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Paper

Studies on *Borassus* fruit fiber and its composites with Polypropylene

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ABSTRACT

This paper summarizes the structural characterization of *borassus* fruit fibers by means of various characterization techniques, optimization of alkali treatment of *borassus* fruit fine fibers (BFF) with a 5% concentration sodium hydroxide solution for different time intervals (1, 4, 8 and 12 h) and the changes occurring in *borassus* fibers. This paper also discusses the manufacturing of BFF/PP compotes using MAPP as a compatibilizer in addition to alkali treatment. Composites were evaluated for their mechanical and morphological properties. The tensile strength and modulus, flexural strength and modulus and impact strength were increased for alkali treated/MAPP composites by 4.5%, 17%, 17.2 %, 9% and 10% respectively.

Key Words : Borassus fruit fiber, Alkali treatment, Polypropylene, Maleated polypropylene

1. Introduction

Over the last few years, ecological concerns have initiated a considerable interest in natural materials to produce ‘green’ products. The use of natural fiber composites has increased due to the unlimited availability of natural fibers, their relative cheapness compared to conventional materials such as glass and aramid fibers, multi-functionality, their ability to be recycled, and for the fact that they compete well in terms of strength per unit weight of material[1-7]. Natural fibers like jute, flax, hemp coir and sisal have all proved to be good reinforcement in thermoset and thermoplastic matrices and have been used in automotive applications, construction as well as in packaging industries[8-13].

Borassus (Palmyra Palm) is a genus of six species of fan palms, native of tropical regions of Africa, Asia and New Guinea, economically useful and widely cultivated. It is a long life tree and can live up to 100 years reaching a height of 30 m, with a canopy of leaves and a large trunk resembling that of a coconut tree. The Palmyra palm has been one of the most important trees of Cambodia and India, where it is useful over 800 different ways. The fruits are eaten either roasted or raw, and the young, jellylike seeds are also edible[14,15]. Preliminary studies on properties of *Borassus* fruit fibers have already been reported by the authors[16]. The authors also further investigated the effect of alkali treatment (for 1, 4, 8 and 12 h) on the *Borassus* fine fiber tensile properties and chemical composition. In addition, the morphology and structural, crystallinity and thermal degradation of *Borassus* fibers were also investigated.[17]. In continuation to the above preliminary research, we further intended to fabricate *borassus* fruit fiber/Polypropylene (BFF/PP) composites by using 5 wt % of fiber. In order to improve the adhesion between fibers and matrix, maleated polypropylene (MAPP) was also used as compatibilizer [The grafting of maleic anhydride (MA) on PP is one popular method for improving the interfacial adhesion] [18,19] in addition to the alkali treatment. Mechanical and morphological properties of untreated, alkali treated and MAPP modified fiber reinforced composites were explored.

In the present paper, we are summarizing the basic research carried out so far on BFF modification by alkali treatment, and the mechanical and morphological properties of BFF/PP composites.

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2. Experimental

2.1 Materials

Borassus fibers were extracted from the ripened fruits based on the procedure reported elsewhere[16]. Acetic acid, benzene, ethanol, sodium bisulphite, sodium chlorite and sodium hydroxide pellets (Merck Chemicals) were used as purchased. The coupling agent, Poly (propylene)-graft-maleic anhydride (MAPP) (Aldrich) and Sodium hydroxide (NaOH) (Dae-Jung Chemicals) were purchased and used as received. Polypropylene was obtained from Honam Petrochemical Corporation, South Korea.

2.2. Composites fabrication

PP, short fine fiber and MAPP (for modified composites) were mixed using a twin-screw extruder (PRISM, TSE 16TC, Thermoelectron Corporation). The temperature of barrel and die was 190 °C and the screw speed was 120 rpm. Subsequently, the extrudate was palletized, dried, and injection molded into standard specimens for mechanical properties testing. The injection-molding temperature and pressure were 200 °C and 60MPa, respectively.

2.3 Instrumentation

Thermal properties were studied by DSC and TGA. The wide-angle X-ray diffraction spectra of the fibers were recorded on a Rigaku Dmax 2500 diffractome. A Universal Testing Machine (Norwood, MA) at a crosshead speed of 5 mm/min with a gauge length of 50 mm was used for single fiber testing. In each case, 10 samples were used and the average was reported. Tensile, flexural and impact properties of BFF/PP composites were studied according to ASTMD 6389, D790-03 and ASTM 256-88 respectively. The morphology of the fibers and nanocomposites were studied by SEM.

