EFFECT OF FIBER LOADINGS ON MECHANICAL PROPERTIES OF WATER HYACINTH FIBER REINFORCED POLYPROPYLENE COMPOSITES


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ABSTRACT
In this work, water hyacinth (WH) fiber reinforced polypropylene (PP) composites were fabricated using a single screw extruder and an injection molding machine. Raw WH fibers were chemically treated, first by benzene diazonium-chloride, then by sodium hydroxide in order to increase the compatibility of the WH fibers with the PP matrix. Different fiber loadings (15, 20, 25, 30, and 35 wt %) were considered for both the raw and treated WH fibers in manufacturing the composites. ASTM standards were followed to carry out experiment in order to evaluate various properties of composites. Scanning electronic microscopic (SEM) images were analyzed to understand the interfacial bonding between WH fibers and PP matrix for both the raw and chemically treated fibers. It was found that all the mechanical properties, except tensile strength, were satisfactorily improved for chemically treated WH-PP composites in comparison with those of raw WH-PP composites.

Keywords: Natural Fibers, PP, Biodegradable Composites, Chemical Treatment, Fiber Loadings.

1. INTRODUCTION
Over the past several years, ecological concerns have resulted in a renewed interest in natural, renewable resources-based and compostable materials, and therefore, issues such as materials elimination and environmental safety are becoming important. For these reasons, material components such as natural fibers (biodegradable polymers) can be considered as interesting – environmentally safe – alternatives for the development of new biodegradable composites (biocomposites) [1]. The interest in using natural fibers as reinforcement in plastic materials has increased dramatically. Also, high cost of synthetic fillers has urged the researchers to use natural fibers as organic fillers. Natural fibers have many advantages compared to the synthetic fibers. For example, natural fibers have low density in addition to their recyclability and biodegradability. Additionally, they are renewable material and have relatively high strength and stiffness [2, 5]. Combination of low density and good mechanical properties of natural fibers has produced a composite which is suitable for structural applications. Recently, a number of results have been reported for thermoplastic composites. Many different types of organic fillers can be added into polypropylene (PP), polyethylene, and other thermoplastic polymers. Several different natural organic fillers such as wood fibers and flour, kenaf fibers, sago, rice starch, cornstarch, henequen fibers, and pineapple-leaf fibers, have been used as fillers in polymer matrices [2–6].

Renner et al. [2] investigated the micromechanical deformations in PP-lignocellulosic filler composites and the effect of matrix characteristics on deformation and failure and reported that the dominating micromechanical deformation process changes with matrix properties. Bouza et al. [3] investigated the effect of particle size and a processing aid on the crystallization and melting behavior of isotactic polypropylene (iPP) with red pine wood flour under dynamic and isothermal conditions in the presence of a processing aid with varying filler particle sizes. Reddy et al. [4] investigated the polypropylene hybrid composite consisting of wheat straw and clay as reinforcement materials. They reported that the increase in wheat straw and clay content in a composite increases the flexural modulus and reduces the resistance for water absorption. The increase in PP-MA coupling agent also increased the flexural modulus and resistance for water absorption, and the addition of clay
as additional filler had no significant role on water absorption and flexural properties of the composite. Haque et al. [6] investigated the physical and mechanical properties of raw and treated coir fiber reinforced polypropylene composites of fiber loading 15, 20, 25, 30 and 35 wt%. They found that chemically treated coir fiber reinforced specimens yielded better mechanical properties compared to the raw ones.

Although many researchers have carried out experiment with different organic filler polymer composites, there are no reports on the research on fabrication and mechanical characterization of water hyacinth (WH) reinforced PP composites. The water hyacinth (Eichhornia crassipes) is a true water plant and floats by means of spongy petioles. Of all the aquatic plants, the water-hyacinth is the most prolific and spectacular [7, 8]. Water hyacinth can cause a variety of problems when its rapid mat-like proliferation covers large areas of fresh water. It can also cause practical problems for marine transportation, fishing, hydropower generation and irrigation. Water hyacinth has a high content of cellulose, which may be useful as filler in polymeric composites because the cellulose of bio-fiber is responsible for most of the physical and chemical properties such as biodegradability, flammability, sensitivity towards moisture, thermo-plasticity, degradability by UV-light, etc [9]. In this study, WH fibers were chosen as filler materials for PP matrix in order to investigate the mechanical and physical properties of WH-PP composites. WH fibers were collected from both the stem and leaf of WH.

