EFFECT OF SURFACE TREATMENT ON MECHANICAL BEHAVIOR OF JUTE FIBER-REINFORCED POLYOPROPYLENE COMPOSITE

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1 Introduction

In the recent years, considerable research and development have been expanded in natural fibers as reinforcement in thermoplastic resinous matrix [1-11]. The various advantages of natural fibers over man-made fibers are low cost, low density, competitive specific mechanical properties, reduced energy consumption, and biodegradability [3-4]. Natural fibers are usually made of cellulose, hemicelluloses, lignin and pectin with a small quantity of water soluble materials. However, the one of primary disadvantage is poor interfacial adhesion and dispersion in olefinic thermoplastic matrix materials due to hydrophilic character of cellulose [5-6].

Plant fibers are covered with pectin and waxy substances, thus hindering the hydroxyl groups from reacting with polar matrices, and forming mechanical interlocking adhesion with non-polar matrices. The presence of surface impurities and the large amount of hydroxyl groups make plant fibers less attractive for the reinforcement of polymeric materials [7].

To produce the reactive hydroxyl groups and the rough surface for adhesion with polymeric materials, plant fibers need to undergo physical and/or chemical treatment to modify the surface and structure. There are various methods for promoting interfacial adhesion in systems where lignocellulosic materials are used as fillers[8-12]. These methods are usually based on the use of reagents which contain functional groups that are capable of reacting and form chemical bonds with the hydroxyl groups of the lignocellulosic material, while maintaining good compatibility with the matrix. During alkali treatment, physical structure of the fibers changes as a result of alkali's bleaching action which removes waxy materials, and impurities. This action often leads to improvement of the interfacial bonding between fibers and matrix [8].

Mwaikambo et al. [6] used FTIR to confirm that hemicellulose was removed by the alkali treatment.

The present study aims to study the effect of a solvent free, catalyst free, anhydride treatment on different lignocellulosic materials, in order to produce cost effective totally biodegradable composites. Two different methods alkali (NaOH) treatment and plasma treatment were investigated in this study. The formal technique was investigated in room temperature condition varying the weight percentage of NaOH and the later method was carried out to make uniform deposition of pin hole free thin film over jute fibers. Surface morphology was investigated by ATR-FTIR and SEM methods. Finally, fibers were made specimens for tensile test and flexural test.

2 Experimental

2.1 Materials

For SEM and ATR-FTIR test, two different country's commercially available jute fibers were used in this study. Fibers which purchased from Vietnam were used for plasma treatment while fibers from Philippines were used for alkali treatment. Tensile test and flexural test specimens of alkali and plasma treatment were made by Philippines fiber. Polypropylene (PP) is used as matrix. Maleic anhydride (MA) is used as coupling agent.

2.2 Alkali treatment

In this process untreated jute fibers were dipped in 1–7% wt of NaOH solutions at ambient temperature (22ºC) over 24 h duration maintaining fiber weight to alkali volume ratio of 1:50. Commercially available sodium hydroxide (NaOH) pellets of 98% purity

were used in alkali treatment of jute fibers. After treatment, jute fibers were washed with distilled water thoroughly. A pH meter was used to measure the acidity or alkalinity of the fibers. Washing was stopped when pH value reached a neutral value nearly 7. Finally, all fibers were dried at 80ºC for 24 h.

2.3 Plasma treatment

Plasma treatment technology, a dry process, is very simple and the cost effective. In addition, this treatment produces no environmental pollution. Plasma treatment has been applied to recover inactivated jute surfaces for better adhesion and bonding and to increase wettability. The schematic diagram of plasma treatment process is shown in Fig. 1. Helium was used as a plasma gas and plasma frequency and voltage were applied 20 KHz and 3 KV, respectively during experiment. The general reactions to be achieved by plasma treatment are the oxidation of the surface of a material, the generation of radicals, and the edging of the surface. As the surface is oxidized, the hydrophilic character is changed to become increasingly hydrophobic by changing to carboxyl group (COOH). Effects of surface modification of jute fibers have been observed by changing the plasma treatment time.