3. Results and discussion

3.1 Structural characterization of *borassus* fruit fibers

Work relating to the extraction of the BFF from the fruit, study of the chemical composition, and the influence of the 5 % alkali treatment on the properties of the fibers were reported in the literature[16]. Usually a *borassus* fruit contains cores and fine fibers as shown in the Fig. 1

The composition of the fibers is estimated by chemical analysis and the analysis of untreated fibers indicates the presence of cellulose, hemicellulose, and lignin. The results also show that the alkali treatment reduces the hemicellulose fraction considerably for both coarse and fine fibers. To confirm the changes in the composition upon alkali treatment of the *Borassus* fibers, FTIR and high resolution carbon ^{13}C NMR (CP-MAS) techniques were performed. The disappearance of the characteristic stretching vibrations in FTIR spectra clearly indicates that the alkali treatment had significantly removed the hemicellulose content. In (CP-MAS) spectra the methyl group of CH_3COO of hemicellulose component observed in the raw fibers has disappeared and accordingly no signal was noticed at 20.85 ppm. The spectral features indicate that the alkali treatment affects only the hemicellulose component leaving the other two components, a-cellulose and lignin unaffected. XRD results show that the index values for coarse fibers are 27.46 (untreated) and 38.90 (treated) whereas for fine fiber the values are 32.0 (untreated) and 36.93 (treated). The increase in crystallinity of treated fibers is due to loss of amorphous hemicelluloses as suggested by chemical analysis, FTIR, and ^{13}C CP-MAS NMR spectroscopy.

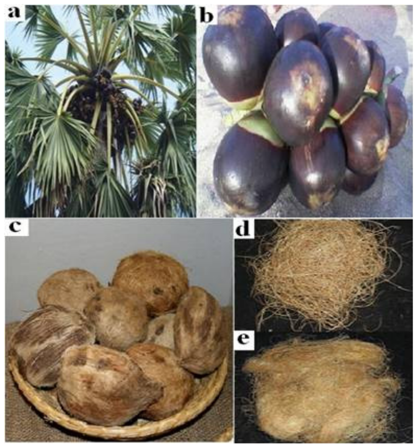

Fig. 1 (a) *Borassus* tree, (b) fruits, (c) dried fruits, (d) *Borassus* fruit coarse fibers and (e) fine fibers.

Surface and cross section of the treated and untreated coarse and fine fibers were examined by SEM analysis at different magnifications. The SEM pictures indicate the roughening of the surface for the treated fibers whereas the cross section of the fiber indicates multicellular structure. Each unit cell of fibers is composed of small particles of cellulose surrounded and cemented together with lignin and hemicellulose. Tensile properties were also evaluated for both untreated and alkali treated coarse and fine fibers. The data revealed that for both coarse and fine fibers, the maximum stress, Young’s moduli, and % elongation at break were increased on alkali treatment. The presence of hemicellulose,

Table 1 Tensile properties of untreated and alkali treated *Borassus* fine fibers

Alkali treatment time (h)	*Tensile properties*		
	Strength (MPa)	Modulus (GPa)	Elongation (%)
0 h	70.8	10.8	34.8
1 h	106.3	26.2	42.7
4 h	117.5	32.9	51.1
8 h	121.3	35.2	58.1
12 h	101.8	25.5	35.7

separating the cellulose chains from one another may create more strain, could be the reason for decreased mechanical properties of untreated fibers[16,20].

TGA analysis of coarse and fine fibers revealed that the alkali-treated fibers have a slightly higher thermal stability than the untreated fibers which is ascribed to the removal of amorphous hemicellulose from the fibers upon alkali treatment. The results also suggested that these fibers can also be used as reinforcement for making thermoplastic matrix materials while maintaining the processing temperature below 270 °C.

3.2 Optimization of alkali treatment of *Borassus* fruit fine fibers

Borassus fruit fine fibers were treated [17] with a 5% concentration sodium hydroxide solution for different time intervals (1, 4, 8 and 12 h) and were characterized by tensile testing, chemical analysis, FTIR, XRD, SEM and TGA in order to optimize the effect of alkali treatment on mechanical properties.

