Physical and mechanical properties of Entada mannii particulates reinforced composites

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ABSTRACT

This paper reports the physical and mechanical properties of Entada mannii particulates reinforced composites. Polypropylene (PP) based composites filled with Entada mannii ash particulates was produced using the compression moulding. The thermal properties, structural characteristic and morphology of the composites and ash particulate were studied. Tensile properties, impact strength and flexural properties were used to evaluate the mechanical behaviour of composites. Tensile properties, elastic modulus and hardness properties of the fiber ash polymer composites were improved with aggregate fiber ash loading by 58 %, 41 % and 56 % relative to pure PP composites while there was a drop in impact strength by 56 % as compared with pure PP. Flexural strength of the fiber ash reinforced composites was enhanced significantly with increasing ash particulate loading by 45% and 32% respectively. The Fourier transform infrared spectroscopy (FTIR) revealed that changes occur due to stretching of hydrogen bond network and lignin was remove completely during carbonation process. Fracture surface morphology of fiber ash reinforced composites gave credence to the fact that good wetting between the particulate and the matrix was achieved.

1. Introduction

Sustainable natural filler and fibers derivatives obtained from corn husk fiber, wheat husk, rice husk, hemp fiber and sisal fiber have been effectively used in development of advanced polymeric composites [1, 2]. These fillers are introduced into the polymer composites to improve their rigidity, stiffness, mechanical, thermal and electrical properties. Particulates fillers are highly demanded in commercial and domestic sectors due to their specific properties, biodegradability, and renewability [3]. In considering environmental awareness, natural fillers are ecofriendly and pose no health hazard as related to their synthetic fibers counterparts [4]. Some of the application of natural filled polymer composites are aerospace, construction industries, automobile parts, and light weight applications [5]. Consequently, automotive body such as bumper and the interior parts are currently replaced by fiber reinforced composites, targeting less fuel consumption at reduced cost [6]. Additionally, studies have shown that fillers such as mineral fillers, fly ash and limestone powder have enhanced the properties of filled polymer matrix composites (PMCs) [7, 8]. Even though natural filled composites have demonstrated their worth in weight reduction of polymer composites suitable for various applications, there are certain drawbacks such as degradation by moisture absorption, poor filler-matrix interfacial adhesion, non-uniform filler sizes and incompatibility of fillers with the matrix.

Previous research works have revealed that mechanical properties of particulates filled polymeric composites depend particularly on the size, shape, and distribution of fillers in the polymeric matrix, as well as the integrity of the interface between the filler and polymeric composite matrix [7, 8]. Conversely, the quest to develop novel fiber reinforced composites (FRP) for both thermoplastic and thermoset matrix using low cost and low density reinforcing fillers with enhanced mechanical and physical properties was studied by several researchers [9, 10]. Asuke et al. from their investigation concluded that periwinkle shell particulates promotes good adhesion with the epoxy matrix as related to unprocessed particulates. Similarly, the wear resistance of process...
carbonized composites was improved while the uncarbonized composites was brittle in nature [11]. Agunsoye et al. in their experimental studies observed that even distribution of the bagasse fillers enhanced the mechanical properties of the composites [12]. Tensile and flexural properties of the polyethylene composites increases with an increase in filler loading up to a peak of 20 wt.% and 30 wt% for unprocessed and processed bagasse filler respectively [12]. Oladele et al. developed polyester composites by varying the bone ash and the particulates [13]. It was found that, reinforcing polyester with bone ash particulates displayed an improvements in tensile strength with 8 wt. % bone particle as compared with unreinforced composites. Njoku et al studied the ability of periwinkle shell on enhancing the properties of polyester composite [14]. Their research findings show that, tensile properties and modulus of elasticity of developed filled composites were enhanced with reduction in the particle size. Akil et al. reported on the influence micro particles and weight percentage of sisal natural fiber and maleic anhydride on mechanical and properties of polymeric composites [15]. They also concluded that, addition of fibers weight percent and silica particulates with the matrix exhibited major improvement on the tensile strength and elastic modulus of the reinforced composites.

Shiyappa et al. studied the potentials of coir and rice husk reinforced vinyl ester composite and established that flexural strength increases with fiber volume [16]. Similarly, the composites tensile strength was increased by 27.8 % and again decreased by 20.85 % respectively.

