



Article Evaluation of the Physical and Mechanical Properties of Short Entada mannii-Glass Fiber Hybrid Composites

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Abstract: This study investigates the physical and mechanical properties of short Entada manniiglass fiber polypropylene hybrid composites. The polymeric hybrid composite was produced by combining different ratios of Entada mannii fiber (EMF)/glass fiber (GF) using the compression molding technique. The tensile properties, compressive strength, impact strength and hardness were evaluated while the fracture surface morphology was examined using the scanning electron microscope (SEM). It further evaluates the moisture absorption and percentage void content of the developed composites. The experimental results show that tensile, compressive, impact and hardness properties of all the hybrid composites were significantly improved as compared with single reinforced composites. Specifically, hybrid composites (EMF/GF5) revealed an overall tensile strength of 41%, hardness of 51% and compressive strength of 47% relative to single reinforced composites, which can be ascribed to enhanced fiber-matrix bonding. The chemical treatment enhanced the EMF fiber surface and promoted good adhesion with the polypropylene (PP) matrix. Moisture absorption properties revealed that the addition of EMF/GF reduces the amount of moisture intake of the hybrid composites attributed to good cementing of the fiber-matrix interface. Morphological analysis revealed that single reinforced composites (EMF1 and GF2) were characterized by fiber pullout and deposition of voids in the composite as compared with the hybrid composites.

Keywords: Entada mannii-fiber; adhesion; glass fiber; mechanical properties; polypropylene; compression molding

1. Introduction

The last decades have seen an increase in demands in the use of environmentally renewable and biodegradable natural fibers to produce thermoplastic hybrid composites. These demands have attracted the attention of both researchers in academia and professionals from the industries. Nowadays, studies of the inherent strength and toughness of the hybrid natural fibers are of high interest [1–3]. These studies have warranted the need to use low-cost, low-density and abundantly available *Entada mannii* natural fibers to produce eco-friendly and renewable thermoplastic composites without compromising the quality of the final product [4,5]. *Entada mannii*-fiber has shown low-density, high toughness, good heat resistance as an ideal material for polypropylene composites, desired in automobile, biomedical and other applications [6]. Despite the tremendous studies into the industrial usage of natural fibers, there exist some drawbacks. The poor mechanical properties of natural fiber and higher moisture absorption compared with synthetic fibers remains a challenge and daydream adventure [7,8]. Additionally, there exist a significant level of difficulties



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). in the fabrication of natural fibers into sheet or laminates due to lack of cohesion partly influenced by high viscose thermoplastic polymer during the consolidation process [9]. These drawbacks can be overcome using a chemical treatment to modify the fiber surface morphology and improve the mechanical interlocking with the polymer composites [10]. Additionally, small amounts of synthetic fibers (glass fibers) can be added to natural fibers which act as a chemical barrier to reduce moisture intake of the composites [11,12].

Presently, the quest of manufacturing biodegradable and sustainable engineering components motivates the use of hybrid composites because they combined the unique mechanical strength of synthetic fibers with the physicomechanical properties of natural fibers [11,13]. Researchers have studied the hybridization of glass fibers with natural fibers, such as Jute/sisal [14], Sugar palm [15], Banana/hemp [16], Curaua [17], Betel nut [18], to produce hybrid composites. This was achieved by modifying the physicochemical properties of the natural fibers and hybridizing with the glass fibers while maintaining the high strength and low density of the finish products [19,20]. These hybrid composites offer major advantages such as easy processing and maintaining a balance between composites cost and performance benefits to both industrial production and academic environment [21]. For instance, in transportation industries, hybrid reinforced composites are employed to reduce weight and increase fuel efficiency without sacrificing safety [21,22]. However, judging from the previous research on hybrid composites, no literature has reported the combination of Entada mannii-fibers and glass fibers to produce hybrid composites. Therefore, it is imperative to evaluate the advantages of intermingling synthetic (glass fiber) and natural fiber (Entada mannii-fiber) to reduce the cost and increase environmental aging resistance. In this study, glass fibers (GF) and Entada mannii-fibers (EMF) of varying weight ratios were incorporated into the polypropylene matrix to improve the properties of the polypropylene hybrid composites at reduced production cost compared to the single reinforced composites. The objective of the research is to fabricate novel graded polypropylene hybrid composites with improved mechanical and physical properties suitable for biomedical applications. Further studies of the significance of Entada mannii-fiber (EMF) and glass fiber (GF) on water absorption properties, void contents and morphological analysis of developed composites were investigated.

