Characterization of Alkali-Treated Jute Fibers for Physical and Mechanical Properties

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Received 6 October 1999; accepted 15 May 2000

ABSTRACT: Changes occurring in jute fibers when treated with a 5% concentration of a NaOH solution for 0, 2, 4, 6, and 8 h were characterized by weight loss, linear density, tenacity, modulus, FTIR, and X-ray measurements. A 9.63% weight loss was measured during 2 h of treatment with a drop of hemicellulose content from 22 to 12.90%. The linear density value showed no change until 2 h of treatment followed by a decrease from 33.0 to 14.5 denier by 56% after 6 h of treatment. The tenacity and modulus of the fibers improved by 45 and 79%, respectively, and the percent breaking strain was reduced by 23% after 8 h of treatment. X-ray diffractograms showed increase in crystallinity of the fibers only after 6 h of treatment with an increased amount of OH groups. By measuring the rate of change of the modulus, tenacity, and percent breaking strain with the time of treatment, a clear transition was apparent at 4 h of treatment with the dissolution of hemicellulose, causing a weight loss and drop in the linear density before and development of crystallinity with an improvement in the properties after the transition time. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 80: 1013–1020, 2001

Key words: jute fiber; alkali treatment; denier; mechanical properties; FTIR

INTRODUCTION

Efforts to exploit the possible use of lignocellulosic fibers like jute, sisal, coir, banana, and $PALF^{1-15}$ as reinforcing composites have been extensive in recent years. Among these, jute fiber is most common because of its easy availability at low cost, especially in the eastern regions of India. For jute fiber to be an effective reinforcing constituent, it is essential that the fiber and the resin matrix have a good compatibility and bonding. To make them suitable reinforcing candidates with adequate bond characteristics for general appli-

Journal of Applied Polymer Science, Vol. 80, 1013–1020 (2001) © 2001 John Wiley & Sons, Inc.

cations, various chemical modifications of jute fibers have been attempted. Among the many surface treatments undertaken, the most economically viable one is alkali treatment.

As early as in 1935, Sarkar¹⁶ treated jute fibers with a cold and dilute NaOH solution (1 and 8%) for 48 h and found a 130% improvement in the tensile strengths. Samal et al.¹⁷ chemically modified the jute fibers through alkali treatment, crosslinking with formaldehyde, *p*-phenylenediamine, and phthalic anhydride, and combined crosslinking–cyanoethylation reactions to transform jute into a strong reinforcing candidate material. He reported a 13 and 8% increase in tenacity and modulus, respectively, after treating the jute fiber with a 2% NaOH solution for 1 h at 35°C.

Roy,¹⁸ on the contrary, showed a steady decrease in the tenacity of the fibers with an in-

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Contract grant sponsor: Council of Scientific & Industrial Research, Government of India.

crease in the alkali concentration when the fibers were treated with a 1–18% NaOH solution for 2 h at room temperature. Fibers treated with a 17.5% solution had only 44% of their original strength. Similar observations were made by Macmillan et al.¹⁹

Coir fibers, when alkali-treated with a 5% concentration for 72–96 h, showed improvement in tensile strengths by 10–15% and modulus by 40%.¹² Treatment of coir fiber with alkali by Samal et al.,²⁰ however, resulted in 24, 19, and 17% increases in tensile strengths when NaOH concentrations were increased by 2, 8, and 10%, respectively. A decrease in strength with increased concentration was demonstrated. In the case of PALF, the tensile strength was improved by 66% when treated with a 4% NaOH solution for 1 h.²¹

Conflicting views have, therefore, emerged responding to the effects of alkali treatment on coir and jute fibers. The effects are more pronounced with a longer duration of treatment and concentration. The effects on properties due to structural changes have been reported.^{18,19,22–24} Yet, any detailed explanation is scanty, particularly on treatment with a low NaOH concentration on jute fibers.

An attempt was made, therefore, to investigate the changes occurring in jute fibers after a 5% NaOH solution treatment for different time periods for 0, 2, 4, 6, and 8 h. The fibers were characterized using chemical analysis, FTIR, and SEM and strength properties were determined so that their ability as a reinforcing composite constituent could be ascertained.

EXPERIMENTAL

Materials

Jute fiber of grade W-2, *Corchorus capsularis*, from the Indian Jute Industries' Research Association (IJIRA) was used for the study. The fibers were combed thoroughly to unwind the entangled pieces and cleaned of foreign elements.

Alkali Treatment

Jute fibers were combed, cut into 50 cm of length, and soaked in a 5% NaOH solution at 30°C, maintaining a liquor ratio of 15 : 1. The fibers were kept immersed in the alkali solution for 2, 4, 6, and 8 h. The fibers were then washed with fresh water several times to remove any traces of NaOH sticking to the fiber surface, neutralized with dilute acetic acid, and again washed with distilled water. Final pH maintained was 7.

Testings

The fibers, after different soaking times in a 5% alkali solution, were tested for their weight change. The loss in weight was calculated by weighing a fixed amount of dry and cleaned fiber (W_1) . The amount of fiber was then weighed again after soaking in an alkali solution (W_2) . The percent weight loss was calculated as

% Weight loss = $(W_1 - W_2)/W_1 \times 100$

It was observed that the fibers were somewhat leached and were finer. Fiber fineness was determined in terms of linear density by the gravimetric method. The linear density was obtained as denier from the weight of 100 single fibers of 60 mm length each.

Chemical analysis of untreated and treated fibers was made to ascertain the cause for the loss in weight. The major constituents (α -cellulose, hemicellulose, and lignin) of the untreated and the alkali-treated jute fiber samples were determined following the standard procedures.^{25–27}

Loss of cellulosic constituents by NaOH treatment improving the crystallinity of the fibres were reported by several authors.^{18,28–31} Hence, the extent of crystallinity formation by NaOH treatment was determined by X-ray diffraction with a Philips PW 1710 X-ray diffractometer on 101, $10\overline{1}$, and 002 planes.

