Structural Characterization and Tensile Properties of *Borassus* Fruit Fibers

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Received 2 December 2008; accepted 5 April 2009 DOI 10.1002/app.30584 Published online 8 June 2009 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The chemical and instrumental analysis of alkali-treated *Borassus* fibers is carried out to explore the possibility of their use as reinforcement in green composites. The chemical analysis shows presence of α -cellulose, hemicellulose, and lignin. This is further confirmed by FTIR and high-resolution solid-state ¹³C NMR spectroscopy. The influence of alkali treatment on morphology and mechanical properties is attempted by SEM and UTM techniques, respectively. The wide-angle X-ray dif-

fraction analysis of the native and treated fibers shows that alkali treatment influences the crystallinity of the fibers. The efficacy of the *Borassus* fibers (native and treated) as a component of green composites is discussed. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 603–611, 2009

Key words: *Borassus* fruit fibers; crystallinity; hemicellulose; lignocellulose; morphology; thermogravimetric analysis

INTRODUCTION

Polymers are quintessential materials that have revolutionized the human civilization for many years.¹ Polymer composites are distinct class of polymers often used for wide range of applications from commodity to exotic areas like space exploration.² However, environmental pollution is a major cause for concern to the polymers.³ The polymers and composites easily do not undergo biodegradation resulting in generation of solid waste. To address such an important issue, efforts are focused on making polymer composites that are biodegradable by incorporating materials that are of natural occurance.^{4,5} Such composites are popularly known as green composites⁶⁻⁹ and the process basically involves incorporation of naturally occurring polymers that can undergo biodegradation. As a consequence, many studies have focused on natural polymers that occur as fibers.¹⁰⁻¹⁴ Among them sisal, coir, pineapple, jute, bamboo, etc. have gained the attention of researchers.¹⁵⁻¹⁹ These natural fibers may have to be modified either by chemical or by other means to make them suitable for making composites for final applications. Although preliminary studies on Borassus fibers are reported in the literature,²⁰ however, a complete and thorough study of coarse and fine *Borassus* fibers is relatively underexplored. The unique elastic nature of these fibers with high modulus has been one of the prime reasons for the choice of these fibers. The prime objective of this study is to explore the potentials of *Borassus* fibers as a green composite reinforcement. The work involves extraction of fibers (coarse and fine) from the fruit, alkali treatment, and their structural characterization using chemical analysis, FTIR, solid-state NMR, and other extensive studies. Studies on morphology, mechanical behavior, and X-ray diffraction are carried out to support our objective on the use and an application of *Borassus* fibers.

MATERIALS AND METHODS

Materials

Coarse and fine fibers were extracted from *Borassus* fruits and commercial grade sodium hydroxide pellets (Merck, India), benzene, sodium chlorite, acetic acid, sodium bisulphate, and ethanol (SD Fine Chem, India) were used.

Extraction of the fibers from the fruits

The *Borassus flabellifer* fibers were extracted from dried ripened fruits. The fruits were dipped in water for 2 weeks and the black skin was separated. Below it, two types of fibers—long coarse and short fine fibers were found. The coarse fibers were present in

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Journal of Applied Polymer Science, Vol. 114, 603–611 (2009) © 2009 Wiley Periodicals, Inc.

fruit nut edge to edge, whereas the fine fibers are adhered to the shell of the fruit. Both types of fibers were thoroughly washed with tap water followed by distilled water and dried in the sun for a week. Then the fibers were kept in hot air oven for 24 h at 105–110°C to remove the moisture. A portion of *Borassus* fibers were treated with 5% aqueous sodium hydroxide (NaOH) solution at room temperature, maintaining a liquor ratio of 30 : 1, and fibers were immersed in the alkali solution for 30 min to remove the hemicellulose and other greasy materials. Then the fibers were washed with water repeatedly and treated with dilute acetic acid. Finally, the fibers were washed with distilled water before drying in hot air oven for a period of 24 h.

INSTRUMENTAL ANALYSIS

FTIR spectroscopy

The FTIR spectra of all the samples were run on ABB-BOMEM FLATA-2000 model spectrophotometer using KBr pellets. The samples were powdered with KBr and the pellets were used for recording the spectra in transmission mode.

