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Characterization of Sodium *meta*-Periodate Modified Betel Nut Husk Fibers Reinforced HDPE Composites

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ABSTRACT

Short betel nut husk fibers (BNHF) were used as reinforcing materials to prepare high density polyethylene (HDPE) matrix based composites. Waste betel nut husk fibers were extracted from betel nut fruit and the chemical composition of fibers were analyzed. Extracted fibers were also chemically treated with sodium meta-periodate to enhance its compatibility with the HDPE matrix. Treated BNHF characterized by Attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopic analyses. Different wt. percentages (5, 10, 15, 20, 25 and 30%) of both treated and untreated betel nut husk fibers were used to prepare compression moulded HDPE composites to achieve better interfacial bonding and mechanical properties. Tensile strength, elongation at break, TGA-DSC, Scanning Electron Microscopy (SEM) etc properties were analyzed for all treated and untreated betel nut husk fibers reinforced HDPE composites. It was found that 10% (w/w) treated fiber reinforced HDPE composites showed better results than all other composites.

Key words: High Density Polyethylene, Betel nut husk fiber, Composites, Tensile strength, Scanning Electron Microscopy

INTRODUCTION

Composites are materials consisting of two or more chemically distinct constituents, on a macro-scale, having a distinct interface separating them. One or more discontinuous phase therefore, is embedded in a continuous phase to form a composite. The discontinuous phase is usually harder and stronger than the continuous phase and is called the reinforcement, whereas, the continuous phase is termed the matrix [1]. Environmental friendly greener materials like natural fibers are promising alternative materials to traditional glass fibers due to their good specific strength, low cost, renewability, market appeal, fully biodegradable nature and non-abrasive character [2]. Natural fiber used as reinforcement can be classified into many categories i.e. inner bark, seed, straw fiber, leaf, and grass fiber. Hardwood saw dusts, jute fibers, palm fibers, coconut fibers, rice straw fibers are used as reinforcements in polymer composites due to low cost, low density and biodegradability [3-7]. In the past agro based waste products have been used as natural sources to collect natural fibers suitable for agro-tech protective clothing [8]. Instead of glass or carbon fiber and inorganic fillers, natural fibers from natural sources are more potential as biodegradable reinforcing materials. From different research work and application in various fields it has been proved that natural fiber reinforced and thermoplastic polymer matrix based composites are much more applicable. To prepare thermoplastic composites various matrix materials like HDPE, polypropylene(PP) [9-11], polyvinylchloride (PVC) [12-13], polyethylene (PE) [14-17], polystyrene (PS) [18] and poly(lactic acid) (PLA) [19] used with reinforcing agent such as betel nut fiber, kenaf, wood, flax, cotton, hemp, Kraft pulp, pineapple leaf, coconut husk, areca fruit, oil palm, abaca, sisal, henequen leaf, jute, banana, ovine leather, and straw etc.

A wide range of agro-based fibers is being utilized as the main structural components or as reinforcing agents in the fiber-matrix based composite materials which are used from decorative, interior design and automotive interior components. Uses of different natural fibers which works as reinforcing agent are also described elsewhere [5-7]. In addition, natural fibers are now being widely used as fillers in the different industries like plastic industry to acquire expected results as well as to reduce the price of the finished product [8].To develop a composite material made from natural fibers with significantly improved strength stiffness, durability and reliability, it is important to have better fiber matrix interfacial bonding. Cellulose, hemi-cellulose and lignin are the main component of natural fiber and hence natural fibers are commonly termed as ligno-cellulosic materials. These constituents make natural fibers hygroscopic and hydrophilic which they have poor compatibility with hydrophobic polymer matrix in natural fiber reinforced polymer composites. Better fiber matrix interfacial bonding can be achieved by the surface treatment of natural fiber. Many Researchers have analyzed that the chemical modification of the natural fibers optimizes the interfacial locking between the fiber and the matrix and improving the other properties. During this process hydroxyl groups which are on natural fibers were activated or some new functional groups will be introduce, which will create effective interlock bonding with the matrix. Chemical treatments used as surface modifications have been studied by many researchers by using base, permanganate, acetic anhydride, silane and maleic anhydride [17].

