Properties of Ligno-Cellulose Fiber *Hildegardia*

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ABSTRACT: Studies on some properties such as the density, the degradation temperatures, the morphology and the spectral features of the ligno-cellulose fiber *Hildegardia* were carried out in both untreated and alkali treated form. The fibers are found to have good morphology and moderate initial and final degradation temperatures. On alkali treatment, the lignin was found to be eliminated. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 84: 2216–2221, 2002

Key words: *Hildegardia* fiber; thermal analysis; spectral analysis; morphology; properties

INTRODUCTION

As the use of the polymer products in general and the composites in particular is increasing day by day, the dangers they pose to the environment are also increasing. Most of the polymer composites have glass fiber as reinforcement. As glass fibers/fabric are nondegradable, the disposal of the composites containing them as reinforcement is a difficult problem. Moreover, the fibers are nonrenewable. At present, the trend is slowly changing toward using natural fibers as reinforcements. The composites made with natural fiber reinforcements are known as "Green composites." In this connection, some green composites were developed by several workers,[1–10] using sisal, banana, bamboo, coir, pineapple leaf fiber, and so forth. In the present article, the authors studied the properties of the natural fiber *Hildegardia*. *Hildegardia populifolia* (Roxb.) Schott & Endl. is a dry deciduous tree species belonging to family Sterculiaceae. The species was reported endemic to India and categorized as endangered in the Indian Red data Book.[11] The plant is available in sizeable numbers in Bukkapatnam and the Kadiri hills of Anantapur district, Andhra Pradesh, India. As data are not available on this fabric/fiber, the authors studied some of its properties such as density, thermal stability, spectral analysis, and morphology. The authors studied these properties for both untreated and alkali-treated fibers derived from the natural fabric.

MATERIALS AND METHODS

The *Hildegardia* fabric was extracted from the branches of its tree. The average length, breadth, and thickness of the fabric was found to be 300–700 cm, 70–100 cm, and 0.18 mm, respectively. The fabric was washed thoroughly with distilled water and allowed to dry in the sun for about one week. Some quantity of the fabric was treated with 2% aqueous NaOH solution to remove the lignin and other greasy material.

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The densities of both the untreated and alkali-treated fibers were determined using density columns maintained at 30°C. The diffuse reflectance infrared Fourier transform (DRIFT) spectrum was recorded using Perkin Elmer System2000 spectrophotometer (Norwalk, CT). The morphology of both the untreated and alkali-treated fabric/fiber was studied using a Hitachi S 530 scanning electron microscope (Tokyo, Japan). The samples were coated with gold using the electrodeposition technique to impart electrical conduction before recording the SEM. The transcrystallization in polypropylene by the *Hildegardia* fiber was observed using a Leica DMLP polarized optical microscope (POM) (Leica, Nussloch, Germany). In this method, the polypropylene granules were placed molten on glass slides and then the chopped *Hildegardia* fiber was mixed into the melt. The mixture was spread by placing the cover slips on it, and it was allowed to solidify slowly. The slide was directly placed in the microscope, and the pattern was recorded on a floppy. The thermogram for both the untreated and alkali-treated fibers was recorded using a Perkin Elmer TGA-7 thermobalance (Norwalk, CT) in N_2 atmosphere at a heating rate of 10°C/min.

RESULTS AND ANALYSIS

Density Measurements

The density of the untreated and alkali-treated *Hildegardia* fibers (separated from the fabric) was found to be 0.9 g/cc and 1.25 g./cc, respectively. The higher density of the treated fiber may be attributed to the removal of the lighter lignin.