¹³C NMR (CP-MAS) spectroscopy

¹³C CP-MAS NMR of all the samples was run on Bruker DSX 300 MHz solid-state NMR spectrometer at Indian Institute of Science, Bangalore, India. The operating frequency for ¹³C nuclei was 75.46 MHz. The samples were spun with filled 5 mm rotor at two different spinning rates of 5.0 and 7.5 KHz, respectively at room temperature. The spinning side bands were identified by comparing the spectra measured at two spinning rates.

Chemical analysis

The chemical analysis of both coarse and fine fibers was carried out as per the standard procedure.^{21,22} In this analysis, the percent values of α -cellulose, hemicellulose, and lignin were determined. In each case, the average of tested five samples was reported.

Thermogravimetric analysis

The thermograms of the fibers were recorded on a Perkin Elmer TGA-7 instrument in nitrogen atmosphere at a heating rate of 10° C/min in the temperature range of 50–600°C.

X-ray analysis

The wide-angle X-ray diffraction spectra of the fibers were recorded on a Rigaku Dmax 2500 diffractome-

ter (Tokyo, Japan). The system has a rotating anode generator with a copper target and wide-angle powder goniometer. The generator was operated at 40 KV and 150 mA. All the experiments were performed in the reflection mode at a scan speed of 4° / min in steps of 0.05°. All samples were scanned in 20 range varying from 5 to 50°.

Morphology

The scanning electron micrographs of the surface of the fibers were recorded on a JEOL JSM 820 microscope (Akishima, Japan). The micrographs of the cross section of the fibers were also recorded. The fiber samples were gold coated before recording the micrographs.

Tensile properties

The tensile properties, such as maximum stress, Young's modulus, and % elongation at break of the fibers, were determined using INSTRON 3369 Universal Testing Machine (Norwood, MA) at a crosshead speed of 5 mm/min maintaining a gauge length of 50 mm. In each case, 10 samples were used and the average was reported.

RESULT AND DISCUSSION

The prime work involved in the methodology is to extract fibers from fruit, study the chemical composition, and investigate the influence of the alkali treatment on the fibers. This approach is carried out on natural fibers for understanding the chemical composition and the mechanical behavior. The alkali treatment facilitates removal of soluble weak components and greasy substances from the fiber. The Borassus fruit belongs to Arecaceae family with B. flabellifer L species. Figure 1 presents the photographs of Borassus tree with fruits, coarse, and fine fibers. The average length, diameter, and aspect ratio of the coarse fibers are found to be 20, 0.028, and 610 cm, respectively whereas for fine fibers these parameters are found to be 12, 0.013, and 556, respectively, which are based on 100 single fibers.

The composition of the fibers is estimated by following chemical analysis procedure reported in recent literature.²³ Table I shows the chemical composition of both native (untreated) and alkali-treated fibers. The chemical analysis of untreated fibers (Table I) indicates the presence of α -cellulose, hemicellulose, and lignin. The chemical structures of α cellulose, hemicellulose, and lignin are already available in the literature.²⁴ The α -cellulose structurally consists of D-glucopyranose units linked by β -position whereas hemicellulose consists of similar units of α -cellulose with branching.²³ As a result of



Figure 1 Photographs of (A) *Borassus* tree with fruits; (B) *Borassus* coarse fibers; and (C) *Borassus* fine fibers. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

branching, hemicellulose exists in amorphous form whereas α -cellulose is crystalline. On the other hand, lignin is a polymer consisting of phenolic units with alkoxy functional groups. Usually, natural fibers are treated with alkali to remove weak amorphous components so that the fiber retains crystalline components thus enhancing the strength to the fiber.25-29 Hence, the Borassus coarse and fine fibers are accordingly treated with alkali and the chemical composition of the treated fibers is shown in Table I. A close examination of the results indicates that alkali treatment reduces hemicellulose fraction considerably for both coarse and fine fibers. However, the reduction in hemicelluloses content is more for fine fibers. Although small fraction of lignin would have been soluble in alkali, chemical analysis predominantly shows loss of hemicellulose for treated fibers.

