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A Study of Fabrication Technique, Structural and Morphological Behavior of Polypropylene Reinforced with Short Natural Fiber Banana

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ABSTRACT

Fiber reinforced polypropylene has been widely accepted as material for structural and engineering applications in recent years. Jute, Banana fibers etc. are the most common low cost, versatile, renewable and abundantly available natural fibers which have biodegradable properties. All these fibers are versatile, renewable and most common agro based fibers that have enormous aspect due to their potentiality in composite manufacture. In comparison to other artificial fibers there are many advantages of natural fibers due to everyday applications such as, paperweight, suitcases, lampshades, helmets, and shower and bath units.

Untreated and alkali treated banana fiber reinforced with Polypropylene matrix composite were fabricated with 10-25% loading of fiber by weight and were fabricated as Polypropylene Banana Composite (PPBC). Using melt mixing hot press molding technique these biodegradable composites were prepared. Different characteristics like morphologies and micro structural analysis of the composites were studied by Scanning electron microscope (SEM) and infrared spectroscopy instrument (IR). Due to the concept of group vibration infrared spectroscopy has the extensive application. Any kind of structural change such as addition or substitution of groups or atoms in a molecule may affect the relative mode of vibration of the group. This causes change in IR spectral band position, change in relative intensities and appearance of new bands and disappearance of any band and splitting of a single band into two or more bands. To increase the utility of fiber infrared spectroscopy can also be used. It deals with the interaction of infrared light with matter. The former can indicate the presence of functional groups qualitatively and the latter can provide a semi quantitative measure of their concentrations. On the other hand Electron Microscopy is most widely used to obtain information regarding the morphology of fiber surfaces, especially SEM (Scanning Electron Microscopy). Using SEM, it is easy to determine the differences of fiber surface topography after and before treatment, and hence the formation of fiber polymer composites. Fiber deboning was also observed for untreated and treated fiber pp matrix composite. The SEM can have a magnification range from a few times to several hundred thousand times.

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1. Introduction

Both thermoplastic and thermosetting polymer matrix reinforced with annually renewable resources bio-fibers provides various bio-degradable aspects. This is because of environment benefits with respect to ultimate disposability and raw material utilization [1]. Hot-press molding process has emerged as a very promising technique in the past few years for enhanced structural property as well as synthesis of PP-B composite. The study of the structural and other physical properties like morphologies of polymer composites reinforced with natural fiber is useful as an analytical technique due to their promising interfacial, photonic and electronic properties. Currently there are many types of artificial reinforcing fibers are used in composite preparation. In many high volume applications composite fabrication technique is carried out by low cost glass fibers reinforcing with polymer matrix. Due to some negative mechanical properties like relatively low elastic modulus and less abrasion resistance glass fibers decrease its potential strength [1]. Due to this disadvantage natural fibers could be used instead of glass fiber as reinforcing agent in the composite for some physical, mechanical and structural applications. There are many advantages of natural fibers compared to other synthetic fibers, for example they have low density, recyclable, low cost, renewable and biodegradable. These are also used for covers of electrical appliances, pipes, post-boxes, roof tiles, grains storage silos, panels for partition and false ceilings, gas containers and in the construction of low cost mobile or pre-fabricated building materials which can be used during natural calamities [2]. There is, however, a major drawback with the application of natural fibers for reinforcement of PP matrix. The moisture uptake in natural fiber is high (approx. 12.5% at 65% relative humidity at 20°C) by dry fiber due to the presence of hydroxyl and other polar groups in various constituents of natural fiber. The natural fiber is biodegradable, abundantly available, easily decomposable in the environment and eco-friendly. Thermoplastic and thermosetting matrix composites derives from natural fibers provide benefits with respect to ultimate disposability and utilization of raw materials. Proper selection of the type, amount, and orientation of fiber is very important, since it influences the following characteristics of a composite. Cellulose-based thermoplastics, aliphatic polyester and biodegradable plastics etc. have attracted much attention in recent years with view of environmental protection [2-3]. Due to highly hydrophilic in nature banana fiber composite is used in industrial and household purposes particularly the electronics, aerospace, and automotive industries.

Research on biodegradable thermoplastics reinforced with the fibers was done by a group of researcher. These reports that higher moisture absorption property is responsible for presence of hydroxyl and polar groups in banana fiber [4]. This causes the poor binding of PP with fiber. Due to better adhesion of polymer matrix composites it is necessary to decrease the hydrophobicity of the fibers by different chemical treatments like bleaching, alkali treatment. This alkali treatment or bleaching improves inter molecular bond strength as well as mechanical strength. This statement carried out to decrease fiber hydrophilicity [5-6] by sodium chloride bleaching. Rao et.al [7] studied the moisture-absorption characteristics of jute composites polyester and epoxy resin systems. In this report it has been carried out that enhancement of moisture absorption vary with increase of fiber volume in the polymer matrix. Better result was found by Ghosh and Gangul [8] after exposure to water and moisture for different periods by surface modification of jute fiber in polyester and found to exhibit greater weathering resistance and better mechanical properties. Low density of fiber was used as reinforcement in polymer composites and examined the properties of the natural, lignocellulosic fabrics by Rajula et.al [9] As this fiber possesses good mechanical properties with low density it can be used as reinforcement in polymer composites. It is important to improve the fibers mechanical properties were done by surface modification [10].