Hence, the research findings have shown the prospective use of filled particulates in strengthening of particulate reinforced composites for various applications. Incorporating filler particulates in polypropylene composites in various weight ratios has successfully improved physico-mechanical properties of polymeric composites [17, 18]. Additionally, to develop a good nano composite material, it is important to consider some factors such as filler size and processing techniques to stimulates good wettability with the matrix interface [19]. However it is imperative to explore the use of carbonized and uncarbonized Entada mannii fiber ash particulate obtained from lignocellulosic fiber as prospective reinforcement of thermoplastics composites. Although, the possibilities of using Entada mannii fiber in reinforcement of polymer composites have drawn the attentions of researchers [20]. The present study investigate the physical and mechanical properties of Entada mannii particulates reinforced composites suitable for light weight applications.

2. Materials and methods

2.1. Materials

The materials applied for the composites development are: Entada mannii bast of density 5.3 g/cm³ was collected in Ondo State, Nigeria; Polypropylene-Homopolymer granules of melt flow rate, 230 °C/2.16 kg, density of 0.903 g/cm³ and size 4 mm was supplied by Safripol (Pty) Limited, South Africa. The 5 % Maleic anhydride granules (MAPP) of size 3 mm, density 0.904 g/cm³ and the melting temperature of 135 °C was purchased by Polymer Division of, Tshwane University of Technology South Africa and supplied by Sasol chemicals, Sasolburg, South Africa.

2.2. Methods

2.2.1. Production of ash particulates from fiber

The fibers were collected from plant stem by conventional peeling technique and dried in sunlight. The dried fibers were later placed in an oven at 65 °C for 2 days to remove essential moisture for proper filler – matrix bonding. The dried fibers obtained were subsequently burnt in a crucible furnace at 200 °C. Afterwards, the ash particulates were conditioned in a muffle furnace at 100 °C to remove retained moisture for 5 h. The unprocessed fibers were similarly reserved for use as control specimen. Entada mannii fiber ash obtained from the controlled burning process was conditioned at 200 °C in a muffle furnace and the average particle size distribution analysed using a Horiba dynamic light scattering analyzer was approximately 0.456 μm in Figure 1.

2.2.2. Composite production

The composites were produced using the compression moulding technique. The Entada mannii fiber ash of 10 wt. %, 20 wt. %, and 30 wt. % were varied with polypropylene (PP) matrix and 5 % Maleic anhydride polypropylene (MAPP, Permanol 603) at room temperature for 5 min using a Jones high speed mixers supplied by Dick Peters, Netherlands for proper homogenization. The samples were fed through the upper chamber of the mixer with two stump blades while maintaining speed of 5000 rpm at 50 °C for 1 h. The total compositional weight was maintained to a 500 g per batch size. The sample materials were transferred to an extruder at 200–250 °C and a rotor speed of 40 rpm to produce the extrudate. The samples obtained was crushed into pellet using the industrial granulator and evenly dispersed in steel mould. The particulates composites were compounded at 190 °C below a constant pressure of 100 MPa. Afterward, the mould was allowed to cool using another compression mould to allow easy removal of the composites under a constant pressure of 100 MPa. The particulates composites sheets formed were approximately 160 × 160 × 4 mm thickness for both untreated and fiber ash composites. Entada mannii ash composites production flow chart is presented in Figure 2.

2.3. Surface morphology examination

The morphology of the fiber ash, untreated fiber and polypropylene reinforced composites were studied using a JEOL JSM-7600F scanning electron microscope (SEM). The samples were thoroughly cleaned and positioned in a void chamber to dry and coat the polymer surface with 100 A thick irradium in JEOL sputter ion coater at 15 keV.

2.4. Fourier transform infrared spectroscopy (FTIR) of the fiber and ash

Functional groups of fiber ash and unprocessed fiber constituents were analysed using the Thermo Nicolet Fourier Transformed Infrared Spectrometer (FTIR). Fiber ash of 2 mg were hard-pressed in a metal disc and the analysis was carried out from 400 to 4000 cm⁻¹ wave numbering and 4 cm⁻¹ resolution. About 100 spectrum scan was performed the weighted average was recorded.
2.5. X-ray diffraction analysis of fibers

The XRD analysis of fiber ash and unprocessed fiber was carried out using the Phillips X’Pert diffractometer. The fiber ash particulate were pressed into metal disks using a steel mould cylinder of 15 mm diameter and pressure of 20 MPa. The diffraction was measured between 5° and 50° at 40 kV and 50 mA [21].

2.6. Mechanical properties of the composites

Composites tensile strength and Young's modulus of elasticity were performed in accordance with ASTM D638 [19, 21] using a tensile testing machine. The composite specimen were cut into 4 mm × 150 mm of thickness and gauge length, respectively at 10 mm/min strain rate and load 10 kN. Six identical samples were tested and the values obtained was recorded.

