Effect of Alkali Treatment on Properties of the Lignocellulose Fabric *Hildegardia*

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ABSTRACT: The uniaxial natural fabric from the tree *Hildegardia populifolia* was treated with 5% aqueous NaOH solution for 0, 2, 4, 6, and 8 h, and the resulting changes were analyzed by density, Fourier infrared spectroscopic, X-ray, polarized microscopic, and scanning electron microscopic techniques. On alkali treatment, absorption of water and elimination of hemicellulose in the fabric were observed. In each case, the tensile strength, modulus, and percent elongation at break were determined. The tensile strength, modulus, and density were found to be maximum at 4 h of alkali treatment. Hemicellulose was found on the surface of the untreated fabric. After 6 and 8 h of alkali treatment, some microcracks developed in the fabric. The area of the void region in the fabric increased with the duration of alkali treatment. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 90: 1604–1608, 2003

Key words: lignocellulose fabric; *Hildegardia;* uniaxial fabric; alkali treatment; physical properties; mechanical properties

INTRODUCTION

Lignocellulose fibers such as jute, sisal, coir, banana, pineapple leaf, and bamboo are widely used as reinforcements in making "green" composites,[1–16] but, generally, these fibers are not available in a natural fabric form. Some of the above fibers can be woven in biaxial directions. Varada Rajulu et al.[17] studied the properties of the lignocellulose natural fabric *Hildegardia populifolia*. Recently, Babu Rao[18] developed *Hildegardia populifolia* fabric/polycarbonate-toughened epoxy composites and studied their performance. He reported that the mechanical properties of the matrix improved substantially on reinforcing it with this natural fabric. The fabric is available with the fibers in a uniaxial direction. In making green composites, it is customary to treat the natural fibers/fabric with alkali and other chemicals to improve their properties. Dipa Roy and Sarkar[19] characterized the physical and mechanical properties after alkali-treated jute fibers. They reported improvement in these properties after alkali treatment. Hill and Abdul Khalil[20] studied the effect of acetylation on the mechanical properties of coir- or oil palm fiber–reinforced polyester composites. They reported an increase in interfacial shear strength between the fiber and the matrix after acetylation treatment of coir. Mannan and Munir[21] characterized jute fibers treated with soap–glycerol micelles. They reported improvement of the mechanical properties after treatment. In the present work the authors treated *Hildegardia* fabric with 5% NaOH solution for various durations, and the resulting changes in density, tensile strength, modulus, and percent elongation at break were determined. The fabric before and after alkali treatment was analyzed employing Fourier transform infrared spectroscopic, X-ray diffraction, polarized optical microscopic, and scanning electron microscopic techniques. The authors selected *Hildegardia* fabric for study as it is naturally occurring with fibers in the uniaxial direction and is suitable[18] as reinforcement.

EXPERIMENTAL

The method for extracting the fabric from the *Hildegardia* tree is described elsewhere.[17] The fabric is present in the tree between the stem and the bark in a multilayer form. To protect the tree, the fabric was separated from the branches only. The extracted fabric was dried in the sun for 7 days, and the layers of the fabric were then separated. The single-layer samples were then treated with 5% aqueous NaOH solution at 30°C for 0, 2, 4, 6, and 8 h, maintaining a fabric-to-liquor ratio of 1:15. These samples were thoroughly

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TABLE I
Thickness and Density of Untreated and Alkali-Treated *Hildegardia* Fabric

Duration of alkali treatment (Hs)	Thickness (mm)	Density (Kg/m^3)
0	0.127	1419
2	0.106	1422
4	0.102	1462
6	0.114	1445
8	0.115	1449

washed with distilled water and later dried in an open atmosphere for several days to remove the water. The fabric was further dried in a vacuum oven maintained at 110°C for 4 h. The dried samples were stored in a vacuum desiccator until they were put to use. Every care was taken to remove water before testing the fabric.

The thickness of the fabric was measured using a Mitutoyo dial-type thickness gauge. The thickness in different regions of the fabric was measured with the gauge length and the average value reported. The density of the fibers in the untreated and alkali-treated fabrics was determined with a Mettler Toledo AG104 electric balance at 25°C, using ethyl alcohol as the reference liquid. As the fabric has void regions, the density for single fibers separated from the fabric was determined. This instruments works on the principle of floatation and displays the density directly. The tensile strength, modulus, and percent elongation at break were determined employing a universal testing machine (SANS CMT 6503, China). A gauge length of 25 mm was maintained for all samples. The test was conducted at a crosshead speed of 10 mm/min. In each case, 10 samples were used, with the average value reported. The presence of void regions makes it difficult to evaluate the exact area of the fibers; hence, the total area of the fibers and the voids was used for the determination of tensile strength. In view of this assumption, the tensile strength values reported are approximate only.