To confirm the changes in the composition on alkali treatment of the *Borassus* fibers, FTIR and highresolution carbon ¹³C NMR (CP-MAS) techniques were used. Table II shows the characteristic absorptions of α -cellulose, hemicellulose, and lignin frac-

tions of the coarse and fine fibers. The FTIR spectra of the native and alkali-treated coarse and fine fibers are presented in Figure 2. In the case of both the native fibers, a strong absorption band at around 3420 cm⁻¹ corresponding to the OH-stretching vibration is observed. The appearance of very intense and broad absorption is a clear indication of many hydroxyl groups in the fiber. This observation supports that the hydroxyl groups are involved in the hydrogen bonding, which is polymeric in nature. The C-H stretching vibrations of methyl and methylene units of all three constituents is seen at 2920 cm⁻¹ as very broad peak. In the region 1750– 900 cm⁻¹, many absorption peaks are noticed whose intensities vary from low, moderate to high. Among them, notable are 1737, 1649, 1458, 1374, 1246, and 1052 cm^{-1} values (Table II). Of them, 1737 cm^{-1} is attributed to ester carbonyl of hemicellulose component. In fact, hemicellulose consists of both ester and acid functionality. The close examination of absorption at 1737 cm⁻¹ indicates presence of a shoulder signal at 1710 cm⁻¹, which is arising from acid

		TABLE I				
Chemical Analysis of	Untreated and	Alkali-treated	Borassus	Coarse	and Fin	e Fibers

	Coa	Coarse fiber		Fine fiber		
Component	Untreated	Alkali-treated	Untreated	Alkali-treated		
% α-Cellulose	45.67	48.24	53.40	60.02		
% Hemicellulose	32.76	25.42	29.60	17.98		
% Lignin	21.53	26.33	17.00	22.00		

Journal of Applied Polymer Science DOI 10.1002/app

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Fiber component	Functional group	Wave number (cm ⁻¹)		
α-Cellulose	O—H stretching	3420		
	O—H bending	1374		
	C—H stretching	2920		
	C—H bending	1458		
	C–O stretching	1052		
Hemicellulose	C=O stretching of (carbonyl ester)	1737		
	C=O stretching of (carboxylic acid)	1715		
	C—H stretching	2920		
	-C-O-C-stretching	1246		
Lignin	-O-H stretching	3400		
0	-C-O-C stretching	1100		
	-C=C-stretching (aromatic ring stretching)	1605-1557		
	Aldehyde (CHO)	1649		
	Ketonic (–C=O)	1667		
	$-CH_3$ bending	1458		
	$-CH_2$ bending	1374		

TABLE II Absorption Bands for Functional Groups of Cellulose, Hemicellulose, and Lignin in Untreated and Alkali-treated Borassus Coarse and Fine Fibers

carbonyl. Another vibration at 1649 cm⁻¹ with a shoulder at 1667 cm⁻¹ is attributed to carbonyls of aldehyde and ketonic groups typically present in the lignin component. The aromatic ring C=C stretching vibrations are noted in the region 1605-1557 and the intensities of them is very low. The C-H bending vibration of alkyl units is noted at 1458 cm⁻¹ and 1374 cm⁻¹. The characteristic C–O–C stretching vibration of ester groups of hemicellulose is seen at 1246 cm⁻¹ whereas the C–O stretching of hydroxyl functionality is observed at 1052 cm⁻¹. The later

absorption is very broad and intense indicating the presence of more hydroxyl groups in the fiber.

The examination of the spectra of alkali-treated coarse and fine fibers shows peaks at 3420 and 2918 cm⁻¹, which are assigned to OH and CH stretching vibrations, respectively. However, the characteristic 1737 and 1710 cm^{-1} which are primarily due to CH₃COO- and COOH functionalities of hemicellulose noted for the raw fiber are now absent. The disappearance of these characteristic stretching vibrations clearly indicates that the alkali treatment



Figure 2 FTIR spectra of untreated and alkali-treated Borassus (coarse and fine) fibers.



Figure 3 ¹³C CP-MAS NMR spectra of untreated and alkali-treated *Borassus* (coarse and fine) fibers.

had significantly removed the hemicellulose content. This is further supported by the chemical analysis of alkali-treated fibers. On the other hand, the appearance of 1649 and 1637 cm⁻¹, indicates that the lignin component is intact. The disappearance of ester and acid carbonyl stretching vibrations is further supported by reduction in the intensity of 1246 cm⁻¹ (C–O–C). For other vibrations, *i.e.*, 1458, 1374, 1053 cm⁻¹ no appreciable change is noticed. Thus, the FTIR studies suggest the reduction of the hemicellulose content on alkali treatment of the fibers. This is in support of the chemical analysis data of the alkali-treated fibers as shown in Table I.

