## **Properties of Lignocellulose Tamarind Fruit Fibers**

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**ABSTRACT:** Untreated and alkali-treated fibers from tamarind fruits were analyzed with Fourier transform infrared, chemical, X-ray, and thermogravimetric methods. The morphology of the fibers before and after the alkali treatment was studied with scanning electron microscopy. The tensile properties of these fibers before and after the alkali treatment were also studied. The Fourier transform infrared and chemical analyses indicated lowering of the hemicellulose content by the alkali treatment of the fibers.

The tensile modulus increased with the alkali treatment. X-ray diffraction revealed an increase in the crystallinity of the fibers with the alkali treatment. The thermal stability of the fibers increased slightly with the alkali treatment. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 1986–1989, 2008

**Key words:** biodegradable; biofibers; biopolymers; composites; renewable resources

#### INTRODUCTION

Because of ever-increasing pollution by polymers, the trend is slowly shifting toward the development of environmental friendly polymers and polymer composites. In this direction, the development of green composites is gaining momentum. Several workers1-14 have used plant- and vegetable-based fibers as reinforcements in the development of green composites. These natural fibers are renewable and environmentally friendly. To identify new natural fibers for use as reinforcements, their properties have to be studied. In this work, we studied the properties of tamarind fruit fibers to assess their suitability as reinforcements. Tamarind is widely used as an important food material in many Asian and African countries. The tamarind fruit has pulp, seeds, and fiber. In India alone, millions of tons of tamarind fibers are going to waste every year after the isolation of the pulp and seeds during food processing. This fruit also has many medicinal values. Because there is not much data available on this fiber, we studied some of its properties, such as its chemical composition, spectral analysis, thermal stability, X-ray diffraction, morphology, and tensile properties. Generally, lignocellulose fibers are subjected to a mild alkali treatment to lower their amorphous hemicellulose content. Such a treatment is expected to improve some of their properties. Hence,

the effect of an alkali treatment on these properties was also studied, and the results are reported in this article.

#### **EXPERIMENTAL**

#### Extraction of the fibers from the fruit

Fully ripened tamarind fruits were chosen for extraction of the fibers. After the removal of the shell of the fruit, the pulp and seeds were removed by squeezing. The isolated fibers were washed thoroughly with occasional shaking to remove any leftover pulp sticking to the fibers. These fibers were then dried in the sun. Some of the fibers were treated with a 5% aqueous NaOH solution and dried before analysis.

# Fourier transform infrared (FTIR) spectroscopy analysis

The fibers were cryogenically cooled and powdered. These powders were diluted to 1% with KBr, and pellets were prepared with a hydraulic press. The FTIR spectra of the untreated and alkali-treated samples were recorded in the 4000–500-cm<sup>-1</sup> region on a PerkinElmer 16PC FTIR instrument (Waltham, MA) with 32 scans in each case at a resolution of 4 cm<sup>-1</sup>.

#### Chemical analysis

The chemical analysis of the untreated and alkalitreated fibers was carried out according to the standard procedure.<sup>15</sup> In this analysis, the percentages of

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**Figure 1** Photograph of tamarind fruit, seeds, and fibers. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

 $\alpha$ -cellulose, hemicellulose, and lignin were determined. In each case, five samples were used, and the average values are reported.

#### Thermogravimetric analysis

The thermograms of the untreated and alkali-treated fibers were recorded on a PerkinElmer TGA-7 instrument in a nitrogen atmosphere at a heating rate of  $10^{\circ}$ C/min.

#### X-ray analysis

The X-ray diffraction spectra of the untreated and alkali-treated fibers were recorded on a Rigaku Dmax 2500 diffractometer (Tokyo, Japan). The system has a rotating anode generator with a copper target and a 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  $4^{\circ}$ /min in steps of  $0.05^{\circ}$ .