2. Materials and Methods

2.1. Materials

Glass fiber of average length of 2 mm length and density 2.9 g/cm³ was obtained from Safripol, South Africa. *Entada mannii* of average fiber length of 2 mm–4 mm was obtained from a tropical forest in Ikare Akoko, Nigeria. Polypropylene of melt flow index of 235 °C and average density of 0.9 g/cm³ was obtained from Safripol, South Africa. The 5% Maleic anhydride polypropylene (MAPP) coupling agent was used to increase the fiber–matrix bonding.

2.2. Fiber Chemical Modification

The chemical modification of the natural fiber was determined using the neutral detergent fiber method. Natural fibers (EMF) were removed from the fiber bundles method and chemically treated with 10% NaOH alkali solution in a water shaker bath at 50 °C for 4 h. This enhanced the removal of the cementing materials (lignin, hemicelluloses, waxes and impurities) thus increasing fiber surface adhesion. Afterward, the residue was washed with water and dried for 2 days. The dried fiber was crushed into a short fiber of 3.5 mm using the guillotine machine for the composite fabrication.

2.3. Composite Mixing and Extrusion Process

The dried fibers and glass fibers of an average length of 3.5 mm were mixed with polypropylene matrix and 5 wt% MAPP compatibilizers using the Tubular T2F mixer at room temperature for 25 min. The mixture was fed into a twin-screw extruder with a diameter of 3 mm equipped with barrel temperature zones of 165–190 °C. The extrudate

produced was passed through the cooling bath and dried for 24 h. The cooled extrudate was granulated into a pellet of an average size of 3 mm using the guillotine machine and the obtained pellets were finally dried for 10 h. The pellets were transferred to the compression molding machine and compressed for 15 min at 185 °C and 100 MPa. The percentage weight for each specimen dimensioning 2.5 mm × 2.5 mm × 4 mm, theoretical density and experimental densities of the fabricated composites are presented in Table 1. Figures 1a,b show the short *Entada mannii*-fiber and hybrid composites, respectively.

Code	EMF	GF	PP	Theoretical Density	Experimental Density	Void
	(wt%)	(wt%)	(wt%)	(g/cm ³)	(g/cm ³)	Content
EMF1	20	0	80	1.1900	1.1830	0.5883
GF2	0	20	80	1.1820	1.1752	0.5753
EMF/GF3	15	5	80	1.1801	1.1751	0.4237
EMF/GF4	10	10	80	1.1728	1.1690	0.3240
EMF/GF5	5	15	80	1.1790	1.1680	0.2482
PPP	0	0	100	1.1650	1.1589	0.1356

Table 1. Percentage weight ratio, theoretical density and experimental density of composites.



Figure 1. (a) Short Entada mannii-fiber, (b) Hybrid composites.

2.4. Density and Void Content

The density of materials is a measure of mass per unit volume and can be used to determine the structural integrity of materials by examining the void contents [23]. However, theoretical and experimental density can be used to determine the composite void percentage presented in Table 1. The percentage void contents in terms of weight percentage can be calculated based on the Soliman et al. [24] equation in accordance with ASTM D2734-94.

$$V_f. = \frac{\rho_{th} - \rho_{ex}}{\rho_{th}} \times 100 . \tag{1}$$

where V_f is the void fraction, ρ_{th} denotes the theoretical density and ρ_{ex} is the experimental density of the composites.