2.4 Attenuated total reflectance-fourier transform infrared analysis (ATR-FTIR)

In order to confirm the changes of functional groups under treatment of jute fibers, ATR-FTIR analysis was conducted. The ATR-FTIR was performed using a ATR-FTIR Matson Galaxy Series 6300 Spectrometer. The spectrometer was used in the transmission mode with a resolution of 4 cm^{-1} in the range of 4000~400 cm^{-1}.

Fig.1. Equipment of plasma treatment

2.5 Scanning electron microscopy (SEM)

The surfaces of untreated and treated with alkali and plasma fibers were observed using scanning electron microscopy, SEM, Model JSM 5610 (JEOL, JAPAN). Prior to the test, the samples were coated with a thin layer of gold to avoid sample charging under the electron beam. The observation was performed in high vacuum mode with secondary electron detector and accelerating voltage between 5 and 10 kV.

2.6 Mechanical properties

Untreated and treated jute fibers, which were treated by 1 wt. %, 3 wt. %, 5 wt. %, 7wt. % NaOH solutions and 2mins plasma treatments, were chopped into short lengths about 2mm to insure an easy blending for both manual and twin-screw extrude processes. A 10 wt % chopped jute fibers, 2 wt. % MA, and 88 wt. % PP were compounded in a manual mixer. The blends were molded in a twin-screw extruder (PRIM TSC 16TC, Thermo Electron Corp.) to form rods of diameter 1.0mm by using the melt mixing process. The temperatures of five extruder sections from first heating zone to the die were set as 180 ºC, 200 ºC, 210 ºC, 200 ºC, and 180 ºC. After extrusion, rods were cut into pellets. Then, pellets were dried in vacuum oven at 70ºC for 24hours. The dried pellets were used to prepare the dog-bone specimens for tensile testing in accordance to the ASTM standard using an injection molding equipment.

The tensile tests were carried out according to the ASTM D 638 Type I standard (dog-bone specimens) using a Universal Testing Machine (R&B 301 Unitech). Crosshead speed was 2.50 mm/min. Simultaneously, extension meter having 25 mm gauge length was used to measure the change of strain.

3 Results and discussions

3.1 ATR-FTIR examination of esterified jute fibers

ATR-FTIR spectra results of untreated jute fibers, alkalized jute fibers were presented in Fig. 2. The broad absorbance peak at 3200~3600 cm^{-1} range is attributed to the O–H stretching of hydrogen bond network, which becomes less intense resulting in alkali treatment. The reduction in intensity is more prominent for 5 wt. % of alkali treated jute. Such decrease is due to the breaking of hydrogen bond

between O–H groups of cellulose and hemi-cellulose molecules. The absorbance peaks at 2900 cm^{-1} represents C–H stretching vibration of methyl and methylene groups in cellulose and hemicelluloses which shows the similar pattern as O-H groups show. A sharp peak appeared at 1740 cm^{-1}, which may be attributed to C=O stretching vibration of hemicelluloses, however it shows the removal of hemi-cellulose molecules upon treatment. The absorbance bands at 1432 cm^{-1} and 1376 cm^{-1} assigned to –CH_3 asymmetric and C–H symmetric deformations of lignin also showed decreasing due to alkali treatment. The peak at 1250 cm^{-1} corresponding to C–O stretching in the acetyl groups in hemicelluloses is also demolishing which is an evidence that hemi-cellulose is removed from fiber surfaces by the alkali treatment.
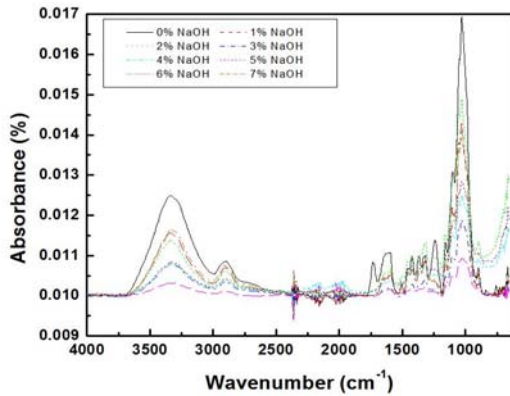
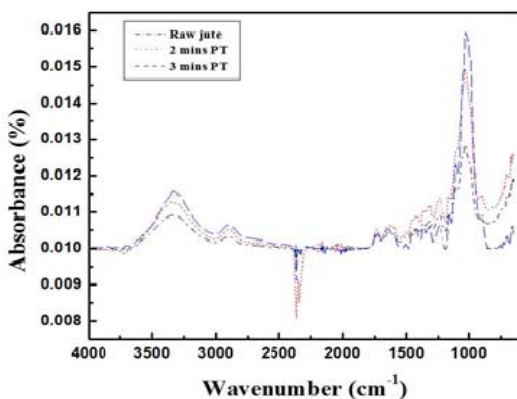Fig.2. ATR-FTIR spectral pattern of untreated and alkali treated jute fibers.