#### Morphology

Scanning electron micrographs of the untreated and alkali-treated fibers were recorded on a JEOL JSM 820 microscope (Akishima, Japan). An accelerating voltage of 10 kV was used for operating the scanning electron microscope to record the surface morphology of the fibers. However, this voltage was



Figure 2 Scanning electron micrographs of tamarind fibers: (a) untreated, (b) alkali-treated, (c) untreated (cross section), and (d) alkali-treated (cross section).



Figure 3 FTIR spectra of untreated and alkali-treated tamarind fibers.

reduced to 5 kV to record the micrographs of the cross section of the fibers at a higher magnification. The fiber samples were gold-coated before the micrographs were recorded.

#### **Tensile properties**

The tensile properties, such as the maximum stress, Young's modulus, and elongation at break (%), were determined with an Instron 3369 universal testing machine (Norwood, MA) at a crosshead speed of 3 mm/min with a gauge length of 50 mm. In each case, 10 samples were used, and the average values are reported. The standard deviation values, as computed by the instrument, are also reported.

#### **RESULTS AND DISCUSSION**

A photograph of the tamarind fruit, seeds, and fibers is presented in Figure 1. The fruit belongs to the Fabaceae family with the *Tamarindus indica* L. species. Scanning electron micrographs of the untreated and alkali-treated fibers are shown in Figure 2(a,b).

TABLE I
Peak Positions and Assignments of Chemical Groups in
the Untreated and Alkali-Treated Tamarind Fruit Fibers

Wave number (cm <sup>-1</sup> )			
Untreated	Alkali- treated	Assignment	
3408	3421	OH stretching of $\alpha$ -cellulose	
2918	2918	Alkyl CH stretching	
1738	_	CO stretching of hemicellulose	
1651	1640	CO stretching of lignin	
1251	1265	Asymmetric $C - O - C$	
1160	1160	stretching of lignin	
1115	1111	Symmetric CO stretching of lignin	
1051	1036		

The cross sections of these fibers are shown in Figure 2(c,d). From Figure 2(a,b), it is evident that the fibers have a roughly cylindrical structure. With the alkali treatment, the surface of the fibers became rough. The morphology of the cross section of the fibers indicates that the fibers have a multicellular structure. The cells are roughly circular in nature but do not have uniform dimensions. Furthermore, in the case of alkali-treated fibers, the cross section reveals a well-defined cellular structure.

FTIR spectra of the untreated and alkali-treated tamarind fibers are presented in Figure 3. The band positions and possible assignments are presented in Table I. From Figure 3, it can be observed that welldefined bands around 3400, 2900, 1740, 1650, 1250, 1110, and 1050  $\text{cm}^{-1}$  are present in the spectra. From Table I, it is evident that the bands around 3400 and 2900 cm<sup>-1</sup> correspond to OH stretching of  $\alpha$ -cellulose. The band around 1740 cm<sup>-1</sup> corresponds to CO stretching of hemicellulose. For the alkali-treated fiber, the intensity of this band decreases, and this indicates lowering of the hemicellulose content. The remaining bands belong to CO and C-O-C stretching of lignin. After the removal of the hemicellulose to a considerable extent, the percentage of  $\alpha$ -cellulose is expected to increase. The increase in the intensity of the bands around 3400 and 2900 cm<sup>-1</sup> for the alkali-treated fiber supports this observation. The chemical analysis values of the untreated and alkali-treated tamarind fibers are presented in Table II. From this table, it is evident that the  $\alpha$ -cellulose content increased slightly whereas the hemicellulose decreased with the alkali treatment. However, the lignin content remained almost the same. This is in agreement with the observations made in the FTIR analysis of the samples.

The X-ray diffractograms of the untreated and alkali-treated fibers are presented in Figure 4. From this figure, it is evident that the crystallinity of the fibers increased with the alkali treatment. This is understandable as the  $\alpha$ -crystalline content increased whereas the amorphous hemicellulose decreased with the alkali treatment. This is in agreement with the FTIR and chemical analyses.