2.5. Moisture Absorption

The moisture absorption behavior of the fabricated composites was calculated in accordance with the ASTM D5229/D5229M-14. Samples of the composites were submerged in distilled water for 10 days. The percentage of moisture absorption M(t) for each sample was calculated according to the equation below [25].

$$M(t) = \frac{w_{t-}w_o}{w_o} \times 100 \tag{2}$$

where w_t is the sample weight after time t and this was carried out for 216 days of immersion.

2.6. Tensile Strength

The composite tensile properties were evaluated based on the ASTM D638 standard on a tensile testing machine. The specimen with a dumbbell shape dimension of $125 \times 20 \times 4$ mm was mounted on the machine under tension until it fractures. The specimen gauge length was carried out with a 5 mm/min strain equipped with a load of 5 kN. The experiment was carried out repeatedly six times and the values were recorded [26].

2.7. Impact Strength

The impact specimens were prepared according to ISO 180 standard using the Izod impact testing machine [27]. The samples were preloaded with a hammerhead of 7.5 J and released to fracture the specimen at a velocity of 2 m/s. The specimen impact strength was dimensioned $55 \times 10 \times 4$ mm and 2 mm deep notch. The influence of strain rate on the fracture and ductility can be measured using the impact test. On average, five samples were analyzed, and the values were recorded.

2.8. The Microhardness of the Composites

The microhardness of the composites was evaluated based on the ASTM E384 standard at 23 °C using the Shimadzu HMV-2 hardness machine equipped with indentation of 0.2 mm radius, and a load of 300 N for 10 s. Ten repeated indentations were carried out and an average value was recorded for the specimen.

2.9. The Compressive Properties of the Composites

The compressive strength and modulus of the samples were prepared according to ASTM D790D standards using three-point bending using a flexural testing machine. The sample was prepared at room temperature of 23 °C and the testing procedure was preloaded at 2 N with a speed of 3 mm/min and humidity of 50%. Six repeated samples were evaluated, and results were recorded for discussion.

2.10. Characterization of the Composite Fracture Surface

The fracture surface analysis of the fabricated composites was carried out using a scanning electron microscope (SEM) [28]. The obtained specimen was put inside a void compartment to dry and protected with 100 A thick iridium at 15 KeV.

3. Results and Discussions

3.1. Effect of Alkali Modification

The influence of chemical modification on the *Entada mannii*-fiber surface and characterization is illustrated in Table 2 and Figure 2, respectively. It was observed that alkali treatment enhanced the removal of lignin, hemicellulose and increased the cellulose contents on the fiber compared to untreated fibers as shown in Table 2. In addition, surface modification using alkaline treatment enhanced the fiber dispersion by fibrillation which effectively increase the hydroxyl groups within the fiber. The removal of the fiber constituents also contributed to the fiber's rough surface, as shown in Figure 2a. Furthermore, the presence of these rough surfaces not only enhances the fiber-matrix interfacial adhesion but also assists with stress distribution and load sharing from the matrix to reinforcement [28–30]. Soliman et al. [25] studied the influence of chemical treatment (NaOH) on a rice straw mat and concluded that the fiber surface was enhanced with the alkaline treatment, which promotes fiber-matrix bonding. Hence, this result validates the increase in tensile and compressive strength of the hybrid composites.

Figure 2 shows the SEM of the treated and the raw fibers. It is obvious that alkaline treatment (NaOH) enhances the removal of these amorphous and semi-crystalline deposits on the fiber surface and exposes the fiber's rough surface while the untreated fiber surface is smooth [30–33]. However, this modification enhanced composites' mechanical interlocking.

Samples	% Lignin	% Ash Content	% Hemicellulose	% Cellulose	Density(g/cm ³)
Treated (NaOH fiber)	6.29	4.35	40.79	61.88	1.228
(Untreated fiber)	8.12	5.81	48.18	51.73	1.541
Glass Fiber	-	-	-	-	2.930

Table 2. The percentage constituents of Entada mannii natural fiber.





Figure 2. Chemical treatment of the fiber: (a) NaOH treated fiber; (b) Untreated fiber.