The Izod impact testing machine was used to conduct the impact test of the composites in accordance with standard ISO180 [22]. The test specimen dimensioning 80 mm × 10 mm x 2 mm were notched at 0.03 mm radius and fractured at 2 m/s impact velocity with hammer head of 75 J. Five specimen were tested and the weighted fraction was recorded.

Flexural test of the specimen was measured according to ASTM D790 on the three-point bending testing method using the universal testing machine. Specimen were prepared dimensioning 140 mm × 140 mm x 2

![Flow Chart of the Experiment](image1)

**Figure 2.** Flow Chart of the Experiment (a) Granulated *Entada manni* fiber (b) Entada manni fiber ash (c) Fiber ash composites.

![SEM Images](image2)

**Figure 3.** Showing SEM of (a) *Entada manni* fiber ash (b) EDX of *Entada manni* fiber ash (c) untreated *Entada manni* fiber.
mm and speed of 2 mm/min. Five repetitive specimen were verified and their average was recorded.

3. Results and discussions

3.1. SEM description of the Entada mannii fiber ash and untreated fiber

Representative SEM micrograph and EDX of the Entada mannii fiber ash produced are presented in Figures 3(a) and 3(b). The particle size from examination of the micrograph is approximately 0.45 μm. The fiber ash was observed to be made up of majorly calcium C, potassium K and carbon C oxygen O as elemental composition. Figure 3(c) revealed the untreated fiber which is characterized with smooth surface owing to the occurrence of fiber constituents (Lignin and Hemicellulose and wax). SEM analysis of the Entada mannii fiber ash particulates revealed the presences of CaO which is hard in nature and irregular in shape.

3.2. Fourier transformed infrared spectroscopy of the ash fiber

The IR spectra of fiber data ash and unprocessed Entada mannii fiber are shown in Figure 4. The absorbance peak at 3020 cm⁻¹ and 3335 cm⁻¹ of the untreated fiber and fiber ash is credited to O–H stretching of hydrogen bond network and complete removal of lignin during carbonation process of the fiber ash [23, 24]. The Peak at 2700 cm⁻¹ and 2900 cm⁻¹ revealed that C–H stretching vibration of aldehyde and methylene groups in the cellulose and hemicellulose molecules [25]. This became less intense with a partial disappearance of the lignin and hemicellulose of the ash fiber with breaking of the hydrogen bond between the cellulose and hemicellulose compounds. The peak 1620 cm⁻¹ and 1550 cm⁻¹ IR spectra regions show a reduction in the occurrence of fiber constituents (Lignin and Hemicellulose and wax). Figure 5(a) revealed that C–C–O stretching vibrations [26]. The peak showing 1450 cm⁻¹ is ascribed to the CH₂ symmetric bending, while the absorbtion peak observed between 620 cm⁻¹ and 1230 cm⁻¹ can be associated to the presence of lignin [20, 27].

3.3. The XRD of Entada mannii fiber ash

The X-ray diffractograms of the fiber ash are presented in Figure 5 (a) and (b). Figure 5(a) revealed the quantitative phase analysis of Entada mannii fiber ash. This confirmed the presence of calcium oxide (CaO) with 53.9% which signify the major crystalline phase. Also, the presence of silicon oxide (SiO₂) with 46.1% as the amorphous phase and the traces carbon respectively. These represent the major chemical compounds present in the Entada mannii fiber ash which is in line with the work of [11, 12]. However, Figure 5(b) illustrates a major sharp peak demonstrating the crystalline phases of the profile at around 2θ = 29.32° which indicates the crystallographic plane (0 0 2). The broad peaks at the profile around 2θ = 13.16° and 18.10° represent the amorphous phase (1 1 0) containing SiO₂ and represented the amorphous region of the fiber ash where the concentration is at minimum. It was found that the peaks of the fiber ash is more crystalline at the higher temperature between 2θ = 29.32° and 33.98° consisting majorly CaO, with increase in calcium and carbon contents. The peaks overlays where there is increase in the amorphous contents of the fiber ash particulates which appears to be broad [28]. When the crystallinity peaks content is high, the fiber ash are more evident and while the ash contained large amount of amorphous materials such as SiO₂ and traces of potassium and carbon from the combustion process, these peaks is flattened and appeared as wide peaks [27, 28].