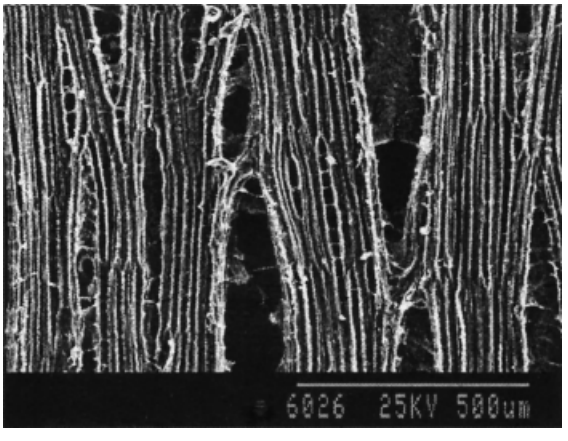
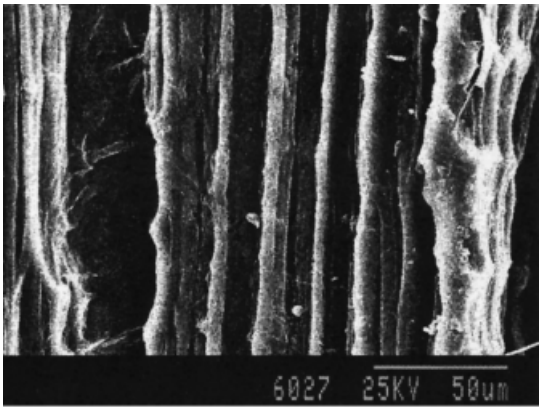
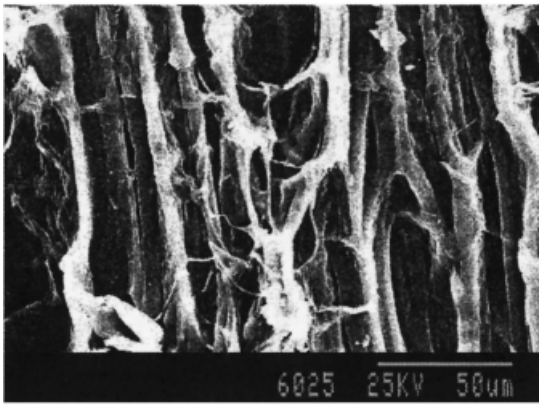


Figure 1 SEM of untreated *Hildegardia* fiber under low magnification.



(a)



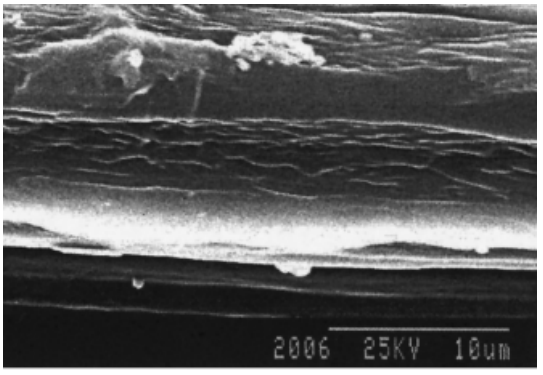
(b)

Figure 2 SEM of untreated *Hildegardia* fiber photographed at two different regions (a) and (b).

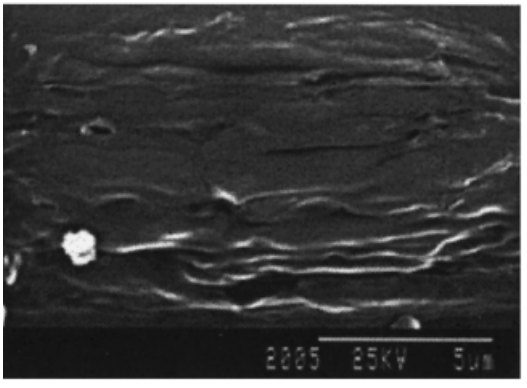
The soluble lignin was found to be 6.7%. The thickness of the untreated and treated single-layer fabrics was found to be 0.18 mm and 0.143 mm, respectively. The average diameter of a single fiber was found to be 0.0052 mm.

Morphology of the Fiber

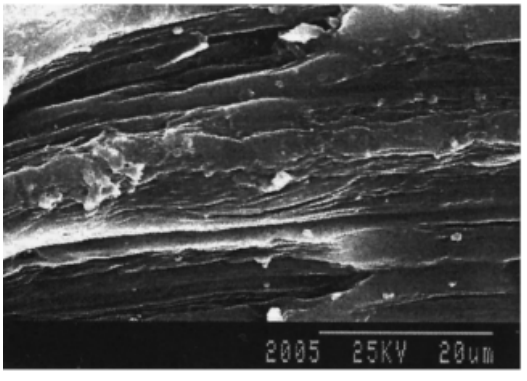
The SEM of the untreated fabric at low magnification is shown in Figure 1. From this microgram, it is clearly evident that the fabric is in the knitted form, with several void regions in between fibers. Such geometry allows the penetration of the resin in the void regions; as a result, the bonding between the matrix and the fabric is expected to be good. The SEM at higher magnification for untreated fabric is shown in Figure 2 (a) and (b), which is taken at two different places.



(a)



(b)



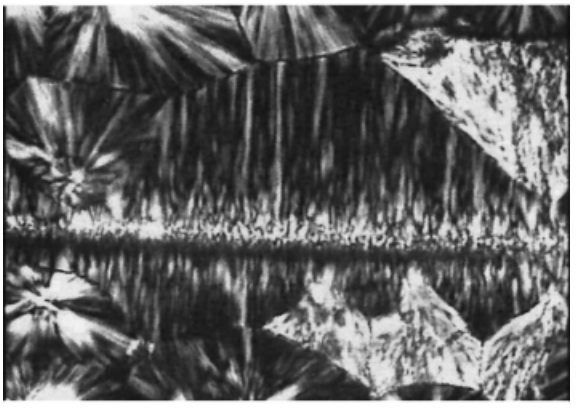
(c)

Figure 3 SEM of NaOH treated *Hildegardia* fiber at different magnifications (a), (b), and (c).