The infrared spectra of the samples were recorded using an Analect RFX-65A FTIR spectrophotometer. To make the pellets, a 2-mg sample of the dried powered fabric was mixed with 100 mg of KBr. The X-ray spectra of the samples were recorded using a Rigaku D/max–100X instrument. The polarized optical micrograms were taken using a Leitz Vario-Orthomat 2 microscope. The scanning electron micrograms of the gold-coated samples were recorded using a JEOL JSM 820 microscope.

RESULTS AND ANALYSIS

The thickness and density of the *Hildegardia* fabric before and after alkali treatment are presented in Table I, from which it is clearly evident that the thickness decreased with alkali treatment up to 4 h and then increased for 6 and 8 h of treatment. In contrast, the fabric density increased up to 4 h after which it decreased. Two factors may be responsible for such behavior: first, removal of alkali soluble components of the fabric, resulting in a decrease in thickness; and, second, an increase in thickness from water absorption by the fabric. The Table I data indicate that both the removal of the soluble components and the absorption of water are taking place simultaneously upon alkali treatment, the latter increasing with duration. The results of a study by Rajulu et al.[17] of thermal degradation of both untreated and alkali-treated *Hildegardia* fabric found that weight losses of 0.6% and 1.0% at 100°C corresponded to water uptake by the untreated and alkali-treated fabrics, respectively.

The tensile strength, modulus, and percent elongation at break of the untreated and alkali-treated *Hildegardia* fabrics are presented in Table II, from which it is evident that tensile strength and modulus were increasing with duration up to 4 h, after which they decreased. The variation in these parameters is similar to that of density with duration with the alkali treatment. However, elongation at break decreasing with duration indicates a decrease in elasticity with the alkali treatment. These studies indicate that the mechanical properties are optimum when the alkali treatment is carried out for 4 h.

The infrared spectra of *Hildegardia* for 0, 4, and 8 h of alkali treatment are presented in Figure 1. The peak positions of the absorption bands, corresponding to the vibrating groups giving rise to the most probable absorption bands, are presented in Table III. From Figure 1 it is evident that there was a visual change in the peaks at 3434, 1704, 1633, and 1024 cm^{-1}. The intensity of the band around 3434 cm^{-1} increased with duration of alkali treatment, indicating an increase in the OH content of the fabric. This may be because of an increase in water absorption on the fabric surface. This is further indicated by the increase in intensity of the peak at 1633 cm^{-1}, which corresponds to the absorbed water.[19,21,22] Moreover, the intensity of the

TABLE II
Tensile Strength, Modulus, and Percent Elongation at Break of Untreated and Alkali-Treated *Hildegardia* Fabric

Duration of alkali treatment (Hs)	Tensile strength (MPa)	Modulus (GPa)	Elongation at break (%)
0	80.1	2.69	3.52
2	95.7	3.01	3.51
4	101.0	3.38	3.32
6	68.8	2.87	2.73
8	66.6	2.81	2.56

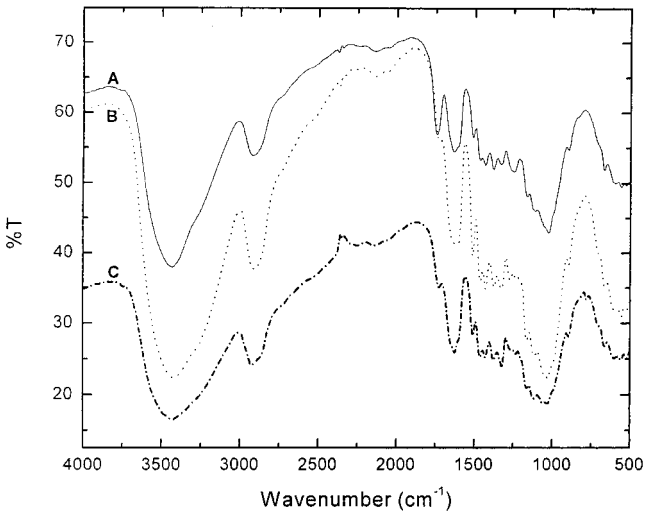

Figure 1 Infrared spectra of *Hildegardia*: (a) untreated; (b) alkali treated for 4 h; (c) alkali treated for 8 h.

peak around 1024 cm^{-1}, corresponding to C—OH stretching, also increased as the alkali treatment lasted longer, confirming an increase in water content with duration of alkali treatment. Though the fabric was dried before recording the infrared spectra, the presence of water is still indicated in the spectra. This, in all probability, corresponds to the bound water attached to the cellulose units, which cannot be removed easily by drying.

The peak around 1740 cm^{-1}, corresponding to the C═O stretching of hemicellulose in the untreated fabric, is almost missing in the spectrum of alkali-treated *Hildegardia* fabric. This indicates the elimination of hemicellulose after treatment with alkali. There was no change in the intensity of the peak around 1508 cm^{-1}, a result of the aromatic skeletal ring vibration of the lignin.[19,20] This clearly indicates that no structural change in the lignin of the *Hildegardia* fabric occurred after the alkali treatment.