2. MATERIALS

The thermoplastic polypropylene (PP) of injection molding grade, used as matrix material, was supplied by Linyi Aosen Chemical Co. Ltd., Shandong, China, in the form of homopolymer pellets. It had melt flow rate of 8 g/min, specific gravity of 0.90–0.91, and crystallinity of 82% [10]. The melting point of this PP was measured and found to be 165°C. The Water hyacinth fibers (WHFs), used as reinforcing materials, were obtained from fresh water hyacinths of a local river and Dhanmondi lake, Dhaka, Bangladesh. Stems of the fresh water hyacinth were cleaned and cut into small pieces. Then they were dried in sunlight. The dried water hyacinth was then ground into powder, and labeled as water hyacinth fibers. Fibers that were smaller than 150 μm were selected by sieving analysis. Chemicals used in this study to treat WHFs were Hydrochloric acid (Merck, Germany), Sodium nitrite (Merck, Germany), Aniline (Merck, Germany), and Sodium hydroxide (Merck, India).

2.1 Chemical Treatment of Water Hyacinth Fibers

Benzene Diazonium Chloride was synthesized according to standard method from the reaction of Aniline (dissolved in hydrochloric acid) and Sodium nitrite diazotization method [11]. 200 gm water hyacinth fibers were submerged into the prepared solutions at about 5°C in an ice bath with constant stirring for 10 minutes. In order to neutralize the acidic diazonium chloride, 70ml of 5% NaOH was mixed in a beaker separately and then poured slowly into the above mixture for 15 minutes at about 5°C in an ice bath. Water hyacinth fiber was then taken out, washed with distilled water, and finally dried in open air.

3. FABRICATION OF COMPOSITES

Both raw and treated WHFs (15, 20, 25, 30, and 35wt%) was initially mixed thoroughly with PP granules. The mixtures were passed through a single screw extruder at a constant temperature of 135°C ±5°C. The extruder gave the melt-mixed WH-PP in the form of rods of small diameters. The rods were then cut into small pieces and called granules. The granules were dried in a vacuum oven at 65°C for 1 hr. The dried granulated products were molded into specimens (tensile, flexural, and Charpy impact test) as per ASTM standard by an injection-molding machine at a molding temperature of 165°C. The pressure, temperature and heating time of the samples were controlled at the same rate to prepare all composites. The injection-molding machine was vertical type and operated manually.

4. EXPERIMENT

4.1 Scanning Electron Microscopy (SEM)

SEM was employed to study the tensile and flexural fracture surfaces of composite samples of 35 wt% fiber content. They were examined using a JSM-5510 SEM analyzer from JEOL Co. Ltd., Japan. This test was carried out to determine the dispersion of fibers in the matrix, adhesion between fibers and matrix, and to detect the presence of any micro defects in the composite.

4.2 Mechanical Properties

In order to investigate the mechanical properties of the composites, the following tests were carried out; (a) tensile test, (b) three point flexural bending test, and (c) Charpy impact test. For these tests, appropriate ASTM standards were followed.

4.2.1 Tensile Test

The static tensile test of the composites were carried out in an universal tensile testing machine, model: Hounsfield UTM (H10KS), capacity: 10KN, Ogawa Seiki C. Ltd., Japan, at a cross head speed of 5 mm/min. Tensile tests were conducted following ASTM D 638-01standard [12] and each test was performed until tensile failure was occurred. Dumbbell-shaped injection molding composite specimens were used as test specimens without having any kind of machining. The dimension of the specimens used was 148 mm × 10 mm × 4.1 mm.