From this figure, the presence of lignin, the bonding material, can be seen as the cover on the individual fibers.

The SEM of alkali-treated fibers at different higher magnification is shown in Figure 3 (a–c). From this figure, it is clearly evident that the

(a)



(b)

(c)

Figure 4 Polarized optical micrograms of *Hildegardia* fiber reinforced in a polypropylene matrix, photographed at different regions (a), (b), and (c).

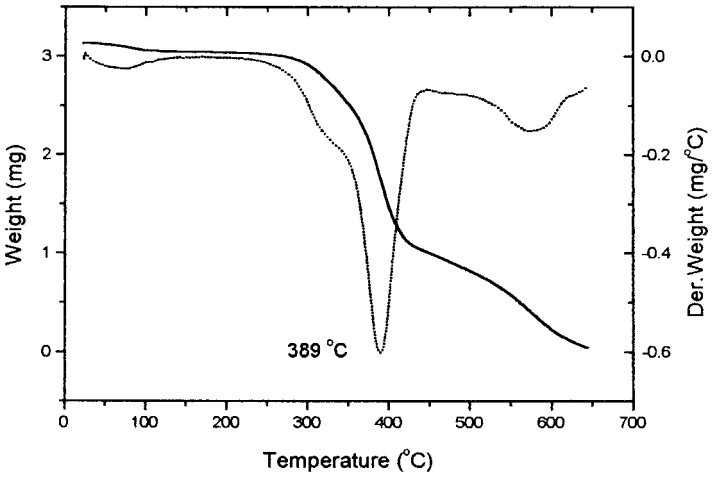


Figure 5 Thermogravimetric analysis and derivative thermogram of untreated *Hildegardia* fiber in N_2 atmosphere heated at the rate of 10°C/min.

fiber has a rough cellular structure. This type of structure might have been formed because of the removal of the lignin. Such an observation was also made by Narasimha Chary[12] and Ramachandra Reddy[13] in the case of bamboo fibers. They further reported good bonding between the matrix and fiber caused by the penetration of the liquid resin into these micropores by capillary action and the subsequent solidification by cross linking. Similar behavior is also expected in the present case, as the pore size is only a fraction of a micron, as seen in Figure 3 (c). All the electron micrograms indicate that the fiber has a favorable morphology for making the composites.

The POM microgram of the fiber embedded in polypropylene (PP) is shown in Figure 4 (a–c). From these pictures, in addition to the presence of spherulites of PP, transcrystallization perpendicular to the fiber axis is also observed. The *Hildegardia* fiber is inducing nucleation; as a result, transcrystallization is taking place. Similar observations were made in the case of polyester fiber reinforced in a polypropylene matrix[14].

Thermogravimetric Analysis

As thermoplastic composites are becoming popular at present, the authors studied the thermal stability of the fibers of *Hildegardia* to assess the possibility of their being used as reinforcement. The thermogravimetric analysis (TGA) thermogram for the untreated fiber is shown in Figure 5. In the same figure, the derivative thermogram is also presented. The initial degradation temperature T_i , the inflection temperature T_{in} (where the degradation rate is maximum), and the final degradation temperature T_f of the untreated fiber are presented in Table I. The integral procedural degradation temperature (ipdt) and the refractoriness T^* were determined using the primary thermogram as described elsewhere.[15] The T^* and ipdt values are also presented in Table I. The thermogram for the alkali-treated fabric is shown in Figure 6. The values of T_i, T_{in}, T_f, T_f^* and ipdt for the alkali-treated fiber are also presented in Table I for comparison. Using the table, it is clearly evident the thermal stability of the treated fiber is higher than that of the untreated one. This is understandable, as the untreated fiber has lignin, which degrades at lower temperature. The TGA analysis indicates that both the untreated and treated fibers are thermally stable at the flow temperatures of polyolefins and that, as such, these fibers or fabric can be effectively used as reinforcements.