The polarized optical micrograms (POM) of the *Hildegardia* fabric before and after alkali treatment are presented in Figure 2. The POM image of the untreated fabric was found to be diffuse. This may be a result of the presence of an amorphous hemicellulose layer on the surface of the fabric. However, the microgram images of the alkali-treated fabric were found to be sharp. This may be because of the elimination of hemicellulose upon alkali treatment (as shown by the infrared spectral analysis). The optical micrograms corresponding to 6- and 8-h alkali treatments indicate the formation of microcracks on the surface of the fabric, as well as an increase in the thickness of the fabric. These two observations indicate the total removal of hemicellulose from the fabric after 6 and 8 h

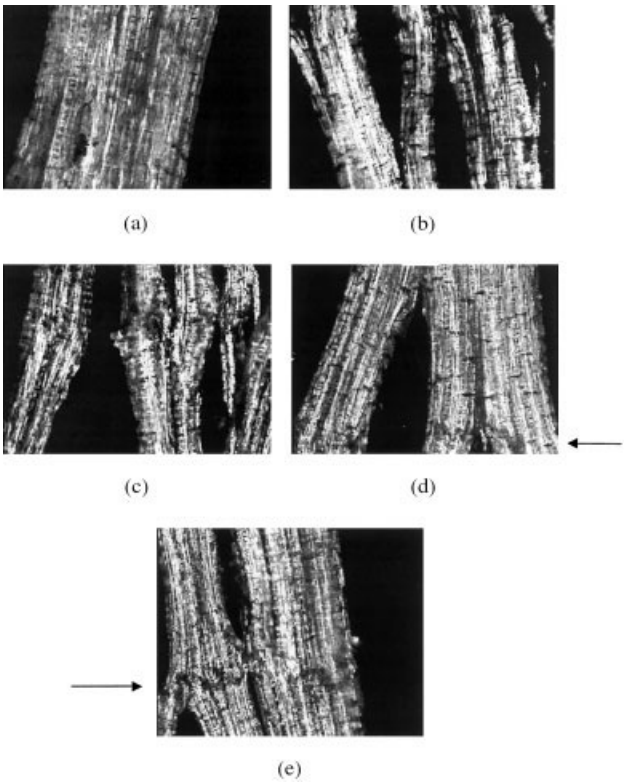

Figure 2 Polarized optical micrograms of *Hildegardia*: (a) untreated; (b) alkali treated for 2 h; (c) alkali treated for 4 h; (d) alkali treated for 6 h; (e) alkali treated for 8 h (microcracks are shown with external arrows).

TABLE III
FTIR Spectral Data of Untreated and Alkali-Treated *Hildegardia* Fabric

Absorption (cm^{-1})					
Duration of alkali treatment (Hs)					
0	2	4	6	8	Possible Assignment
3434	3427	3425	3433	3429	O–H stretching
2920	2912	2904	2923	2923	C–H vibration
1740	—	—	—	—	C=O stretching of Hemicellulose
1633	1601	1602	1630	1626	Absorbed water
1508	1508	1510	1510	1508	Aromatic skeletal ring vibration due to lignin
1429	1427	1427	1429	1429	CH_2 symmetric bending
1377	1375	1375	1375	1375	CH bending (deformation)
1024	1028	1028	1028	1028	C=OH stretching

of alkali treatment. The tensile properties also decreased after 6 and 8 h of alkali treatment (Table II). The loss of hemicellulose resulted in the loss of polar groups such as COOH or the conversion of polar groups such as COOH and OH, possibly to COO Na and O Na, thereby repelling the fibers from each other in the fabric. However, the surface of the alkali-treated fabric was found to be rough (Fig. 2) when compared to that of the untreated one. This suggests that better bonding between the alkali-treated fabric and the matrix is possible if this fabric is used as reinforcement in composites. Babu Rao[18] examined the fractured surface of *Hildegardia populifolia*/polycarbonate-toughened epoxy composites and reported an improvement in bonding between the reinforcement and the matrix on alkali treatment.