3.2. The Assessment of Composite Void Contents

Table 1 shows the void percentage fraction determined from theoretical and experimental density with volume fractions of the EMF/GF single and hybrid composites. It was observed that the addition of EMF/GF increases the theoretical and experimental density as the fiber weight increases. Among the hybrid composites, EFM/GF5 exhibited a higher reduction in void fraction and followed by EMF/GF4 and EMF/GF3, respectively, as compared with the single reinforced composites. This improvement supported the uniform distribution of fibers in the matrix. This study further described that voids and pores are relatively evolved during the fabrication process by the compression molding technique. Chaudhary et al. [34] studied the development of a jute/hemp/flax hybrid composite and reported that the fabrication process assists the removal of pores and voids within the composites, which indicated a perfect processing technique was adopted. Similarly, due to the hydrophilic nature of EMF, chemical modification of the fiber surface facilitates the removal of moisture contents, which support the cementing and strengthening of EMF/GF with a matrix interface.

3.3. Assessment of the Moisture Absorption

The water absorption properties of the composites are presented in Figure 3. It is evident that the moisture intake of all the composites increases as the weight increases and happens the least in pure PP. Single reinforced composites (EMF1 and GF2) were observed to exhibit the highest water absorption. This can be ascribed to poor fiber–matrix bonding supported by the presence of the void contents in the composites obtained in Table 1. On the other hand, hybrid composites EMF/GF5 had the lowest water uptake of 50% compared to single reinforced composites. This can be ascribed to the addition of 15 wt% glass fiber (GF) into the polypropylene matrix, which assists in reducing the water absorption intake and enhances fiber–matrix interfacial bonding. Hence the addition of glass fiber into the EMF polypropylene matrix enhances the mechanical properties and also reduces the rate of moisture intake of the hybrid composites. In the Masunder et al. [35] research work, the hybridization of glass fibers and silk fibers with a thermoplastic matrix

decreased the water-resistance of the hybrid composites. Furthermore, this finding is also supported by the work of Kushwaha and Kumar [36], who confirmed that the addition of fiberglass into epoxy and polyester/bamboo hybrid composites reduces the amount of moisture intake of the hybrid composites.



Figure 3. The moistures absorption behavior of the composites.

3.4. Stress–Strain Plot of the Composites

The comparison stress–strain plot is presented in Figure 4. A progressive increase was observed for all the composites as the fiber weight increases up to 20 wt% EFM/GF. However, hybrid composites EMF/GF5 had the highest strength relative to other composites as a result of the presence of higher GF contents in the composites, supported by the efficient fiber loading. On the other hand, composites EMF1 and GF1 exhibited a reduction in tensile properties due to uneven dispersion and fiber pullout in the polypropylene matrix. As a result, single reinforced composites (EMF1 and GF1) became brittle, which led to untimely failure at lower strain values.



Figure 4. Stress-strain curve for the short Entada mannii-glass fiber hybrid composites.

3.5. Evaluation of the Tensile Strength of the Composites

The comparison of tensile properties of the short EMF/GF reinforced composites is presented in Figure 5. It was observed that the tensile strength of all the composites is enhanced with increased fiber contents. Among the fabricated composites, hybrid composites (EMF/GF5) gave the highest tensile strength of 57.3 MPa and increased by 41% compared to single reinforced composites. It is worthy to note that the addition of 5% MAPP compatibilizers contributed to the uniform fiber distribution of the fiber and promote hybrid composite stiffness [37]. However, a similar trend was also observed for the hybrid composites (EMF/GF3 and EMF/GF4), respectively. This improvement can be attributed to efficient load distribution and strong interfacial bonding with the thermoplastic matrix. This confirmed the result of the scanning electron micrograph obtained in Section 3.10, which demonstrates the even dispersion of the fibers in the matrix. Atiqah et al. [38] similarly worked on the incorporation of glass fibers into the sugar palm polyurethane matrix and observed significant improvement in the tensile properties of the hybrid composites. Sapuan et al. [39] reported that the inclusion of fiberglass into the natural fiber (basalt/wool) polyester resin enhanced the tensile properties because the matrix acts as a binder and provides rigidity when a load is applied resulting in tensile strength increase. On average, the hybrid composites (EMF/GF5) gave the best tensile strength. This was achieved due to optimal performance of the fiber-matrix bonding and uniform cementing with the matrix interface [40].





