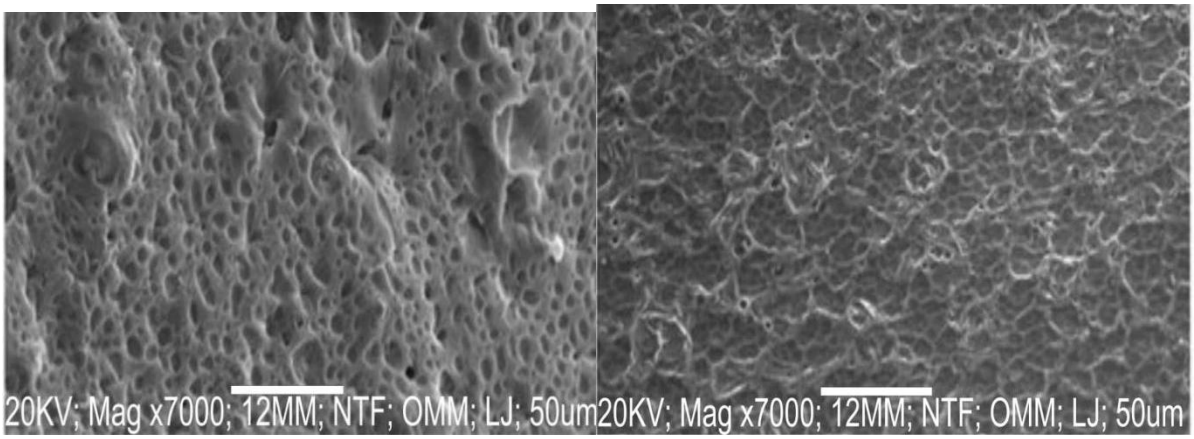


Figure 4: Effect of filler content on hardness of treated and untreated Cocoa fruit shell (CFS) with unused and waste polypropylene-based composites.

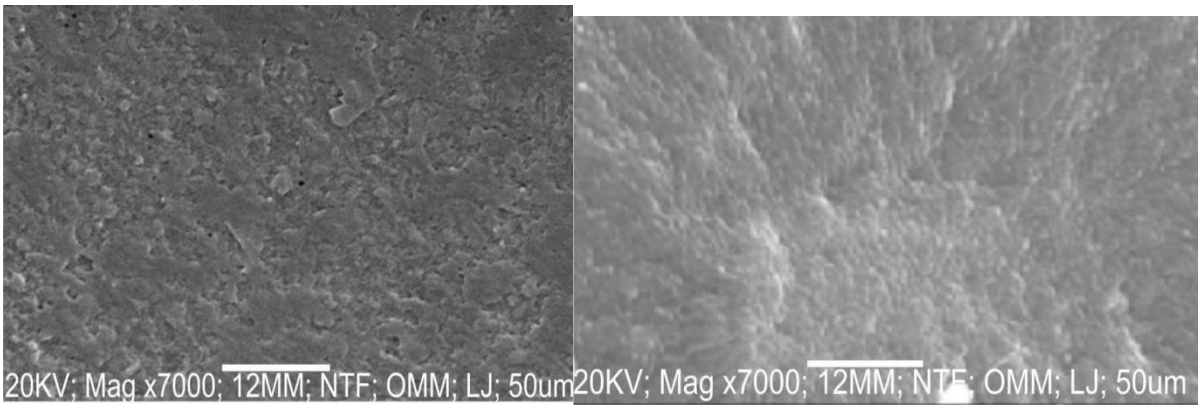
Scanning Electron Microscopy

Scanning electron microscope images of the polypropylene films/CFS wood dust particle composites at a filler loading of unused polypropylene (10 g) / CFS (5 g) composite and waste polypropylene (10 g) / CFS (5 g) matrices are presented in Figure 5(A) and (B) with a magnification of 7000×, respectively. These images reveal distinct clusters and gaps between the polymer matrix and the wood. It appears that wood fillers that were weakly bonded to the matrix have detached from it. The failure surface exhibits undulations with clear wood flour surfaces, showcasing visible tracheids and lumen. This indicates weaker areas at the wood-wood interface and the weakest points in the polymer matrix. It suggests that the interface between the wood and polypropylene matrix is compromised due to inadequate dispersion and wettability between the two phases. The dispersion of wood dust particles in the unused polypropylene, shown in Figure 7 (C), and in the waste polypropylene, shown in Figure 6 (D), is uniform. This uniformity may be due to the different grades of plastic or the higher ratio of plastic used. In some instances, parts of the wood lumen were filled with plastic, which could enhance the strength of the composites through mechanical interlocking. However, as the wood content increased, the polymer matrix became less uniformly distributed, leading to many wood fillers being in direct contact with each other, which resulted in poor bonding and adhesion at the interface. Nonetheless, as the number of plastic films increases, better bonding and adhesion at the interface are observed due to a more even distribution within the composite matrix.



(A) (B)

Figure 5: SEM images (×7000) of (A) unused polypropylene 10 g / CFS 5 g composite and waste polypropylene 10 g / CFS 5 g composite (B) respectively.



(C) (D)

Figure 6: SEM images (×7000) of (C) unused polypropylene 10 g / CFS 5 g composite and waste polypropylene 10 g / CFS 5 g composite (D) respectively.