3.4. Tensile strength and modulus of elasticity of the composites

The stress-strain plot of polypropylene matrix and the variants in tensile properties of Entada mannii particulates composites are presented in Figure 6 (a) and (b). Apparently, the tensile strength of the fiber particulates composites increases with an increase in particulates loading. On the average, composites reinforced with Entada mannii fiber ash improved considerably compared with untreated fiber and pure PP composites. Tensile strength of the composites increases by 58% relative to pure. This improvement is credited to good wettablility of the ash particulates in the matrix which promotes good adhesion between ash particulates; and the matrix impinges the movement of the crack propagation when stress is applied. The fiber ash penetrates into the matrix interphase and displaces air bubbles trapped within the polymer [26, 28]. At 20 wt. %, untreated particulate composites experienced a slight drop in tensile strength by 27%. This could be as a result of poor agglomerations of fiber particulates with the matrix which act as hindrance that imping the chain crack propagation, and hence, fractures at lower

![Figure 4. IR spectra of the Entada mannii fiber ash and untreated Entada mannii fiber.](image-url)
stress level. The tensile strength of untreated fiber composites had a lower value as compared with the ash composites. This is because of the presence of fiber constituents such as lignin and hemicellulose which may account for poor fiber-matrix interfacial bonding and the fiber fractured at lower values.

The representative variations in stress-strain curve of the pure polypropylene matrix, and comparison of tensile strength of the *Entada mannii* fiber ash with untreated *Entada mannii* fiber are as displayed in Figure 6 (a) and (b) respectively.

The Young’s modulus of elasticity of the *Entada mannii* ash particulates composites is presented in Figure 7. The experimental result show that variations in elastic modulus is similar to results obtained for tensile strength in Figure 6(b). The elastic modulus of *Entada mannii* ash particulates composites increases with respect to increase in particulate volume. This improvement is owed to the stiffness of composites and removal of the fiber constituents which enhanced the fiber ash-matrix interfacial adhesion. This is comparable to the findings reported by Agunsoye et al. [12]. The tensile modulus increases from 1566.8 MPa to 1783.2 MPa for both 20 wt.% and 30 wt.% of ash particulates composites, respectively. It is also observed that during the carbonation process, the fiber constituents and moisture contents that could be detrimental to ash particulates – matrix interface were removed and thereby increases the even distribution of the ash in the matrix. On the other hand, presence of the fiber constituents accounts for the poor fiber-matrix adhesion and restrict the mobility of polymer molecules when stress is applied. The incorporation of ash particulates into thermoplastic matrix improves the elastic modulus of the ash composites by promoting good wettability of the ash particulate in the matrix up to 30 wt. %.

3.5. Impact strength

The comparison of the impact strength of the fiber ash particulates and unprocessed fiber composites are shown in Figure 8. Apparently, impact strength of fiber ash composites decreases with increase in particulates loading. This is remarkable for the 20 wt. % and 30 wt. % ash particulates and untreated fiber composites, respectively. With a drop
amounting to 56% and 46% as compared with unreinforced ash composites. This could be ascribed to uneven wettability of the particulate composites [28, 29, 30]. The decrease in composites strength at higher particulates loading is attributed to weak interaction between the filler and matrix as the particulates loading increases. However, the energy to initiate the crack decreases and hence the composites fracture at lower values [29, 30]. Untreated fiber composites also had poor fiber-matrix interfacial adhesion as a result of presence of the fiber components such as lignin and hemicellulose. Progressive improvement was observed for the 10 wt.% in the impact strength as a result of the improved distribution of the ash particulate in the matrix. Modification of the ash

Figure 6. Showing (a) stress-strain curve of the pure polypropylene matrix and (b) comparison of tensile strength of the Entada mannii fiber ash and untreated Entada mannii fiber.

Figure 7. Comparison of Elastic modulus of the Entada mannii fiber ash and Untreated Entada mannii fiber.

Figure 8. Comparison of impact strength of the Entada mannii fiber ash and untreated Entada mannii fiber.

Figure 9. Comparison of flexural strength of the Entada mannii fiber ash and untreated Entada mannii fiber.

Figure 10. Comparison of flexural modulus of the Entada mannii fiber ash and untreated Entada mannii fiber.
particulate during combustion also led to increased rigidity and stiffness of the composites compared pure composite.