In Bangladesh, *Areca catechu* tree is found everywhere as well as widely found in the coastal area. Betel nut husk fiber which is generally unusable is extracted from the betel nut fruit. This biodegradable fiber is cheap and easily used to prepare composite materials. The aim of this research work is to characterize the extracted betel nut husk fibers and to use it as a reinforcing material with HDPE matrix at different wt. percentages. Another aim of this research work is to treat the extracted fibers chemically, which could improve the properties of the composites. Sodium *meta*-periodate was used for treatment with betel nut husk fibers. The novelty of this work is the chemical modification of betel nut husk fibers with sodium *meta*-periodate. Different types of properties such as tensile strength, elongation at break, water uptake, TGA-DSC analysis, soil degradation test as well as scanning electron [17]microscopy (SEM) etc were analyzed for all treated and untreated BNHF reinforced HDPE composites and reported in this paper. These composites can be used in automobile sector, interior design, aircraft sector and making furniture etc.

EXPERIMENTAL METHODS

Materials

Betel nut empty fruits were collected from a local plantation field in Bangladesh. A commercial grade high density polyethylene (HDPE) from PTT Global Chemical Public Company, Thailand was used as polymer matrix. Analytical grade sodium *meta*-periodate and other reagents were used from Merck, Germany.

Fiber Extraction

Betel nut husk fibers were soaked in water at room temperature for 15 days for retting. The retted fibers were separated by hand stripping. The retted fibers were brushed and cleansed with agitated water, and washed thoroughly with distilled water. Fibers were then dried in sunlight for about three days. The extracted fibers were dried at 105°C in an oven for constant weight.

Chemical treatment of betel nut husk fibers with sodium *meta*-periodate

The dried betel nut husk fibers were clean manually and cut into 1 cm in length, fibers were then washed with distilled water and dried in open air. The air dried fibers were oven dried at 105 °C for 5 hours. Sodium *meta*-periodate solution (0.6 M) was prepared in distilled water. Dried fibers were immersed in sodium *meta*-periodate solution. Fiber to liquor ratio was 1:16 (w/v). The pH of the solution adjusted to 5 by adding sulfuric acid. The reaction was carried out for 4 hours at 90 °C. The reaction mixture was stirred occasionally. The mixture was cooled and filtered to isolate oxidized fibers. The fibers were thoroughly washed in tap water and finally washed with distilled water. The washed oxidized fibers were air dried. The air dried fiber was dried in an oven at 105 °C for constant weight. These oxidized fibers were used for composite fabrication.

Preparation of BNHF reinforced HDPE polymer Composites

The fabrication of untreated and sodium *meta* periodate treated BNHF reinforced HDPE composites were carried out by compression molding technique. The mould (12×15 cm^2) was cleaned and filled with dry blended mixture of fibers and HDPE powders to prepare composites. The weight fraction 5%, 10%, 15%, 20%, 25%, 30% of untreated and treated BNHF was carefully controlled during the mixing of two ingredients. The composites were hot pressed under 160°C temperature and 200 N pressures.

Table -1 Different weight fraction of BNHF reinforced HDPE composites

Formulation	Fiber content (wt %)	HDPE powder (wt %)
No. 1	5	95
No. 2	10	90
No. 3	15	85
No. 4	20	80
No. 5	25	75
No. 6	30	70
No. 7	00	100

Table -2 Different weight fraction of $NaIO_4$ treated BNHF reinforced HDPE composites

Formulation	Fiber content (wt %)	HDPE powder (wt %)
No. 8	5	95
No. 9	10	90
No. 10	15	85
No. 11	20	80
No. 12	25	75
No. 13	30	70

Characterization of Fibers and Composites

ATR-FTIR spectroscopic characterization of untreated and treated betel nut husk fibers

The ATR-FTIR spectra of untreated and treated BNHF were recorded on a FT-IR/NIR Spectrometer (Frontier, PerkinElmer, USA).

Mechanical Properties of the Composites

Tensile strength and elongation at break test

The tensile properties of all untreated and chemically treated BNHF reinforced HDPE composites prepared were carried out using a universal testing machine, model -1410 Titans, James Heal, UK. The load capacity was 5000 N, efficiency was within ± 1%. The crosshead speed was 50.00 mm / min and gauge length was 50.00 mm. The tensile strength measurements were carried out according to ASTM D-3039 method at a standard laboratory atmosphere of 30 °C and relative humidity.

Thermo gravimetric analysis (TGA) and differential scanning calorimetric (DSC) analysis

The thermo gravimetric analysis and differential scanning calorimetric analysis of the prepared 30 wt% treated and untreated BNHF-HDPE composites were recorded on a STA 449F3 NETZSCH thermal analysis instrument in the temperature range of 0-900 °C.