3.6. Young's Modulus of the Reinforced Composites

The comparison graph of Young's modulus of the hybrid EMF/GF fiber-reinforced composites is presented in Figure 6. The addition of EMF/GF significantly increases Young's modulus of the hybrid composites as compared with the single reinforced composites. Moreover, incorporating up to 10 wt% GF into hybrid composites demonstrates an improvement in the modulus of the composite as compared to the single reinforced composite. This suggested that the presence of fiberglass increases the load-bearing capacity of the hybrid composites and provides an effective transfer of stress to the matrix. On average, the EMF/GF5 gave the highest Young's modulus of 53.2 GPa and increased by 33% relative to the single reinforced composites. This can also be linked to efficient interaction between EMF/GF and uniform distribution of the stress, which increases the stiffness of the composite in the linear elastic region [33]. Similar works performed by



Haque et al. [41] concluded that the addition of fiberglass into betel nut hybrid composites contributed to the increasing modulus of developed composites.

Figure 6. Young's modulus of the hybrid reinforced composites.

3.7. Compressive Strength and Modulus Fabricated Composites

The evaluation of the compressive strength of EMF/GF polypropylene composites is presented in Figure 7. It is evident that the compressive strengths of all the composites gradually increase as the fiber loading increases. The hybrid composites were observed to increase with the addition of varying weight percent of EMF/GF in the polypropylene matrix relative to a single reinforced composite. This can be ascribed to the alkaline treatment of the *Entada mannii*-fiber, which reduces the fiber cell wall thickness and promotes good wetting with the matrix. A similar study was investigated by Atiqa et al. [42] on the effect of alkaline treatment on sugar palm fiber surface roughness, which enhances the even distribution of fibers and increases the compressive strength of the hybrid composites. The ultimate compressive strength obtained from the hybrid composites EMF/GF5 was 86.8 MPa and increased by 62%, followed by EMF/GF3 of 48% and EMF/GF4 of 56%, respectively, relative to single reinforced composites. On average, the tensile strength of the hybrid composite EMF/GF5 exhibits better mechanical interlocking with matrix interfaces and reduces composite shear failure [43].

Figure 8 shows the compressive modulus of the EMF/GF reinforced composites. A progressive increase in compressive modulus was observed for all the composites produced as the fiber loading increased. On average, the compressive modulus of both hybrid composites EMF/GF4 and EMF/GF5 were 27% and 34% higher than the single reinforced composites. This indicated that the presence of GF enhanced the compressive modulus of the hybrid composites because of the higher modulus of the fiberglass compared with *Entada mannii* natural fiber. This trend was also observed for the EMF/GF3 hybrid composites with a flexural strength of 49.9 MPa, 32% higher than single reinforced composites. Sanjay and Yogesha [44] reported that hybridization of natural fibers with fiberglass increases the strength and modulus of the composites as the weight increases.



Figure 7. Compressive strength of the reinforced composites and pure PP.



Figure 8. The compressive modulus of the composites.

3.8. Comparison of the Impact Strength

The impact strength of the EMF/GF fiber-reinforced composites is presented in Figure 9. Basically, the impact properties of all composites were observed to increase as the fiber weight is increased. The impact strength increase can be attributed to the presence of EMF/GF compared to pure PP and single reinforced composites. Among the hybrid composites produced, composites EMF/GF4 had the highest impact absorbing the energy of 43 kJ/m⁻², followed by EMF/GF5 of 35 kJ/m⁻² and least was EMF/GF3 38 kJ/m⁻², respectively. This can be ascribed to the inclusion of 10% wt glass fiber, which supports the resistant crack propagation when the load is transferred from the matrix–fiber reinforcement. Additionally, it is observed that the incorporation of glass fibers into the polypropylene matrix enhanced the impact-absorbing capacity attributed to glass fiber-reinforced composites increased due to higher fiberglass loading that helped to attain uniform distribution of the banana fiber in the matrix interface.