In this article, banana– polypropylene composites fabrication technique, Structural (FTIR spectroscopy) and morphological (SEM morphology) behavior of polypropylene reinforced with short natural fiber banana of both treated and untreated is reported designated as PPBC. IR reports the absorption due to a particular bond deformation occurs at approximately the same frequency (n) for all molecules that contain that bond. Polymer identification is the simplest application of IR spectroscopy [11]. The stretching and bending vibration of interest for infrared spectroscopy for two atoms are joined by a covalent bond around tetrahedral carbon. In stretching, the distance between two atoms increases or decreases, but the atoms remaining in the same axis. These vibrations require higher energy and occur at higher frequency. In bending or deformation, the distance between two atoms remains constant, but the positions of the atoms may change with respect to the original bond axis. This type of vibration requires lower energy and occurs at lower frequency [12]. This is because of some kind of interfacial contact between banana fiber and polypropylene in the presence of NaOH. That was approved by SEM reports.
In this case alkali reacts with the hemicelluloses as cementing materials of the fiber. This leads to the destruction of the mesh structure of the fiber and splitting the fibers into finer filaments matrix [13]. Due to alkali treatment fiber bonding increases as a result crystalline of the fiber cellulose increases and making the fiber more brittle and rigid [14].

2. Experimental

2.1 Materials

Banana fibers were collected locally with the help of PP and PDC, BCSIR, Dhaka. We have used Polypropylene (PP T101) containing specific gravity of 0.9. For experimental purposes banana fibers were chopped within the length of approximately 2-3 mm and sieved with 2 mm sieve (DIN ISO3310/1 w=2mm, FRITSCH). To remove moisture all these fibers and PP granules were kept at 110°C for 24 hours to remove moisture.

2.2 Alkali Treatment

20% NaOH solution was placed in to RB flask container and sufficient amount of chopped banana fibers were immersed in to the solution for 15 minutes at room temperature. Another container was taken for washing the fibers and had done thoroughly by distilled water several times. Dilute sulfuric acid solution was used for neutralized alkali treated fibers and rinsing them for several minutes. Again distilled water was used for washing the banana fiber following the method of Guha Roy et al [15].

2.3 Composite Fabrication

Fiber and PP were mixed at room temperature and a blender (Philips, India) was used and it was done for a period of 4 minutes at 410 rpm. Mold releasing agent was used in the die or mold. Then fiber pp mixtures were taken in a die. Paul-Otto-Weber Press machine was used for composite fabrication and load of 50 KN was given to top of the mold area (6"×6"). The mold was kept in press machine. Microprocessor controller controlled the total heating system. Total heating system was controlled electrically and temperature was set at 180°C. It took only 25–30 minutes to reach the above temperature. The temperature was set at that temperature for 30 minutes. To get void free and actual shaped sample as designed final load of 50 KN was set over the sample area. Pressure was increased up to 100KN when the temperature was reached at 180°C and the heating system was stopped. For cool down the heated sample water circulation system was arranged through the outer area of mold. The sample had been cooled for 10 hours. After that the Specimens were separated from the mold. After that the samples were cut in different shapes according to different tests and labeled of these samples before stored in a desiccators. ASTM standard was maintained for sample preparation.

2.4 Oven

Electronically controlled oven (UE / BE MEMNERT, UAE / SLF 600, GERMAN) was used for sample preparation.

Ambient Conditions: Ambient temperature 5°C - 40°C
MAX 80% oven voltage category II contamination degree according to IEC 664.

Temperature Range: Ambient to 280°C

2.5 IR Sample Preparation

Both untreated and alkali treated banana fibers were dried at 105°C for 24 h and powdered in a mortar. For IR analysis were obtained by mixing and grinding a small amount of powdered fiber materials (1mg) of fiber compound samples with dry and pure KBr (200 mg). Mixing and grinding were done in a mortar by a pestle. Powdered mixture was then compressed in a metal holder to produce a pellet and pressure of 8-10 tons was required. For IR measurements the pellet samples were then placed in the path of the infrared beam. Composite fabrication of both treated and untreated PP banana fiber composites containing 10 wt % short fibers were done in a rotary mixer (Philips, India) and was used for 5 minutes at a speed of 30 rpm. The mixing was set at 180°C for 20-22 minutes. For composite fabrication compressed and moulded them into sheets (1-2 mm thickness) with a Paul-Otto-Weber Press machine. The temperature was set at 180°C for 20 minutes. After reaching the above temperature a load of 50 KN was set to have required shape, thickness and void free samples. For cooling the mold water circulation system was arranged through the out sides of the mold area. Specimens were separated after cooling and pellets were cut from the sheet. The pellet was then placed in the path of the infrared beam for measurements.