Scanning electron microscopic (SEM) investigation

Scanning electron microscopy (SEM) is an important tool to the surface morphology study of materials. The raw untreated betel nut husk fiber, treated betel nut husk fiber and the fracture surface of tensile specimens of prepared 30 wt% treated and untreated BNHF-HDPE composites were examined using a Hitachi S-4000 field emission scanning electron microscope, operated at 5 kv.

RESULTS AND DISCUSSION

Characterization of BNHF

BNHF was extracted and analyzed. The analyzed ingredients are found in BNHF are presented below:

1. Aqueous Extract = 0.5647%
2. Fatty and waxy maters = 1.3796%
3. Pectic maters = 0.9173%
4. Lignin = 14.865%
5. α-cellulose = 51.0776%
6. Hemi cellulose = 14.8653%
7. Ash = 2.6877%

Surface modification of raw betel nut husk fiber with sodium *meta*-periodate

Fig. 1 shows the ATR-FTIR spectra of treated and untreated betel nut husk fibers. Both treated and untreated fibers were characterized by ATR-FTIR spectroscopy to confirm the chemical reaction of sodium *meta*-periodate with lignocelluloses element of fibers. The IR spectrum of treated fibers clearly showed the characteristic bands of aldehyde group at the region of 2920 cm^{-1} due to C-H stretching. The peak at 1738 cm^{-1} seen in untreated fiber which was disappeared upon chemical treatment. This was due to the removal of carboxylic group by chemical treatment.

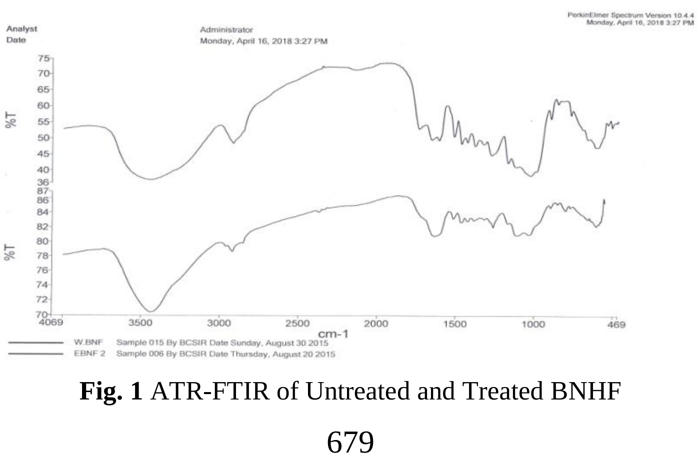

Fig. 1 ATR-FTIR of Untreated and Treated BNHF

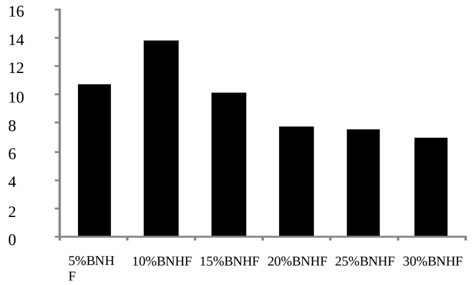

Fig. 2 Tensile strength *vs* wt % of untreated BNHF of untreated BNHF-HDPE composites

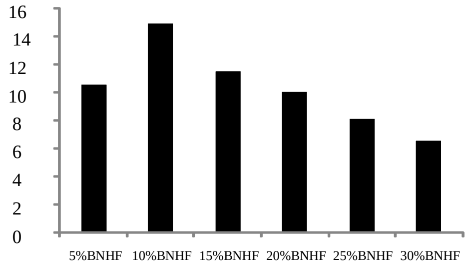

Fig. 3 Tensile strength *vs* wt % of $NaIO_4$ treated BNHF of BNHF-HDPE composites

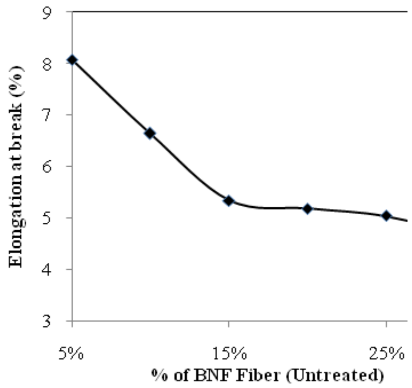

Fig. 4 Elongation at break (%) *vs* wt % of untreated BNHF of untreated BNHF-HDPE composites