As shown in Table 1, the tensile strength, modulus and % elongation of the fibers improved by 41, 69 and 40% respectively, up to 8 h of alkali treatment. This is due to the fibers tending to get closely packed owing to the large scale removal of hemicellulose by alkali treatment and formation of new hydrogen bonds in between the chains of cellulose fibrils. Thereby the fibrils rearrange themselves in a more compact manner resulting in closer fiber packing[20] as evidenced from Fig. 2, for the fibers treated for 4 h. After 8 h alkali treatment, the tensile properties were decreased, possibly due to the degradation of cellulose in longer duration of alkali treatment. These results were further supported by XRD, thermal and chemical analysis. The crystallinity index of untreated and 5% alkali treated fibers (for 1, 4, 8 and 12 h) were found to be 38.4, 47.1, 47.4, 58.4 and 47.2 respectively.

The higher crystallinity index of alkali treated fibers can be attributed to the removal of amorphous hemicellulose from the fibers and the rearrangement of the crystalline regions in such a way that the fiber exhibits a more crystalline nature. Table 2 shows the changes in chemical composition of untreated and alkali treated *borassus* fruit fine fibers. From the results, we can infer that the Hemi-cellulose content decreased continuously with the increase in time of alkali treatment. However, the α-Cellulose content also decreased after 12 h of alkali treatment, which could be due to the damage of molecular structure of cellulose with increasing the time of alkali treatment[21].

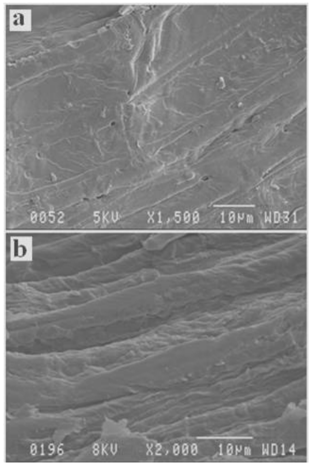

Fig. 2 SEM photographs of Untreated (a) and alkali treated (4 h) (b) *Borassus* fruit fine fibers.

Table 2 Chemical composition of untreated and alkali treated *Borassus* fruit fine fibers

Alkali treatment time (h)	*Chemical composition*		
	α-Cellulose (%)	Hemi-cellulose (%)	Lignin (%)
0 h	53.4	29.6	17.0
1 h	60.3	16.3	23.4
4 h	61.6	14.4	24.0
8 h	63.0	12.6	24.4
12 h	62.3	12.3	25.4

Based on the properties determined for the *Borassus* fibers, the authors[16,17] suggested that these fibers will be suitable for use as reinforcement in green composites.

3.3 Fabrication of BFF/PP composites

With a view of exploring the potential use of BFF, we made an attempt to fabricate new *borassus* fruit fine fiber/Polypropylene (BFF/PP) composites using 5 wt % of fiber as well as MAPP [23]. According to the literature reports [18,19, 23, 24] the optimum dosage of MA-g-PP as a compatibilizer into PP is 5%. Therefore, in this study, BFF/PP composites were fabricated after surface chemical treatment of the fiber by alkali treatment to improve the binding capability of the fiber with the PP matrix in the presence of MAPP as a compatibilizer. Though the results[17] show the optimized alkali treatment condition is 8 h, we have treated the fibers with a 4 % alkali treatment for 5 h in order to minimize fiber damage.

The BFF were treated with 4% NaOH for 5h at room temperature maintaining the material-to-liquor ratio of 1: 20, washed with distilled water until the fibers showed no residual NaOH (neutral pH). Then the fibers were allowed to dry at 70 °C for 12 h. The dried alkali treated and untreated fibers were chopped into 5-10 mm, stored in a sealed plastic bag for further processing.

PP, short BFF and MAPP (for modified composites) were mixed by using twin-screw extruder and the extrudate was palletized, dried, and injection molded into standard specimens for mechanical properties testing. All the components were first dried in an oven at 80 °C for at least 24 h. The tensile strength was measured as per the ASTMD 6389 standards with a cross-head speed of 5 mm/min and the flexural strength was measured according to the ASTM D790- 03 with a cross-head speed of 2 mm/min using three-point bending mode tests. Impact test was performed employing an Izod impact testing machine followed by ASTM 256-88 specifications. The impact test was carried out at room temperature and impact energy was reported in J/m. For each test, five replicate test specimens were taken and the average value was recorded.