XRD for Cocoa Fruit Shell

In contrast, the untreated composite exhibits broader and less intense peaks, indicating a less crystalline structure. This implies that the untreated composite contains more amorphous content, which can lead to weaker mechanical properties. The absence of a well-defined crystalline structure results in weaker fiber-matrix adhesion, consequently reducing tensile strength and potentially impairing mechanical performance [20]. The comparison between treated and untreated composites shows that alkaline treatment significantly enhances crystallinity, leading to improved structural ordering and better mechanical properties. The X-ray diffraction pattern for alkaline-treated Cocoa fruit shell in Figure 7 displays several peaks at specific 2-theta angles that correspond to diffraction from various crystallographic planes. These peaks, identified at Miller indices (111), (101), (112), and (114), indicate a well-defined crystalline structure. The presence of these peaks signifies a crystalline phase, which is typically linked to a specific atomic arrangement [21]. Quantitative XRD analysis involves measuring the intensity and area under each peak to assess the abundance of each crystalline phase. This process allows for a more thorough evaluation of the phase composition. The intensity of the peaks as shown in Figure 8, especially the (111) peak, can suggest high-density atomic planes, commonly found in

face-centered cubic (FCC) structures, while the (101), (112), and (114) peaks provide further insights into the crystal's lattice spacing [22]. The observed XRD pattern aids in phase identification by comparing the experimental data with standard reference patterns. The alkaline treatment may have modified the crystal structure or induced phase transformations, such as recrystallization or partial dissolution, thereby enhancing the crystallinity of the CFS sample [23]

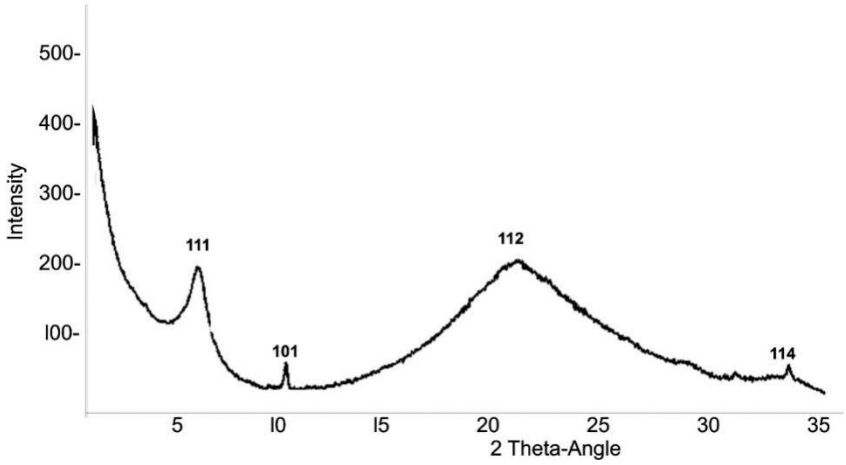


Figure 7: X-ray diffraction results for Alkaline Treated Cocao Fruit Shell

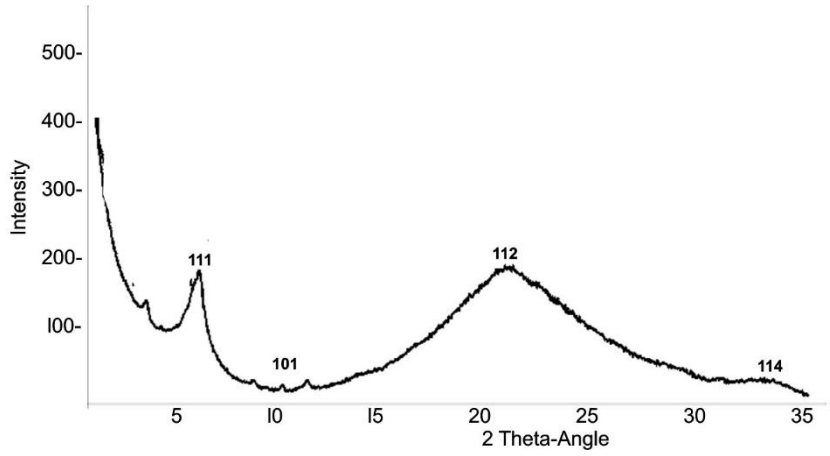


Figure 8: X-ray diffraction result for Untreated Cocao Fruit Shell

The X-ray diffraction pattern for untreated Cocoa Fruit Shell displayed in Figure 8 shows peaks at similar 2-theta angles as the alkaline-treated sample, specifically at 111, 101, 112, and 114.

This similarity indicates that both samples have a comparable crystalline structure or phase. However, the untreated CFS has lower peak intensities, particularly for the (111) peak, which suggests reduced crystallinity or differences in phase composition due to the absence of treatment. The untreated sample also presents broader peaks, especially at the (111) and (101) planes, implying smaller crystalline domains or a higher proportion of amorphous content [21]. In contrast, the alkaline treatment improves crystallinity, resulting in sharper and more intense peaks, likely due to changes in crystallite size or phase transformation. This enhancement in crystallinity and phase growth is evident from the sharper XRD peaks in the treated sample. The alkaline treatment may have facilitated crystallite growth, deprotonated surface components, or decreased amorphous content, leading to a more organized atomic arrangement [22]. A quantitative analysis of peak intensities can shed light on the relative amounts of crystalline and amorphous content. The untreated CFS displays broader peaks and lower intensity, indicating reduced crystallinity compared to the treated sample. The alkaline treatment likely boosts crystallinity, as shown by the sharper and more intense peaks in the treated sample. Further quantitative analysis would be necessary to accurately assess the differences in crystallinity and phase composition [23].

CONCLUSION

Agricultural byproducts such as cocoa shell powder present sustainable options for composite production. This study emphasizes the potential of blending *Theobroma cacao* shell powder with polypropylene to create eco-friendly materials. Characterization shows that the amount of filler affects water absorption, flammability, tensile strength, and hardness. Composites that have been treated demonstrate enhanced crystallinity and mechanical properties as a result of alkaline treatment. SEM and XRD analyses validate the effects of filler dispersion and crystallite structure. These results offer valuable insights for optimizing composite materials for use in domestic applications.

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