Figure 9. Impact strength of single and hybrid composites.

3.9. Micro Hardness Properties

Figure 10 shows the microhardness of the single and hybrid composites. The hardness value of pure PP and EMF/GF5 was observed to increase from 34 Hv to 70 Hv with the *Entada mannii*/fiberglass loading. It was observed that the obtained microhardness of the hybrid composites EMF/GF5 gave the optimum hardness value and increased by 60% compared to single reinforced composites. This progressive increase is largely attributed to the presence of minimal void content present in composites. Furthermore, incorporating 15 wt% glass fibers into the matrix increases the hardness value because glass fibers possess higher hardness that supports the strengthening of the EMF with the polymer matrix. Overall, EMF/GF5 composite exhibits the highest hardness value due to reduced matrix flexibility and thus increases the rigidity of the composite. Tripathy et al. [46] reported that hybridized low-cost jute/fiberglass possess a higher hardness value and better adhesion due to increased fiberglass loading in the matrix.



Figure 10. Microhardness of the single and hybrid composites.

3.10. Scanning Electron Micrograph of the Fracture

Figure 11 reveals the morphological characterization of single and hybrid reinforced composites. From Figure 11a,b, the EMF1 and GF2 single reinforced composites were characterized with fiber pullout and matrix yielding as a result of poor loading carrying capacity under tensile loading conditions. Furthermore, void formation and matrix yielding were present due to the poor distribution of the EMF/GF supported by the weak mechanical bonding between the reinforcements and the matrix interface [47]. The presence of hydrophilic *Entada mannii*-fibers tends to absorb higher moisture, which could be linked to uneven fiber–matrix mixing and fiber pullout from the polymer matrix.



Figure 11. Morphological characterization of single and hybrid reinforced composites: (a) EMF1; (b) GF2; (c) EMF/GF3; (d) EMF/GF4; (e) EMF/GF5.

Figure 11c shows a relatively uniform distribution of the 10 wt% EMF/GF in the polymer composites supported by the increased tensile strength of the EMF/GF4 composites compared to the counterparts. Hence the addition of EMF/GF enhanced the tensile properties and compressive of the produced composites.

From Figure 11d, it is evident that less fiber pullout and debonding occur in the composites EMF/GF5, which clearly show a uniform distribution of fibers with the matrix interface. Fewer void contents and fractures were observed in EMF/GF4/and EMF/GF5 hybrid composites attributed to superior mechanical interlocking of the hybrid composites. This improvement confirmed the superior tensile properties compared with other counterparts presented in Figure 5.

4. Conclusions

This research evaluates the physical and mechanical properties of short *Entada mannii*glass fiber hybrid composites produced using the compression molding technique. The results obtained are as follows.

Surface modification of the *Entada mannii*-fiber using NaOH effectively removed the lignin, hemicellulose and increased the hydroxyl group within the fiber surface for better fiber to matrix bonding. On average, the hybrid composites EMF/GF5 demonstrated an improvement in the tensile strength and compressive strength with the addition of 15 wt% fiberglass and promoted good compatibility between the fibers and the matrix. The result of the Young's and compressive modulus show that the hybrid composites possess good fiber–matrix interactions as a result of the uniform distribution of the stress in the linear elastic region. The incorporation of glass fibers enhanced the impact-absorbing capacity of the hybrid composites due to the higher tensile properties while it exhibited the highest hardness value due to the increased rigidity of the hybrid composites. The surface morphology of the single reinforced composites (EMF1, GF2) was characterized with fiber pullout and matrix yielding as a result of poor loading carrying capacity under tensile loading conditions. Overall, the hybridization of *Entada mannii*-fiber and fiberglass composites enhanced the mechanical and physical properties of hybrid composites and this can be used in automotive applications.

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