Mechanical, Morphological and Thermal Properties of Alkali Treated Ladies Finger Fiber Reinforced Polypropylene Composites

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Abstract: Fiber reinforced polymer composites have played a dominant role for a long time in a variety of applications for their high specific strength and modulus. The fiber which serves as a reinforcement in reinforced plastics may be synthetic or natural. Although glass and other synthetic fiber-reinforced plastics possess high specific strength, their fields of application are very limited because of their inherent higher cost of production. Present research investigated the effect of alkali treatment and loading of ladies fiber on mechanical and thermal properties of ladies finger fiber reinforced polypropylene composites. Composites were prepared using compression molding technique. Ladies finger fiber loading were varied at 5, 10 and 15 wt%. Both raw and alkali treated ladies finger fiber were utilized to reinforce polypropylene. Mechanical (tensile, flexural and hardness) and water absorption tests and thermogravimetric and scanning electron microscopic analysis of the prepared composites were subsequently carried out. Tensile and flexural strength decreased with fiber loading, while opposite trend was observed in case of Young's modulus, flexural modulus, hardness and water absorption. Alkali treatment of ladies finger enhanced mechanical properties of prepared composites due to better adhesion between the fiber and matrix in treated fiber composites. According to thermogravimetric analysis, thermal stability of composites increased with fiber loading.

Keywords: Alkali Treatment; Ladies Fiber-Polypropylene Composite; Mechanical Properties; TGA; SEM

1. Introduction

Replacement of the conventional synthetic fibers with natural fibers as reinforcement in polymer matrix composites could lead to a green, renewable path of applications. Natural fibers have received great attention from researchers and industrialists due to their biodegradability, better mechanical properties, easy manufacturing, and overall cost effective quality [1–3]. While most of the synthetic fibers are costly and might cause skin irritation, natural fibers are completely free from these problems. Natural fiber reinforced polymer composites has increased noticeably since last few decades. Fiber reinforced composites are presently used in automotive application, aerospace parts, naval architecture and membrane as they are less corrosive, less abrasive, and have good mechanical properties. Hot pressing, fiber extrusion and hand lay-up methods are the most common composite manufacturing techniques used in preparing natural fiber reinforced polymer composites [4–6]. However, with all these advantages and promise ahead, the biggest obstacle that is yet to overcome is assuring uniformity. As nearly all natural
fibers are collected as husk or skin, assuring uniformity in surface condition, strength and other properties are in question. To ensure homogeneity in properties, natural fibers are required to undergo some treatments varying from chemical to physical. In many cases, natural fibers are chemically treated before composites manufacturing in order to increase the interfacial adhesion between the fiber and polymer, which in turn increases the mechanical properties [7-9]. Current day’s polymers of two kinds are widely used: thermoplastic and thermosetting. Polypropylene (PP), polyethylene (PE) and polyvinyl chloride (PVC) are three of the most popular types of thermoplastic materials, while epoxy, polyester, phenolic dominates the thermosetting domain. To be used as matrices for bio-fibers, the thermoplastic materials are commonly used. In present research raw and alkali treated ladies finger fiber reinforced polypropylene composites were prepared by varying fiber loading. Prepared composites were subsequently characterized using mechanical tests and thermogravimetric analysis.

2. Materials and Methods

2.1 Materials
The matrix materials PP were collected in pellet form from Polyolefin Company Private Limited, Singapore. The reinforcing agent (ladies finger fiber) was collected from a village of Bangladesh. Sodium hydroxide (NaOH) was used during chemical treatment ladies finger of fiber. It was purchased from Merck, Germany.

2.2 Alkali Treatment of Ladies Finger Fiber
Ladies finger fiber solution treated in 5% NaOH solution. 5% NaOH solution was prepared by adding 5 gm NaOH to 100 ml water in a beaker. Fiber was then treated in 5% NaOH solution at 70°C for two and half hours. The fiber was washed and dried in an oven at 80°C for 20 minutes.