Fig.3. ATR-FTIR spectral pattern of untreated and plasma treated jute fibers.

3.2 ATR-FTIR examination of plasma treated jute fibers

Fig. 3 describes a typical series of ATR-FTIR spectra for untreated and treated jute fibers. Plasma polymerization fibers shows reverse trend compared to alkali treated fibers. The absorbance peak at 3200~3600 cm^{-1} range presents the O–H stretching of hydrogen bond network, which becomes more prominent upon plasma treatment. This is because of strengthening of hydrogen bond between O–H groups of cellulose and hemi-cellulose molecules. The band in the spectrum near 2900 cm^{-1} shows the same behavior. The absorbance bands at 1740 cm^{-1} which is assigned to C=O stretching vibration of hemicelluloses shows no change upon plasma treatment whereas the absorbance peak at 1432 cm^{-1} and 1376 cm^{-1} show lower intense which means lignin is less active on surfaces after plasma treatment.
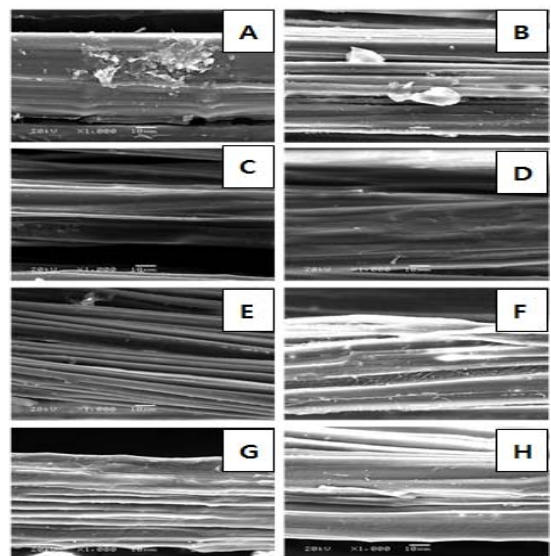
3.3 Surface morphology by SEM

Fig.4. SEM images of untreated (A) and treated by (B) 1%, (C) 2%, (D) 3%, (E) 4%, (F) 5%, (G) 6% and (H) 7% NaOH jute fibers.
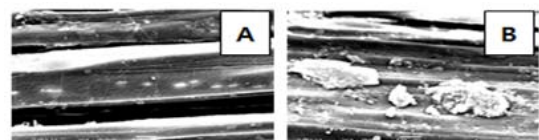

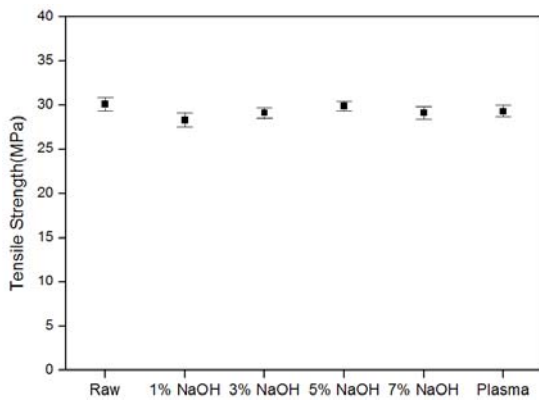Fig.5. SEM images of untreated (A) and treated by plasma for (B) 2 min jute fibers.