3.6. Flexural strength and modulus of the composites

Figure 9 revealed the flexural strength of the ash particulate composites filled with fiber ash and untreated Entada mannii fiber ash are obtainable in Figure 9. The result show that the flexural strength of the ash particulates and the untreated fiber ash reinforced composites significantly increases as the particulates loading increases. The increase in particulates loading was remarkable for 30 wt. % composites increasing by 45 % and 34 % relative to pure polypropylene. This could be linked to the even spread of the ash in the matrix, and the consequent improvement of the fiber ash-matrix interfacial bonding [30]. The 30 wt.% composites exhibited a higher loading carrying capacity, and which indicated the less brittleness of the composites and slow down the crack propagation when load was applied [29, 30].

Flexural modulus of the fiber ash and untreated fiber ash composites are presented in Figure 10. The results confirmed that flexural modulus increases with an increase in ash particulates volume for all the composites. This could be ascribed to the presence of the coupling agent (MAPP) which enhanced better adhesion between the filler and the matrix. In addition, this also implies that increase in surface area of the fiber ash offered good enhancement with the matrix as compared with the unprocessed fiber ash. The trend obtained is similar to the flexural strength in Figure 9. However, the good dispersion of the fillers in the matrix interface improved the bonding strength and subsequently reduces the accumulation stress on the composites. This is in agreement with [12, 30].

3.7. Hardness properties of the composites

Figure 11 illustrates the hardness value of the fiber ash and unprocessed composites. It is apparent that the hardness value increased with ash particulates weight percent. The increase in the hardness was due to hard surface of the composites; and could be accredited to large ash surface area. Hence, leading to even dispersion of the ash particle and the fiber into the matrix. However, due to the absence of the fiber elements such as lignin and hemicellulose from the ash, the addition of the ash particle into the matrix interface resulted in the superior hardness values relative to pure PP and untreated composites. The increase in the hardness value also led to an inter-particle gaps, which tend to slow down the nucleation of the crack in the matrix interphase by evenly distribution in the nucleation site thereby increasing the hardness of the composites [7, 29]. This is evident with the 30 wt.% ash particulate which is higher than Pure PP by 60 % relative to the tensile properties in Figure 6(b). The hardness value was observed to follow a higher trend as the filler
aggregate increases and consequently promoting a hard and brittle interphase between fiber ash and fiber within the polymer matrix [7, 30].

3.8. Fracture analysis of the composites

The surface features of untreated and Entada mannnii fiber ash reinforced polypropylene composites are presented in Figure 12. Figure 12(a) which shows the image of the unprocessed Entada mannnii fiber ash particulates matrix composites, reveal that the fiber was characterized with fiber debonding and fiber pullout. This indicated a weak interfacial bonding between the fiber ash and the matrix due to existence of fiber constituents such as lignin and hemicellulose. Similarly, Figure 12(b) which shows the fracture images of the 10 wt.% fiber ash reinforced composites revealed a homogeneous dispersion of the fiber ash particulates in the matrix. The wavy pattern is an indication of greater energy dispersion [30].

Figures 12(c) and 12(d) show the fracture images of the 20 wt.% and 30 wt.% Entada mannnii fiber reinforced composites, respectively. At 20 wt.% and 30 wt.% the morphological features reveal smooth surfaces having higher surface area interactions with the matrix. This suggest good wettability between the ash particulates with the matrix. Hence, corroborating the results obtained from the tensile strength in Figure 5. This also implies that mechanical properties of the composites was improved with good interfacial bonding. Additionally, an increase in surface area of the ash particulates through an efficient wettability in the composites is attributed to the increase in tensile strength as observed in Figure 5.

4. Conclusions

The physical and mechanical properties of Entada mannnii particulates reinforced composites were investigated. From the results presented, incorporating fiber ash particulates within polypropylene composites improved the mechanical properties of the various composites, with respect to fiber/ash particulates loading. The tensile properties of the ash particulates composites were improved by 58 % compared to pure PP composites. This was basically due to higher concentration of particulate loading, good wettability and excellent interfacial adhesion between the ash particulates and polypropylene matrix. There is a significant increase in the flexural strength and Young’s modulus of elasticity with an increase in the particulates weight percent of the composites. Marginal reduction in impact strength of ash particulates composites at 20 wt. % and 30 wt.% peak point respectively was observed as the fiber loading increases. Fractographic studies showed a fairly uniform distribution of the ash particulate within the matrix with increasing particulate loading while the untreated fiber was characterized with fiber pullout and debonding. The research has revealed the successful fabrication of Entada mannnii fiber/ash particulates by compression moulding with improved mechanical and structural properties of the composites for light weight.

Declarations

Author contribution statement

O. P. Balogun, K. K. Alaneme: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

J. A. Omotayoibo: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

A. A. Adediran: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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