2.6 Infrared Spectrophotometer

The spectrophotometer used for IR spectral analysis was IR 470, SHIMADZU CORPORATION, KYOTO, JAPAN. It is a double-beam spectrophotometer. The photometer works in direct ratio method with the use of the independent dual frequencies modulation. Its wave
number range in 4000 - 400 cm\(^{-1}\) and resolution in case of normal operation is 2.7 cm\(^{-1}\) around 1300 cm\(^{-1}\) and in case of the stretched optimum parameter, the resolution is 0.8 cm\(^{-1}\) around 1000 cm\(^{-1}\). The spectrophotometer has its repeatability of the transmittance, 0.5%, except the wave number range, where the absorption bands of the water vapor exist. Its measuring time is 5 mins 45 secs with fast and record off. Two milligrams of the powered fibers were mixed with 100mg of KBr to prepare pellets of sample.

2.7 Morphology (SEM)

The surface topography of fibers and cords treated under different conditions was investigated using a Hitachi S-2600N Scanning Electron Microscope with 20 nm resolution, and equipped with Oxford Isis Energy Dispersive Spectroscopy system including a light element detector and digital imaging. The samples were stuck on carbon tapes mounted on the SEM sample holder. Then they were coated with a thin layer of vapor-deposited silver to induce conductivity before examination under SEM. The acceleration voltage was 25 KV.

3. Results and Discussion

3.1 FTIR Report

The infrared spectra of both untreated and the alkali treated banana fibers were taken with good resolution and are shown in Figure 1 and 2. The peak assignments of the absorption bands corresponding to various groups are summarized in Table 1. It is evident that the characteristic feature of the infrared (IR) spectra of banana fibers are due to its lignin and hemicelluloses constituents. The H-bonded O-H stretching causes the broad band between 3434 and 343 per cm. The O-H is present Cellulose and hemicelluloses fiber. The usual glucose in the cellulose is characterized through the C-H stretching and bending for establishing the presence of aromatic nucleus around the band. The usual glucose linkage in the cellulose is characterized through the C-H stretching and bending for establishing the presence of aromatic nucleus around the band 2910-2900 cm\(^{-1}\). It is evident that the absorption peaks of C=O (small) stretching of uronic acid of the hemicelluloses is present in all the banana fiber at 1720 cm\(^{-1}\). There are linkage vibrations in the intensity of the peaks around 1505-1500 cm\(^{-1}\) and are attributed to the aromatic skeletal ring breathing with C-O stretching in syringyl units of lignin, but the peaks at 1445 cm\(^{-1}\) are due to the C\(_2\) symmetric bending. The normal or common C-OH stretching is characterized through the C-OH stretching around the 1160-1150 cm\(^{-1}\) band.

Figure 1. FTIR spectra of the short untreated banana fiber

Figure 2. FTIR spectra of the short treated banana fiber

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Figure 3. FTIR spectra of PP

Table 1. FTIR Spectral Data of untreated and alkali treated banana fibers.

| Wave Number (cm⁻¹) | Possible Assignment                      |
|-------------------|------------------------------------------|
| 3434              | Broad O-H stretching                     |
| 2910              | C-H-stretching (vibration)               |
| 1720              | C=O (small) stretching of hemicellulose  |
| 1640              | Broad (absorbed water)                   |
| 1500              | Aromatic skeletal ring vibration due to lignin |
| 1455              | CH₃ symmetric bending                   |
| 1260              | ---                                      |
| 1150              | ---                                      |
| 1020              | C-OH stretching                           |

3.2 IR Spectra of PP and PP-B Composites

Figure 3, 4 and 5 show the infrared spectra of PP, untreated and alkaline- treated PP-Banana fiber composites. In Table 2 and 3 show the peak assignment of the absorption bands corresponding to various groups are summarized. The characteristic feature of the infrared (IR) spectra of PP-B composites are due to its lignin and hemicelluloses constituents. The IR spectra of composites were taken with good resolution. As evident from the Figures, there is no obvious change in the intensity of peak around 2910 cm⁻¹ and 1370 cm⁻¹ that corresponds to the usual glucose linkage in the cellulose is characterized through the C-H stretching vibration and bending for establishing the presence of aromatic nucleus. It indicates that there is no structural change for glucose linkage in the cellulose through the C-H stretching and bending in treated and untreated PP-B composites. It is necessary to mention that the C=O stretching of the uronic acid of the hemicelluloses, and the unconjugated keto groups of the lignin can be observed around 1720-1710 cm⁻¹.There is no obvious change in the intensity of the peak around 1455 cm⁻¹ that corresponds to CH₃ symmetric bending in PP-B composites aromatic skeletal ring vibration of lignin. This clearly indicates that no structural change for the lignin in the jute fabric was observed upon alkali treatment. It is evident that the absorption peak at 1000 cm⁻¹ corresponding to the C=O (small) stretching of uronic acid of the hemicelluloses is present in the spectrum of both alkali- treated and untreated PP-B composites. This clearly indicates that no structural change occurs due to hemicelluloses.