4.2.2 Flexural Test

The static flexural tests of the composite specimens were carried out by same machine that was used for
tensile test only by changing the attachment. Dimension of flexural test specimen was 100 mm length, 10 mm width, and 4.1 mm thickness. Flexural tests were conducted following ASTM D 790-00 standard [13]. The flexural strength and modulus were calculated using the following equations:

\[
\sigma_f = \frac{3PS}{2bd^2} \quad (1)
\]

Flexural modulus, \( E_b = \frac{S^3m}{4bd^3} \quad (2) \)

where \( P \) is the maximum applied load, \( S \) is the length of support span, \( m \) is the slope of the tangent, \( b \) and \( d \) are the width and thickness of the specimen, respectively.

4.2.3 Charpy Impact Test

Dynamic Charpy impact tests were conducted according to ASTM D 6110-97 standard [14] using a Universal Impact Testing Machine (Type: TIT-30, Tokyo Testing Machine MFG. Co. Ltd., Japan). Notched composite specimens were used during the experiment. The dimension of the specimen used was 79 mm \( \times \) 10 mm \( \times \) 4.1 mm.

4.3 Water Absorption Test

In order to evaluate the water absorption property of the composites, rectangular shaped test specimens were cut from each category of samples having dimensions of 39 mm \( \times \) 10 mm \( \times \) 4.1 mm. The samples were dried in an oven at 105\(^\circ\)C for 2 hrs, cooled in a desiccator using silica gel and immediately weighed to the accuracy of \( \pm 0.0001 \) gm using Denver Instron balance. The dried and weighed samples were immersed in distilled water for about 24 hrs at room temperature as described in ASTM D570-99 standard [15]. The samples were taken out from water and excess water on the surface of the samples was removed by using a soft cloth. Then the weight of the samples was taken again. Three replicate specimens were tested and the average results of the three tests were presented. The percentage increase in weight after immersion was calculated as follows [15]:

\[
\text{Increase in weight} = \frac{(W_f - W_i)}{W_i} \times 100 \% \quad (3)
\]

where \( W_f \) = weight after 24 hrs immersion in distilled water and \( W_i \) = initial weight before immersion.

5. RESULTS AND DISCUSSION

5.1 Tensile Properties

The importance of natural fiber reinforced composites of polymeric materials comes from the substantial improvement of strength and modulus that offers a possibility of composites in practical applications. The tensile strengths of the raw and chemically treated WH fiber reinforced PP composites at different fiber loadings are shown in Fig. 1. The tensile strengths decreased with the increase of WH fiber loading. The same trend was observed for other NF-PP composites [2-8] in the past.

In order to increase the compatibility of the WH fiber with PP matrix, raw WH fibers were chemically treated with benzene diazonium salt; the tensile strength was found to be higher than that of the raw WH-PP composites with fiber loading up to 20 wt%.

In general, for both the raw and treated fibers, the tensile strength decreases with the fiber loading. This is because the addition of rigid WH particles into the soft PP makes the composite brittle. As a material become more and more brittle, its strength decreases and stiffness increases. The increasing trend of stiffness with the addition of WH particles can be observed from Fig. 2.

![Fig 1. Tensile strength as a function of WH fiber loading.](image1.png)

![Fig 2. Variation of the Young’s modulus as a function of WH fiber loading.](image2.png)
Due to the chemical treatment of WH fiber, fiber-matrix adhesion was improved. Consequently, the flexural strengths were improved for treated WH-PP composites. The highest value of flexural strength was observed for 35 wt% treated WH-PP composite, which was 40.8 MPa and much higher than that of the untreated WH-PP composites.