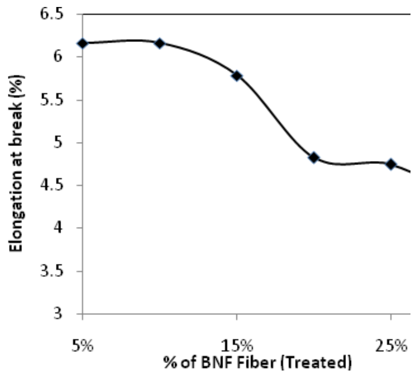

Fig. 5 Elongation at break (%) *vs* wt % of BNHF of treated BNHF-HDPE composites

Mechanical Properties of Untreated and Treated Betel nut Fiber Reinforced HDPE composites

Tensile strength, elongation at break and of untreated and chemically treated BNHF reinforced HDPE composites were studies and there results are presented below:

The tensile strengths of the untreated and treated BNHF-HDPE composites are shown in figure 2 and figure 3. It is observed from the figure that the tensile strengths of the composites increased up to 10 wt% fibers loading & then decreased. The elongations at break of untreated and treated BNHF-HDPE composites are shown in figure 4 and figure 5. Elongation at break of all composites decreased with increasing fiber loading. Tensile properties of all treated BNHF-HDPE composites were higher than that of untreated BNHF-HDPE composites. The tensile strength and ductility were better in the case of treated BNHF-HDPE composites than that of untreated BNHF-HDPE composites. This may be the reason for the improvements of the fiber-matrix interfacial adhesion in composites made by sodium periodate treatment.

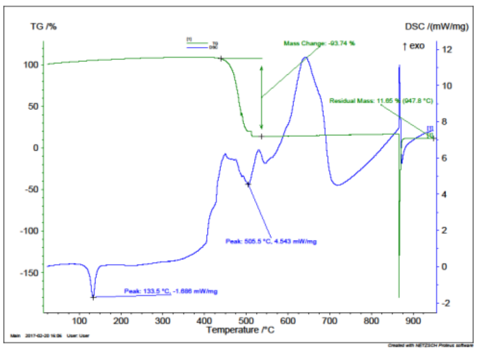

Fig. 6 TGA &DSC curve of HDPE

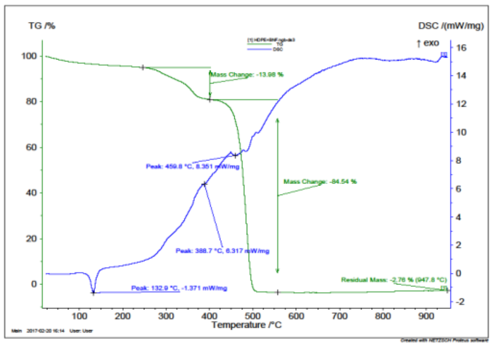

Fig. 7 TGA &DSC curve of 10 wt% untreated BNHF-HDPE composite

Thermal properties of Untreated and Treated Betel Nut Fiber Reinforced HDPE Composites

Thermal properties of neat HDPE, 10 wt% untreated and treated BNHF-HDPE composites were evaluated by Differential scanning calorimetery (DSC) and thermo gravimetric analysis(TGA) and the pictures of DSC and TGA curves were presented in the figures 6, 7 and 8 respectively. It is observed from the figures that almost same thermal properties were found for treated and untreated BNHF-HDPE composites. But neat HDPE showed slightly higher thermal stability than the BNHF-HDPE composites. The melting temperature of neat HDPE and untreated and treated BNHF-HDPE composites were found at 133.5^{0}C, 132.9^{0}C and 132.8^{0}C respectively. So addition of treated and untreated BNHF with HDPE didn't affect the melting temperature of neat HDPE.