Figure 4. FTIR spectra of untreated PP banana

Table 2. FTIR Spectral Data of pure PP sample

| Wave Number (cm⁻¹) | Possible Assignment |
|-------------------|-------------------|
| 2910              | C-H stretching vibration |
| 1720              | C=O                |
| 1660              | Broad peak (absorbed water) |
| 1455              | CH₃ symmetric bending |
| 1370              | CH bending         |
| 1000              | C-OH stretching    |
banana fibers pulled out in PP-B composites prepared by alkali treated fiber which represent the microstructure in SEM report. On the other hand 6(c) shows a appropriate evidence of interfacial brittle failure nature. It is a great indication of interfacial contact between banana fiber and PP matrix in the presence of NaOH. This may happen because NaOH reacts with hemicelluloses as cementing materials of the fiber. This leads to the destruction of the mesh structure of the fiber and splitting the fibers into finer filaments. This splitting of fiber bundle increases the effective fracture surface area available for wetting by the matrix \[17\]. Thus the bonding between the banana fiber and the PP matrix at the interface may be improved. The increase in fiber bonding and stiffness is thought to be due to an increase in crystallinity of the fiber cellulose making the fiber more rigid and brittle \[18\].

3.3 Fractography (PP-B)

SEM image gives more clear idea about the characteristics of fracture. In Figure 6(a) and (b) show the image of 10% and 25% untreated PP-B composites respectively. In this image it observed that around the banana fibers there are some voids and also some spaces in where banana fibers have been pulled out. This may occur between fibers and polymer matrix due to the poor adhesion in the absence of compatibilizer. There were clear evident of poor fiber matrix interfacial adhesion due to debonding and fiber pull out with fairly alkali treated clean fiber surface without matrix adherence \[16\]. In Figure 6(c) and 6(d) show the relatively less void spaces and lower proportion of voids. A→Fiber Pull out, B→Fiber bend

Figure 5. FTIR spectra of treated PP banana.

Table 3. FTIR Spectral Data PP-B sample

| Wave Number (cm\(^{-1}\)) | Untr. Fiber | Trt. Fiber | Possible Assignment       |
|--------------------------|------------|------------|---------------------------|
| 2910                     | 2910       | C-H stretching (vibration) |
| 1720                     | 1710       | C=O/ small pea |
| 1640                     | 1625       | Absorbed water (broad)    |
| 1460                     | 1460       | CH\(_2\) symmetric bending |
| 1370                     | 1370       | CH bending (small)         |
| 1310                     |            | --                      |
| 1000                     | 1000       | C-OH stretching (small)    |

Figure 6(a). SEM of fracture surface of 10% untreated PPBC.

Figure 6(b). SEM of fracture surface of 25% Untreated PPBC.
In the present study there is no structural change due to alkali treatment for the lignin in the banana fiber. The absence of some IR bands and appearance of new bands of low intensities in alkali – treated PP-fiber also support the fact that on alkali treatment. Aggregation occurs thereby stabilizing the structure of PP-fiber composites. From the analysis of fracture surfaces of the composites by SEM it indicates that fracture surfaces indicate the fracture behavior to be brittle. Fiber deboning and less void spaces observed in PPB composites due to less fiber strength of banana fiber. Also we observed improvements in the mechanical properties in the composites which are related to the state of polypropylene impregnation into the banana fiber- PP matrix.

4. Conclusions

Short banana fibers matrix composites were prepared by melt mixing hot press compression molding technique. The structural and morphologies of the PPB treated and untreated fiber composites were fabricated and investigated. There is an improvement in mechanical strength and interfacial bonding of the PPBCs are due to removal of both natural and artificial impurities. In PPBCs NaOH played a vital role in improving the interfacial bonding and adhesion between natural fiber banana and polypropylene matrix. Important factor fiber volume percentage in the PPB Composites might have caused to their superior strengths. Infrared (IR) spectra of PP-B composites indicates that there is no structural change for glucose linkage in the cellulose through the C-H stretching and bending in treated and untreated PP-B composites. Obviously it is clear from fractography that increase in stiffness and fiber bonding occurs for increase in crystallinity factor of the fiber cellulose as a result making the fiber more rigid and better strength.

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