From Fig. 4, it is observed that the flexural modulus increases with fiber loading in accordance with other studies in literatures [2-8]. In general, the flexural modulus of the treated WH-PP composites exhibits better characteristics in comparison with that of raw WH-PP composites over the entire range of fiber loading. The flexural modulus of benzene diazonium-chloride (by using aniline)/sodium hydroxide treated WH-PP composites is found to be the maximum (2.5 GPa) for 15 wt% fiber loading compared to that of the untreated one (1.93 GPa) for 15 wt% fiber loading. This corresponds to 22.8% increase in the flexural modulus, which is attributed to the good interfacial bonding.

5.3 Charpy Impact Strength

The notched Charpy impact strength of WH-PP composites with respect to fiber loading is presented in the Fig. 5. The Charpy impact test is a standardized high strain-rate test which determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of a given material’s toughness and acts as a tool to study brittle-ductile transition. It is observed from Fig. 5 that the notched Charpy impact strength shows similar trend as flexural strength properties, i.e. impact properties increase with fiber loading in accordance with other similar studies in the literatures [2-8].

The highest value of impact strength is observed for 35 wt% of fiber loading corresponding to benzene diazonium-chloride (using Anl)/sodium hydroxide treated WH-PP composites. One of the factors of impact failure of composite is fiber pull out. With increase in the fiber loading, bigger force is required to pull out the fibers. This consequently increased the impact strength. The maximum value of impact strength of the treated WH-PP composites is found to be 67.395 KJ/m² which is greater than that of the untreated WH-PP composites with maximum value of 52.626 KJ/m². This corresponds to 21.98% increase in the impact strength.

5.4 Water Absorption Characteristics

Water absorption characteristics of the WH-PP composites against fiber loading are shown in Fig. 6. In general, water absorption (%) increases with fiber loading [2, 7] and this trend is observed in the present case too. Also, it is observed that chemically treated WH fiber reinforced PP composites have higher water content compared to the raw WH-PP composites after 24 hrs immersion in distilled water at room temperature. Thus, although the chemical treatment increased the mechanical properties of the composites, it was unable to decrease the water absorption of the composites.
Fig 6. Variation of water absorption as a function of fiber loading.

5.5 SEM Analysis
The SEM morphology of the fracture surfaces of tensile and flexural test samples shows the phase information reflecting the reasons why the mechanical properties of the composites fabricated under different conditions of the fibers are different. The SEM images of WH-PP composites are shown in Fig. 7 for the 35 wt% fiber loading of raw and chemically treated WH-PP composites.

Figures 7 (a) and 7 (b) illustrate the SEM micrographs of fracture surface of tension and flexural specimens (raw WH fibers with 35 wt% fiber loading), respectively. It is observed from both the figures that the fiber surfaces have very clear boundary or interface indicating no strong bonding between WH fibers and PP matrix. Also, there are some black holes which imply the fiber pull-out.

Figures 7 (c) and 7 (d) illustrate the SEM micrographs of composites of treated WH fibers with 35 wt% fiber loading. Fiber pullout and de-bonding reduced significantly. This improved the adhesion between fiber and matrix, which can be understood from the fact that there is no sharp interface between WH fibers and PP matrix. This happens as a result of the formation of ester linkages between PP and OH groups of cellulose.

6. CONCLUSIONS
In this study, WH-PP composites were fabricated by using a single screw extruder and an injection molding machine. Both raw and treated WH fibers of different fiber loadings were used to manufacture the composites.
From the analysis of the results, following salient conclusions can be made:

1. The tensile strength of the composites decreased with an increase of the WH fiber loading. However, the 15wt% treated WH fibers reinforced PP composite exhibited better tensile properties than corresponding raw WH-PP composites.

2. The Young’s modulus, flexural strength, flexural modulus, and Charpy impact strength of the WH-PP composites increased with an increase in the fiber loading. However, in general, all of these properties were much better for treated WH-PP composites in comparison with raw WH-PP composites.

3. The chemical treatments of WH fibers increased the interfacial bonding between the fibers and matrix. For this reason, above properties of composites were improved due to chemical treatments. However, water absorption property did not improve because of chemical treatments.

7. REFERENCES


8. MAILING ADDRESS

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