To further confirm the changes on alkali treatment of the fiber, high-resolution ¹³C NMR (CP-MAS) spectroscopy is used. To eliminate the spinning side bands in the spectrum, the samples are spun at two different speeds viz. 5 and 7.5 KHz, respectively. Because the spinning side band intensity is lower, spectra run at 7.5 kHz are only considered for discussion. Figure 3 shows the ¹³C CP-MAS NMR of the raw and alkali-treated fibers. The spectra of native coarse and fine fibers show a broad peak at 171.05 ppm with moderate intensity. The signal is contributed by the CH₃COO and COOH groups of hemicellulose. Another poorly resolved broad signal at 152.02-149.49 ppm is arising from the aromatic rings adjacent to alkoxy/phenol functionalities. The observation of this signal supports the presence of lignin in the fiber. This is arrived based on the fact that only lignin consists of aromatic carbons whereas

cellulose and hemicellulose are constituted by aliphatic and carbonyl carbons.³⁰ Yet, another low intense and broad peak in the region 129-126 ppm, which is primarily from aromatic ring carbons (ortho/meta) further corroborates the lignin fraction.³¹ Among the other important peaks seen in the spectra, there is a sharp and intense signal observed at 104.60 ppm. Earlier ¹³C CP-MAS NMR studies of cellulose-based natural polymers/fibers showed that this signal is contributed by the C1 carbons of both cellulose and hemicellulose units.^{31–33} The other constituted carbons of the cellulose include those appeared at 72-75 ppm, which are due to C2, C3, and C5 whereas the C4 carbon of the cellulose is noticed at 88.09 ppm.³⁴ The characteristic C6 carbon of cellulose is located at 64.33 ppm in the spectra. Among the signals noticed for cellulose carbons, those appeared at 72-75 ppm are of highest in intensity. The chemical shift values and intensity pattern suggest that these signals are also contributed by hemicellulose units. For instance, a signal at 83.09 ppm which is seen as shoulder to 88.09 ppm is contributed by the hemicellulose fraction. The appearance of 55.72 ppm signal, however, is arising from lignin fraction which has OCH₃ carbons. In the region, 32–30 ppm, a broad signal is noticed, which is assigned to the -CH₂ carbons of lignin fraction. A sharp signal with moderate intensity is seen at 20.85 ppm, which is arising from CH₃COO- group characteristic of hemicellulose. The chemical shift values and intensity characteristics of the spectra are



Figure 4 X-Ray diffractograms of untreated and alkali-treated Borassus (coarse and fine) fibers.

supportive of presence of cellulose, hemicellulose, and lignin in the native coarse and fine fibers.

The influence of alkali treatment on the ¹³C CP-MAS NMR chemical shift values is also investigated. The ¹³C CP-MAS NMR spectra of alkali-treated fibers are also shown in Figure 3. The notable features of the spectra include reduction in the intensity of carbonyl peak and deshielding of its chemical shift. This is also followed by an increase in the intensity of 152.35 ppm peak. Although the other important signals, *i.e.*, those appeared in the region 60-110 almost remain the same. The methyl group of CH₃COO of hemicellulose component observed in the raw fibers has now disappeared and accordingly no signal is noticed at 20.85 ppm. The spectral features indicate that the alkali treatment affects only the hemicellulose component leaving the other two components viz. *α*-cellulose and lignin unaffected. These observations are very much in consistent with the FTIR spectral data and chemical analysis. Almost similar trends are noticed for the fine fiber samples (treated and untreated).

The wide-angle X-ray diffraction patterns of coarse and fine fibers are shown in Figure 4. The diffractograms show two reflections, corresponding to 20 values of around 16 and 22°, respectively. Among these, the low angle reflection (16°) is of low intensity whereas the other reflection (22°) has higher intensity. Wang et al.³⁵ also observed two peaks for regenerated cellulose and $2\theta = 21.8^{\circ}$ and $2\theta = 16^{\circ}$ and attributed these to (002) and amorphous background, respectively. Further, to calculate the degree of crystallinity, they used the eq. (1). As the peak positions in the present case are similar to those observed by Wang et al.,³⁵ we have assumed the reflections at $2\theta = 22$ and 16° to (002) plane and amorphous background, respectively and calculated the degree of crystallinity using eq. (1).

$$I_c = \frac{[I_{(002)} - I_{(am)}]}{I_{(002)}} \times 100$$
(1)

Where $I_{(002)}$ ($2\theta = 22^{\circ}$) represents the intensity of crystalline peak whereas $I_{(am)}$ ($2\theta = 16^{\circ}$) denotes intensity of the amorphous peak in the diffractograms. Accordingly, 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), respectively. The increase in crystallinity of treated fibers is due to loss of amorphous hemicelluloses as suggested by chemical analysis, FTIR, and ¹³C CP-MAS NMR spectroscopy.