The scanning electron micrograms of the untreated and alkali-treated *Hildegardia* are shown in Figure 3. The micrograph of the untreated fabric shows a white covering layer on the surface of the *Hildegardia*, which may be a result of the hemicellulose. This layer is absent in the micrograms of the alkali-treated fabric, confirming the elimination of hemicellulose. These micrograms also reveal the uniaxial orientation of the fibers in a parallel direction. The fibers also had many

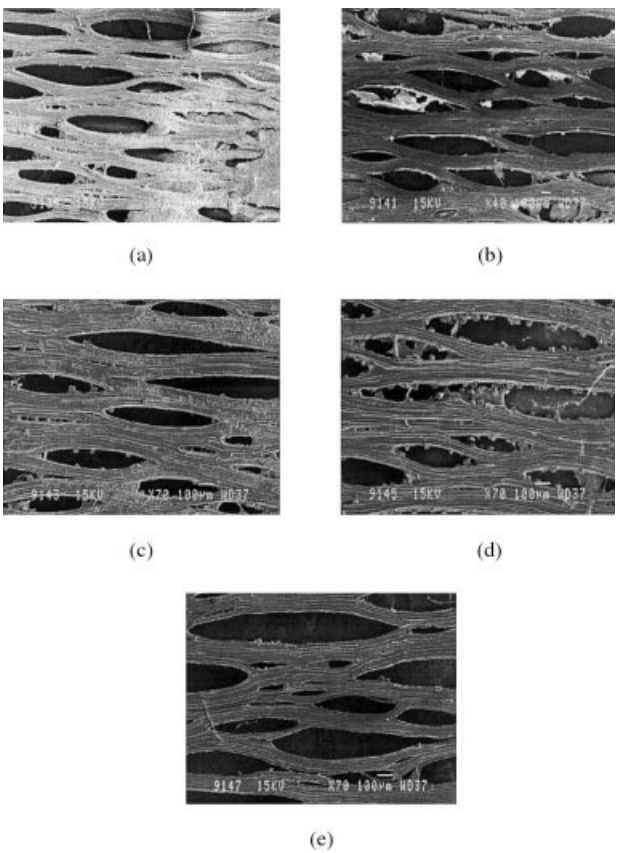

Figure 3 Scanning electron micrograms of *Hildegardia*: (a) untreated; (b) alkali treated for 2 h; (c) alkali treated for 4 h; (d) alkali treated for 6 h; (e) alkali treated for 8 h.

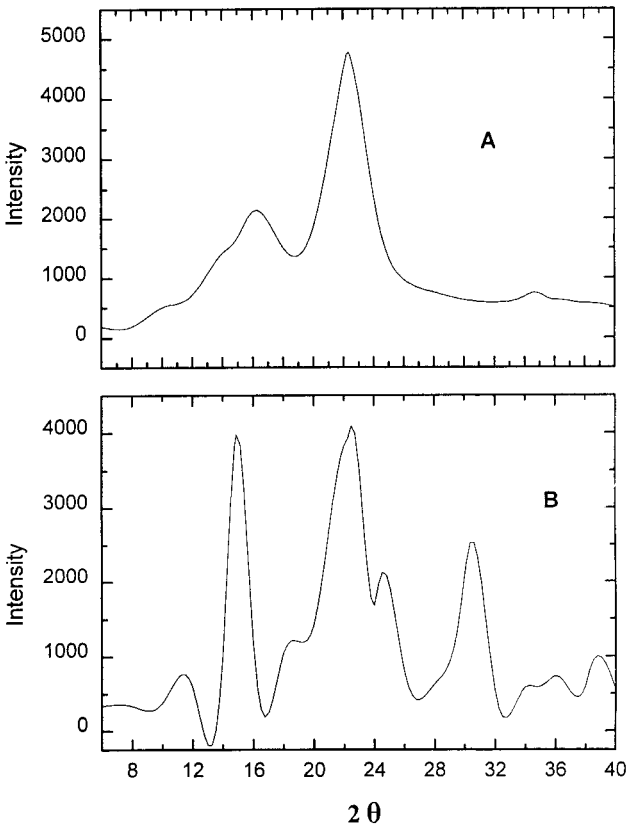

Figure 4 X–ray difractograms of *Hildegardia*: (a) untreated; (b) alkali treated for 8 h.

void regions. In the 8-h alkali-treated fabric the void regions were larger, indicating a complete loss of hemicellulose.

The X-ray diffraction spectra of the untreated and alkali-treated fabric are shown in Figure 4. Up to 6 h of treatment the spectra of the alkali-treated fabric did not show any change and hence are not shown in Figure 4, from which it is clearly evident that the fabric became more crystalline only after an alkali treatment of 8 h. Dipa Roy and Sarkar[19] made a similar observation for alkali-treated jute fiber.

CONCLUSIONS

The density, tensile strength, and modulus were found to be at a maximum when the *Hildegardia* fabric was treated with 5% NaOH solution for 4 h. The percent elongation at break marginally decreased with duration of alkali treatment, indicating a slight loss in elasticity. The infrared spectra revealed a loss of hemicellulose on alkali treatment. This was further confirmed by polarized optical and scanning electron microscopic techniques. Infrared spectral analysis showed an increase in bound water absorption because of the alkali treatment. X-ray difractograms showed an increase in crystallinity only after 8 h of

alkali treatment. The surface of the fibers in the fabric became rough with alkali treatment.

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