Table I Initial Degradation, Final Degradation, Inflection Point, and Integral Procedural Degradation Temperatures (IPDT) and the Refractoriness T^* of *Hildegardia* Fiber

Parameter (°C)	Untreated	Treated
Initial Degradation Temperature	290	307
Final Degradation Temperature	412	421
Inflection Temperature	386	389
IPDT	218	231
T^*	313	326

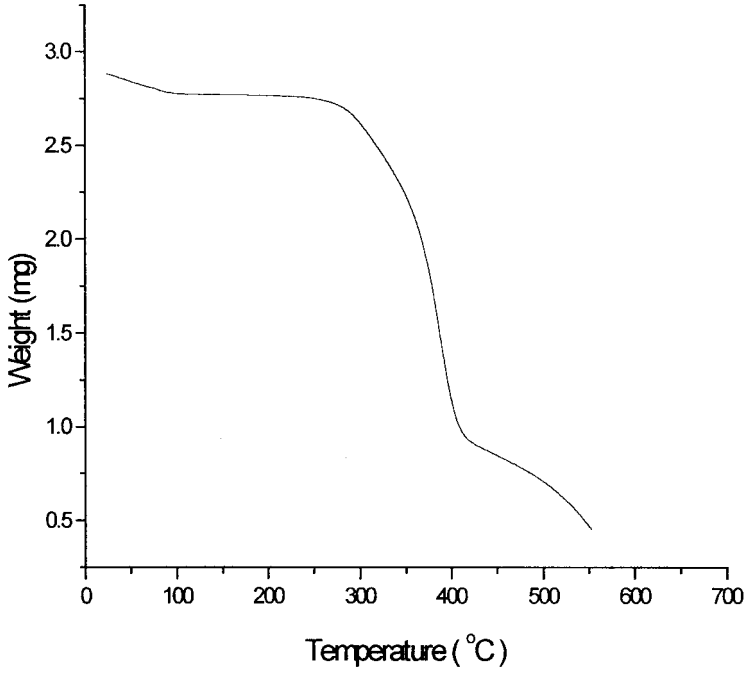


Figure 6 Thermogravimetric analysis thermogram of NaOH-treated *Hildegardia* fiber in N_2 atmosphere heated at the rate of 10°C/min.

Diffuse Reflectance Infrared Fourier Transform Spectrum of Fiber

The DRIFT spectrum of the untreated and alkali-treated *Hildegardia* fiber is shown in Figure 7. It shows the presence of hydroxyl, carbonyl, ether groups, and absorbed water. The assignment of bands is presented in Table II. It indicates that the fiber contains—apart from cellulose—some amount of lignin material. In the case of the DRIFT spectrum of alkali-treated fiber, some changes are observed. The peak at 1740 cm^{-1} (belonging to the carboxyl group of lignin) has completely disappeared, indicating the removal of lignin material. However, the intensity of the peak at 1640 cm^{-1} has slightly increased, indicting the absorption of more water on the surface of

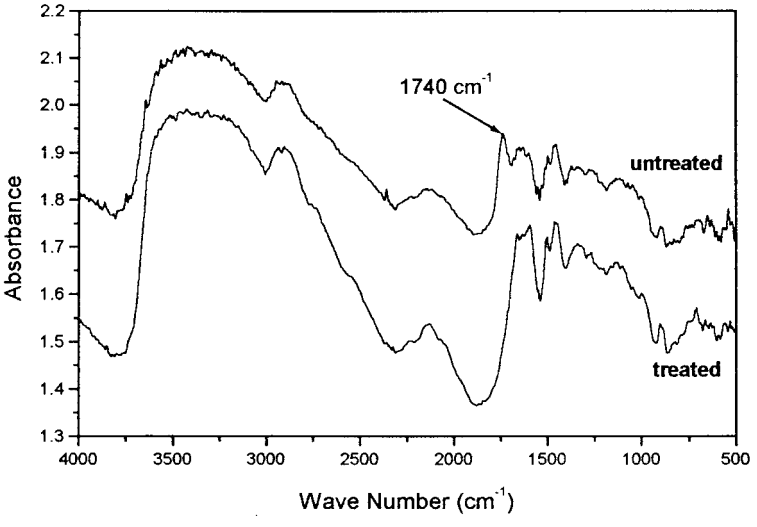


Figure 7 Diffuse reflectance infrared Fourier transform spectra of *Hildegardia* fiber with and without NaOH treatment.

Table II Peak Positions and Assignments of Chemical Groups in the Treated and Untreated *Hildegardia* Fiber

Wave Number (cm^{-1})	Assignments
3600	OH—Stretching
2900	CH—Stretching
1740	Carbonyl stretching
1640	Absorbed water
1450	CH—Bending
1230	C–O Stretching
1130	C–O–O Asymmetry vibration

the fiber. No significant changes are observed in the intensity or position of the other peaks in the spectra.

CONCLUSIONS

The properties of the natural fiber from *Hildegardia* fabric indicate that it can be favorably considered as a reinforcement in Green composites.

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