Scanning electron micrographs of the surface and cross section of the treated and untreated coarse and fine fibers at different magnifications are shown in Figure 5. 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. Similar features are noticed for lignocellulose fibers by earlier workers.^{16,19,27,28,36} Alkali treatment of the fibers tends to react with



Figure 5 Scanning electron micrographs of untreated and alkali-treated Borassus fibers: (A) untreated coarse fiber (×200); (B) alkali-treated coarse fiber (\times 200); (C) untreated fine fiber (\times 500); (D) alkali-treated fine fiber (\times 500); (E) cross section of untreated coarse fiber (×1500); (F) cross section of alkali-treated coarse fiber (×1500); (G) cross section of untreated fine fiber (\times 2000); (H) cross section of alkali-treated fine fiber (\times 2000).

cementing hemicellulose material and increase the effective surface available for wetting the resin.

The tensile properties of the coarse and fine fibers are presented in Table III. The data support that for both kind of fibers maximum stress, Young's moduli, and % elongation at break have increased on alkali treatment. As hemicellulose remains dispersed in the interfibrillar region separating the cellulose chains from one another for the native fiber, the cellulose chains are in a state of strain.³⁵ When the hemicellulose is removed by the action of alkali, the internal strain is released and the fibrils become more capable of rearranging themselves in a more compact manner and results a close packing of the fibers. Further, it is unambiguous (Table III) that the maximum stress and the modulus of the fine fibers are higher than those of the coarse fibers, especially, the average modulus of the fine fibers is nearly eight times higher than that of the coarse fibers. The increment in % elongation at break in the case of coarse fibers on alkali treatment is only marginal.

TGA thermograms of the coarse and fine fibers are presented in Figure 6. It is evident that the alkali-treated fibers have a slightly higher thermal stability than the untreated fibers. This may be due to the removal of amorphous hemicellulose from the fibers on alkali treatment. Further, the results indicate that these fibers can also be used as reinforcement in thermoplastic matrix materials whose processing temperature is below 270°C.

		TABLE III					
Tensile Properties	of Untreated an	d Alkali-treated	Borassus	Coarse	and F	Fine	Fibers

	Coarse fiber		Fine fiber		
Parameter	Untreated	Alkali treated	Untreated	Alkali treated	
Maximum stress (MPa)	50.9	53.5	65.2	90.7	
SD	5.2	4.7	2.7	6.0	
Young's modulus (MPa)	1221	1630	4918	9866	
SD	480	448	104.5	290	
% Elongation at break	41.2	41.9	47.2	58.5	
SD	2.5	6.7	1.6	2.3	

SD, standard deviation.

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Figure 6 Primary thermograms of untreated and alkali-treated Borassus (coarse and fine) fibers.

CONCLUSIONS

The Borassus fibers were extracted from the fruit by water treatment. The resultant fibers are classified as coarse and fine fibers based on dimensions. The composition of the native fibers is determined by chemical analysis, which indicates the presence of α cellulose, hemicellulose, and lignin, respectively. The alkali treatment of fibers resulted in solubilization of hemicellulose leading to reduction of its content in the fibers. This is further supported by FTIR and solid-state ¹³C NMR spectroscopy. The SEM analysis of untreated and treated fibers indicates changes in morphology. The decrease in the content of hemicellulose resulted in void formation as noticed for treated fibers. Wide-angle X-ray diffraction that showed an increase in the intensity of I_{002} (crystalline domain) further supported the spectral studies. The tensile properties of the fibers revealed that young's modulus, % elongation at break are higher for treated fibers in contrast to native fibers. The tensile properties of the Borassus fibers suggest that they are potentially significant as reinforcement component in green composites.

We (KOR and BRG) dedicate this article to Dr. (Mrs) B. Urmila Devi, wife of Professor A. Varada Rajulu, who treated us as her own children and who unfortunately passed away recently. We thank Dr. T. Narasimha Swamy, polymer Division CLRI (Chennai) and Professor K. V. Ramanathan, NMR Research center, IISc (Bangalore) for valuable suggestions in the preparation of the manuscript.

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