Fig.6. Result of tensile strength for jute fiber reinforced composites
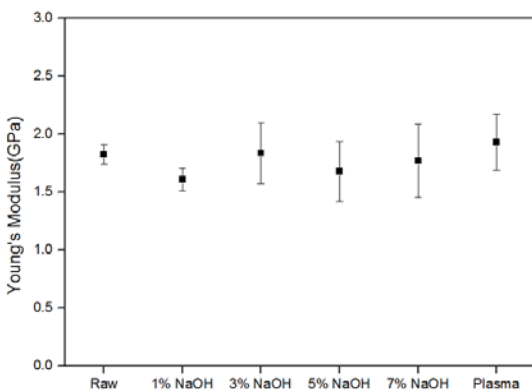

Fig.7. Result of tensile modulus for jute fiber reinforced composites
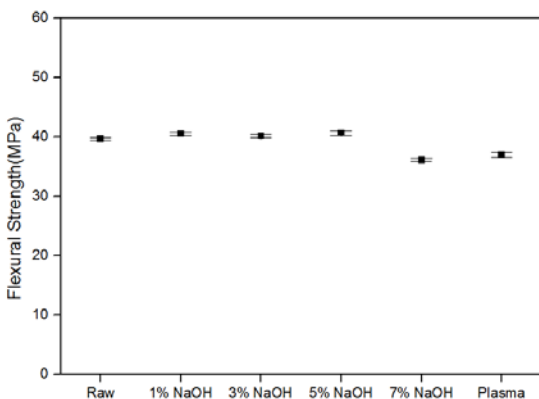

Fig.8. Result of flexural test for jute fiber reinforced composites

SEM images of untreated and esterified jute fibers are shown in Fig. 4. Significant changes in surface morphologies were observed after alkali treatment. It shows that the untreated fibers are covered with a layer, which was mainly composed by waxy substances. After alkali treatment the surface of jute fibers become smoother which is caused by removal of waxy substances. In case of alkali treatment with over the 2 wt. % NaOH, the results show a good removal of wax and rougher surface compared with untreated and others treated cases. Fig. 5 shows SEM images of untreated and plasma treated jute fibers. The untreated fibers are smoother than plasma treated jute fibers. Surfaces of jute fibers were covered with carboxyl group after plasma treatment.

3.4 Mechanical properties

Figures 6, 7 and 8, these figures show that treatment did not have obvious effect on increasing tensile and flexural test on strength comparing raw results. Flexural strength is stable raw to 5wt. % NaOH treated fiber reinforced composite however, flexural strength decrease when 7wt. % NaOH treated.

4 Conclusions

ATR-FTIR experiments was observed through SEM analyses that the surface of the untreated fibers is rich in waxes and wax like substances, and the treatments altered the fiber surface characteristics, by removing the outer surface layer and producing a smoother fiber surface and alkali treatment with 5 wt. % NaOH shows the best result. Plasma treatment has been conducted in atmospheric conditions to recover inactivated jute surfaces for better adhesion and bonding between fiber and matrix in composites. From ATR-FTIR and SEM observations, it is clear that jute surfaces have carboxyl group (COOH) because of plasma polymerization of hexamethyl-disiloxane.

Jute fiber reinforced polypropylene matrix composites were successfully produced by the injection molding technique. However, the improvement of tensile strengths and modulus of the composites could not be observed. It was not match with that the addition of NaOH contents as treatment solution improved the composite performance by enhancing the adhesion between jute fibers and MAPP.

1) MA effect is stronger than surface treatments. MA was already treated between PP and jute fiber adhesion.

2) Fiber treated process was not good. Fiber was cut after treatments. It could damage surface treated results.

3) Fiber length is not enough to affect surface treatment. Fiber length is shorter than 1mm. If fibers shape is cylindrical, diameter and length are 1mm, treated surface is just 60% of surface. Fiber length would be shorter than 1mm percentage is decreased fast.

The treated surface caused that was not enough to improve the mechanical properties by previous reasons. Therefore, it needs modification for experiment.

7 wt. % NaOH treated fiber reinforced composite flexural strength is decreased less than 5 wt. % treated (worse than raw). It shows that jute fiber dipping in 7 wt. % NaOH 24h is too strong to be used for reinforced composite. It causes damage on fiber conditions.

Acknowledgement

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