Table 3 shows mechanical properties such as tensile, flexural and impact properties of virgin PP and its corresponding untreated (UPP), alkali treated (TPP) fiber composites, with MAPP (TPP-MAPP) and without MAPP (UPP-MAPP). The results show that the mechanical properties of untreated and alkali treated fiber composites were lower to those composites with compatibilizer. This is because the incorporation of compatibilizer reduces the surface hydrophilicity of the fibers, leading to the enhanced fiber wetting and dispersion within the matrix, decreasing the water absorption and increasing the mechanical properties[7]. The tensile strength and modulus, flexural strength and modulus and impact strength were increased for TPP-MAPP by 4.5%, 17%, 17.2 %, 9% and 10% respectively.

Figure 3 shows SEM photographs of tensile fractured surfaces of UPP and UPP-MAPP composites. As evidenced from Fig. 3 (a) and (b) of UPP and TPP composites, fiber pull out from the matrix during the fracture process indicates poor interfacial interaction between fiber and PP matrix causing poor mechanical properties.

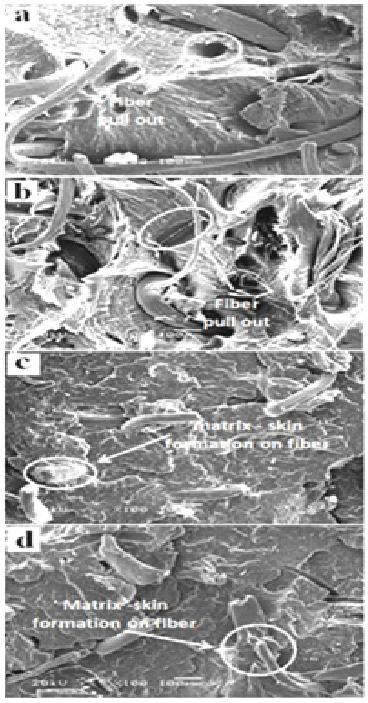

Fig. 3 Fractured surfaces of UPP (a), TPP (b), UPP-MAPP (c) and TPP-MAPP (d) composites.

Table 3 Mechanical properties of alkali treated and untreated BFF/PP/ MAPP composites

Name of the sample	*Tensile strength (MPa)*	*Tensile modulus (MPa)*	*Flexural strength (MPa)*	*Flexural Modulus (MPa)*	*Impact strength (J/m)*
PP	22.45	1250.65	35.09	1083.24	22.82
UPP	22.31	1460.76	39.40	1077.37	23.89
TPP	22.26	1411.34	40.47	1086.27	21.81
UPP-MAPP	22.98	1486.34	41.16	1113.39	24.93
TPP-MAPP	23.48	1508.14	42.40	1189.96	25.36

On the other hand, in composites with MAPP compatibilizer (Fig. 3c and d), fibers broken during the tensile test indicates the strong interactions between fiber and matrix. This interaction is also evidenced by the matrix-skin formation on the fibers which causes the lower number of fiber pullouts and voids due to fibers not coming out totally out of the matrix. The fractured surfaces were also distributed more uniformly.

4. Conclusion

The prime objective of the authors of this study is to explore the potential use of *Borassus* fruit fibers as a green composite reinforcement. The following salient features were discussed in this paper. The composition of the native fibers was determined by chemical analysis, which indicates the presence of cellulose, hemicellulose and lignin.

The tensile properties of the fibers revealed that young's modulus, % elongation at break are higher for treated fibers in contrast to native fibers. It is also found that the alkali treatment for 8 h is identified as an optimized condition for improved tensile properties attributed to the large amount of hemicellulose dissolution. The crystallinity index of alkali treated fibers is found to be higher than that of the untreated fibers, attributed to the removal of amorphous hemicellulose from the fibers and the rearrangement of the crystalline regions in such a way that the fiber exhibits a more crystalline nature. The removal of hemicellulose from fiber cells leads to a closer packing of cellulose chains. Longer period of alkali treatment would have caused damage to the molecular structure of cellulose.

Regarding BFF/PP composites, the mechanical properties of untreated and alkali treated fiber composites were lower than composites with compatibilizer. The tensile strength and modulus, flexural strength and modulus and impact strength were increased for TPP-MAPP by 4.5%, 17%, 17.2 %, 9% and 10% respectively.

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