2.3 Composite Preparation
Firstly reinforced composites of polypropylene matrix and varying amount of (5%, 10% and 15%) were manufactured using hot press technique. Initially fibers were weighted according to the required weight fraction. Then both fibers were cut into 3-5 mm size in length. In order to allow the removal of moisture, fibers were dried in an oven at 100°C for 20 minutes before preparing each composite. The required amount of polypropylene was weighted. Mold surface was cleaned very carefully and mold releasing agent (silicon spray) was sprayed over the mould surface properly for the easy removal of the product. Then the fibers and polypropylene were mixed thoroughly on to the lower die. The lower die was covered by the upper die according to the indication provided in the both mold. The die was placed in a hot press machine. In present research a hydraulic type machine having capacity of maximum load of 50 kN and maximum temperature of 300°C was used. The fiber-matrix mixture was allowed to press at 30 kN pressure. The temperature was initially raised to 160°C and hold there for around 20-25 minutes, after that the temperature was raised to (190-195)°C depending on the thickness required. The die was cooled to room temperature, pressure was released and the specimen was carefully withdrawn from the die.

2.4 Characterization Techniques
To characterize the properties of ladies finger fiber reinforced PP composites mechanical, water absorption and thermal analysis were performed. The mechanical analysis involved tensile, flexural and hardness tests. During tensile test, a universal testing machine (Model MSC-5/500, Agawn Seiki Co., Tokyo, Japan) was used along with ASTM D 638-10 method maintaining 10 mm/min crosshead speed. The same universal testing machine was used for the flexural test with same crosshead speed where the method used was ASTM D 790-10. The experimental work involved a Shore hardness tester for measurement of hardness of various composites. Following ASTM D 570- 98, water absorption tests was performed. Rectangular specimens were immersed in water for 24 hours. The weight of the specimen was measured before and after the test. Finally the difference between final and initial weight of the specimen
was calculated to observe the water uptake capacity of composites. Surface morphology of prepared composites was evaluated using scanning electron microscopic analysis. Surface of the composites were made conductive by giving platinum coating using a sputtering machine. Composite surfaces were then observed in vacuum condition into the SEM machine. TGA method used was based on continuous measurement of weight during increase of sample temperature in an inert atmosphere. Data were recorded as a thermogram of weight versus temperature.

3. Results and Discussion
3.1 Tensile Properties
Figure 1 (a) shows the variation of tensile strength with fiber loading. The figure shows that tensile strength gradually decreased with increase in fiber loading. As fiber content increases, the weak interfacial area between the fiber and matrix increases, which decreases tensile strength [10]. It can be noticed that the tensile strength of chemically treated fiber reinforced composite was higher than those of raw fiber reinforced composite. The increased tensile strength of chemically treated composite was due to stronger adhesion between treated fiber and matrix [10]. The range of tensile strength of prepared composites was 22-35 MPa, while the same of ladies finger fiber reinforced polymer composite found elsewhere was 23-69 MPa [11].

Figure 1. Variation of (a) tensile strength and (b) Young's modulus against fiber loading.