The primary thermograms of the untreated and alkali-treated tamarind fibers are presented in Figure 5. From these thermograms, it is evident that the thermal stability of the alkali-treated fiber is only

TABLE II Chemical Analysis of Untreated and Alkali-Treated Tamarind Fibers

Component	Untreated	Alkali-treated
α-Cellulose	59.0%	64.5%
Hemicellulose	22.0%	16.0%
Lignin	19.0%	19.5%



Figure 4 X-ray diffractograms of untreated and alkalitreated tamarind fibers.

marginally higher than that of the untreated fiber. However, the residue at 600°C for the alkali-treated fiber is higher (25.3%) than that of the untreated fiber (12.9%).

The tensile properties of the untreated and alkalitreated fibers are presented in Table III. The standard deviation values in each case are also included in the same table. From this table, it is evident that within the experimental error, the maximum stress and elongation at break (%) before and after the alkali treatment of the fibers are almost the same. However, Young's modulus of the alkali-treated fibers is approximately 40% higher than that of the



Figure 5 Primary thermograms of untreated and alkalitreated tamarind fibers.

TABLE III Tensile Properties of Untreated and Alkali-Treated Tamarind Fibers

Parameter	Untreated	Alkali-treated
Maximum stress (MPa)	61.16	66.26
SD	5.19	7.03
Young's modulus (MPa)	2183.70	3085.69
SD	125.70	231.10
Elongation at break (%)	6.22	7.97
SD	1.19	1.08

SD = standard deviation.

untreated fibers. Hence, because of the higher values of the modulus (3085 MPa), renewable and environmentally friendly tamarind fibers can be considered as reinforcements in the preparation of green composites.

We (C.U.M. and B.R.G.) dedicate this article to B. Urmila Devi (wife of A.V.R.), who treated us as her own children and who unfortunately passed away recently.

#### References

- 1. Mock, J. A. Mater Eng 1979, 89, 60.
- Kulkarni, A. G.; Satyanarayana, K. G.; Rohatgi, P. K.; Vijayan, K. J Mater Sci 1983, 18, 2290.
- Mansur, M. A.; Aziz, M. A. Int J Chem Compos Light Weight Concr 1982, 4, 75.
- Beimares, H.; Berrera, A.; Castillo, E.; Veheugen, E.; Monjaras, M.; Patfoort, G. A.; Bucqueuye, M. E. N. Ind Eng Chem Prod Res Dev 1981, 20, 555.
- 5. Satyanarayana, K. G.; Kulakarni, A. G.; Rohatgi, P. K. J Sci Ind Res Dev 1981, 20, 222.
- Kulakarni, A. G.; Satyanarayana, K. G.; Sukumaran, K. G.; Rohatgi, P. K. J Mater Sci 1981, 16, 905.
- 7. Jinda, U. C. J Compos Mater 1986, 20, 19.
- Varada Rajulu, A.; Ramachandra Reddy, G.; Narasimha Chary, K. Ind J Fibre Text Res 1996, 21, 223.
- 9. Varada Rajulu, A.; Ramachandra Reddy, G.; Narasimha Chary, K. Ind J Fibre Text Res 1998, 23, 49.
- Varada Rajulu, A.; Allah Baksh, S.; Ramachandra Reddy, G.; Narasimha Chary, K. J Reinforced Plast Compos 1998, 17, 1507.
- Li, X. H.; Meng, Y. Z.; Wang, S. J.; Varada Rajulu, A.; Tjong, S. C. J Polym Sci Part B: Polym Phys 2004, 42, 666.
- 12. Varada Rajulu, A.; Rama Devi, R. J Reinforced Plast Compos 2007, 26, 629.
- Varada Rajulu, A.; Rama Devi, R. J Reinforced Plast Compos 2007, 26, 1657.
- Guduri, B. R.; Rajulu, A. V.; Luyt, A. S. J Appl Polym Sci 2006, 102, 1297.
- 15. Ray, D.; Sarkar, B. K. J Appl Polym Sci 2001, 80, 1013.