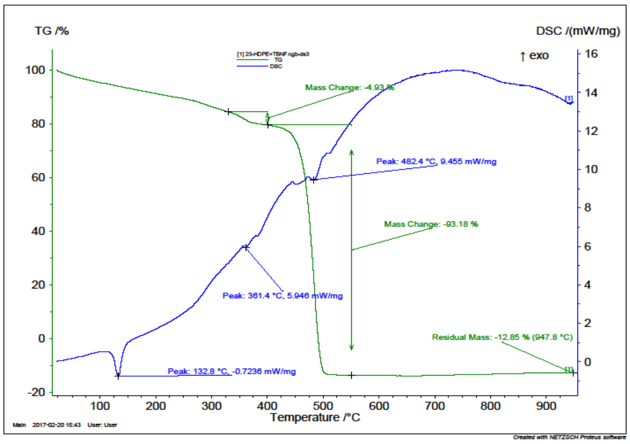

Fig. 8 TGA &DSC curve of 10 wt% treated BNHF-HDPE composite

Morphological Characterization of Untreated and Sodium meta periodate Treated Betel Nut Fiber reinforced HDPE composites

The surface morphology of the untreated fiber, treated fiber and their composites of 30 wt% untreated and treated BNHF with HDPE were studies by scanning electron micrograph (SEM) images and presented in the figures 9 to 12 respectively. From the analyses of SEM images of untreated and treated BNHF, it was found that treated BNHF was smoother surface than the untreated BNHF. The SEM images of treated BNHF-HDPE composite indicated the better interfacial adhesion between treated BNHF and HDPE matrix as compared to untreated BNHF-HDPE composite.

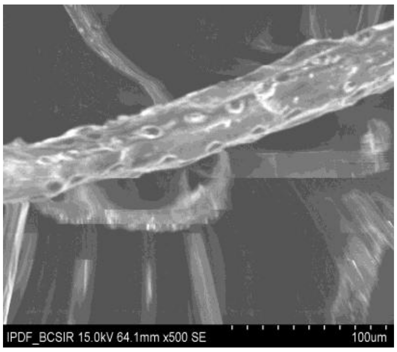

Fig. 9 SEM image of untreated BNHF

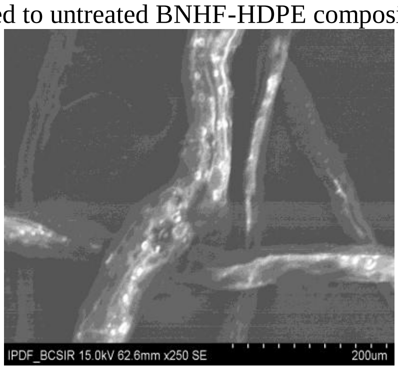

Fig. 10 SEM image of treated BNHF

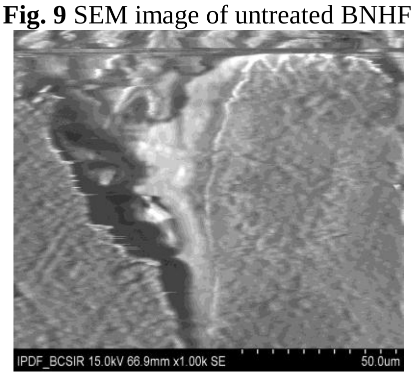

Fig. 11 SEM image of untreated BNHF-HDPE composite

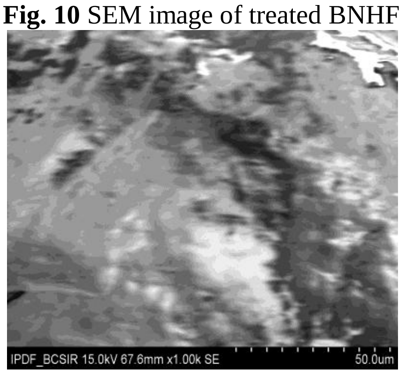

Fig. 12 SEM image of treated BNHF-HDPE composite

CONCLUSIONS

The extraction and characterization of betel nut husk fibers were done and used as a reinforcing material with HDPE matrix at different wt. percentages. Chemical treatment of extracted fibers with sodium meta periodate showed improved mechanical properties for the treated BNHF-HDPE composites comparing with the untreated BNHF-HDPE composites. ATR-FTIR spectra and SEM analyses of fibers also evidenced the occurrence of chemical modification in treated BNHF. The mechanical properties of the composites showed that the treated BNHF-HDPE composites had significant improvement at 10 wt% treated BNHF loading. SEM micrographs also revealed the evidence of improved mechanical properties for treated BNHF-HDPE composites than untreated BNHF-HDPE composites. No significant changes have been found from thermal properties of treated and untreated BNHF-HDPE composites. Thermal stability of pure HDPE was slightly higher than that of treated and untreated BNHF-HDPE composites. Therefore, BNHF-HDPE composites would be biodegradable materials.

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