Figure 1 (b) shows the variation of Young's modulus against fiber loading. Young's modulus increased with increase fiber loading. During tensile loading, partially separated microspaces are created, which obstructs stress propagation between the fiber and the matrix. As the fiber loading increases, the degree of obstruction increases, which in turn increases the stiffness [12]. The Young’s modulus of composites made of treated ladies finger fiber reinforced composites was higher than those of untreated ladies finger composite due to the change in the structure of the cellulose unit of ladies finger. The chemical treatment of ladies finger fiber reduced the hydroxyl group of the cellulose unit by coupling with basic sodium sulfate salt [10]. The range of Young's modulus of prepared composites was 520-780 MPa, while the same of ladies finger fiber reinforced polymer composite found elsewhere was 507-1025 MPa [11].
3.2 Flexural Properties
Variation of flexural strength and flexural modulus of ladies finger fiber reinforced polypropylene composite against fiber loading are shown in Figure 2. The flexural strength decreased with increase fiber loading (Figure 2 (a)). As fiber loading increased, the interfacial area between the fiber and matrix increased, which was weak because of worsening interfacial bonding between cellulose based hydrophilic filler (ladies finger) and hydrophobic PP matrix. This consequently decreased the flexural strength [12]. The range of flexural strength of prepared composites was 20-26 MPa, while the same of ladies finger fiber reinforced polymer composite found elsewhere was 39-118 MPa [11]. According to Figure 2 (b), flexural modulus increased with fiber loading. Since ladies finger is high modulus material, higher fiber concentration demands higher stress for the same deformation. So the incorporation of the filler (ladies finger) into the soft polypropylene matrix results into the increase in modulus [10]. The range of flexural modulus of prepared composites was 1200-1700 MPa, while the same of ladies finger fiber reinforced polymer composite found elsewhere was 3370-10135 MPa [11].

3.3 Hardness Properties
Variation of hardness of prepared composites is shown in Figure 3 (a). Hardness of ladies finger fiber reinforced PP composites increased with increase in fiber loading. Due to the increase of stiffness of respective composite, hardness of PP composites showed a slight increasing trend with an increase in the fiber content [10].

3.4 Water Absorption Characteristics
Natural fiber has a tendency to absorb water due to the presence of hydrophilic hydroxyl groups of cellulose, hemicelluloses and lignin. The experimented data shown in Figure 3 (b). It can be seen that with increase in fiber loading, the water absorption increased. It is also observed that the water absorption tendency decreased after chemical treatment due decrease of hydroxyl groups in ladies finger fiber with alkali treatment [10].

Figure 2. Variation of (a) flexural strength and (b) flexural modulus against fiber loading.
Figure 3. Variation of (a) hardness and (b) water absorption against fiber loading.

3.5 Surface Morphology
In order to study surface morphology and interfacial adhesion between the ladies finger fiber and PP of prepared composites, SEM images were taken for different fiber loaded composites. SEM micrographs of composites with raw and treated fiber are shown in Figure 4. 15 wt% fiber loaded composite showed better adhesion as compared to 5% and 10% fiber loaded composites. Very good wetting between fiber and matrix is also observed in both raw and treated 15% fiber loaded composites [13]. Alkali treated ladies fiber reinforced composites showed better adhesion between ladies finger fiber and polypropylene as compared to raw fiber reinforced composites [12].
3.6 TGA Analysis Results

TGA analysis was performed for both raw and treated ladies finger fiber PP composites to observe the thermal stability of those composites (Figure 5). Composite specimens showed thermal stability at the range of 250–375°C. Stability of composites increased with increase in fiber content. No significant difference was observed between the raw and treated ladies finger fiber composites. From the DTG curves of 5% fiber composite, the sharp peak is found at about 335°C. For 10% fiber composite, two sharp peaks are found at about 325°C and 350°C. For 15% fiber composite, two sharp peaks are found at about 350°C and 375°C. On the other hand, for 15% treated fiber composite a sharp peak is found at about 350°C.

Figure 4. SEM micrographs of (a) 5% raw, (b) 10% raw, (c) 15% raw and (d) 15% treated ladies finger fiber reinforced PP composites.
4. Conclusion
Present research investigated mechanical and thermal properties of ladies finger reinforced polypropylene composite. The fiber loading was varied at 5, 10 and 15 wt% during composite preparation. Tensile and flexural strength decreased with fiber loading, while opposite trend was observed in case of Young’s modulus, flexural modulus, hardness and water absorption. Chemical treatment increased the adhesion between ladies finger fiber and polypropylene matrix. Thus, chemically treated ladies fiber reinforced polymer composites had better properties as compared to raw ladies finger fiber reinforced polymer composites. Thermal stability of composites increased with fiber loading, while no significant difference was observed between raw and treated fiber reinforced composites.
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