Infrared spectra of untreated and alkalitreated jute fibers were recorded with KBr pellets in a Nicolet Magna-IR 750 spectrometer. Surface morphologies of the fibers were examined by SEM in a LEO S-440, using a voltage of 15 kV.

Tensile tests of single jute fibers were carried out using an Instron 1195. A gauge length of 20 mm was employed with a crosshead speed of 10 mm/min in accordance with ASTM D 3822-91. Fifty single fibers were tested after each treatment and the mean value was reported.

RESULTS AND DISCUSSION

The loss in weight of the jute fibers after alkali treatment is shown in Figure 1. There was a 9.63% loss in weight within 2 h of treatment. The loss with time of treatment thereafter was to a



Figure 1 Loss of weight of the constituents during alkali treatment of jute fibers.

maximum of 10.45%. This is in agreement with the observations made by Macmillan et al.¹⁹

Chemical analysis showed (Table I and Fig. 1) that the losses have been due to the dissolution of hemicellulose reacting first with the NaOH solution. The hemicellulose content of the jute fibers was reduced from 22 to 12.90% by about 41% with treatment time, in agreement with Ganguly and Chanda.³² α -Cellulose and lignin remained unchanged by the alkali treatment.

The change in linear density with time after alkali treatment is shown in Figure 2. The linear density showed no change until 2 h of treatment, followed by a decrease from 33.0 to 14.5 denier by 56% after 6 h of treatment. Progressive destruction of the mesh network and splitting of fibers into finer filaments occurred simultaneously during 2–6 h of treatment. The dissolution of cellulosic constituents in alkali created voids in the fiber structure and swelling was observed to have occurred. The strands of fibers became well separated. The enlargement in volume with loss in weight caused its linear density to fall sharply.

Crystallinity of the fibers was observed to have increased only after 6 h of treatment, shown in



Figure 2 Change of linear density with alkali treatment times of jute fibers.

Figure 3. By 8 h of treatment, the I_{002} peak height improved by 23.4% from the untreated fiber.

The improvements in the mechanical properties of the fibers with alkali is shown in Table II. The tenacity of the fibers showed a drop after an initial 2 h of treatment, followed by progressive improvement from 3.24 to 4.7 g/den, as shown in Figure 4, and so did the fiber modulus, having 309.1 g/den for untreated fibers to 553.5 g/den for 8 h of treatment, an increase of 79%. The percent strain at the breakpoint of the fibers (see Fig. 4), however, showed a decrease from 1.16 to 0.89 for 8-h treated fibers, indicative of the fibers becoming more brittle in accordance with the crystallinity of the fibers.

It was interesting to note that most of the changes occurred within 2–4 h of alkali treatment; the maximum was at 4 h of treatment. Thereafter, the change was nominal. This is shown more prominently in the rate of the change

Sample Type	Weight Loss (%)	α -Cellulose (%)	Hemicellulose (%)	Lignin (%)
Untreated	0.00	63.00	22.00	13.70
2-h alkali-treated	9.63	62.70	12.90	13.48
4-h alkali-treated	10.06	62.60	12.65	13.40
6-h alkali-treated	10.43	62.52	12.40	13.35
8-h alkali-treated	10.45	62.50	12.40	13.35

Table I Chemical Compositions of Raw Jute and Alkali-Treated Jute Fibers

^a All figures are expressed on 100 g of untreated oven-dry jute.



Figure 3 X-ray diffractogram of raw and 8-h alkalitreated jute fibers.

of tenacity, modulus, and percent strain with time of treatment, as shown in Figure 5. A change in the mechanism having different slopes before and after 4 h of treatment was indicated. The change in tenacity and percent strain were more severe. The slope of the rate of change in percent strain to 4 h of treatment was steeper than that of others, the change having continued after 4 h, while the modulus and tenacity slowed down. The dissolution of hemicellulose was predominant to 4 h of treatment, followed by the development of crystallinity at a slower rate between 4 and 8 h of treatment. The heavy loss of hemicellulose as a cementing constituent in the first region to 4 h of treatment contributed to the closer packing of the cellulose chains, leaving the fibrils with the freedom for reorientation along the direction of the tensile force.²⁴ Beyond 4 h of treatment is the



Figure 4 Effect of alkali treatment on the tenacity, modulus, and percent breaking strain of jute fibers with varying treatment times.

region where crystallinity had been improved with further closer packing of the cellulose chains and molecular reorientation, as was seen for coir^{28,29} and flax.³⁰

The improvement in the mechanical properties could be attributed to the reduction of linear density of the fiber after treatment. The large loss of hemicellulose made the fibers lose their cementing capacity and to separate out from each other, making them finer, as seen from the SEM photomicrographs. Figure 6(a) shows the multicellular nature of jute fiber. A smooth surface of the raw jute fiber is clearly visible. The surface features of 2-h alkali-treated fiber is shown in Figure 6(b). Here, the cementing material from the multicellular matrix was removed to some extent and the individual cells became more prominent. In 4-h alkali-treated fiber [Fig. 6(c)], the cementing materials were removed extensively, leading to better separation of the individual cells, causing thinner fibers. The fibrillar structure is clearly visible.

Table II Mechanical Properties of Untreated and Alkali-Treated Jute Fibers

Type of Fiber	Tenacity (g/den) S.D.ª		% Strain at Breakpoint	S.D.ª	Modulus .ª (g/den) S.D.ª		
Untreated	3.246	1.1	1.1650	0.40	309.10	65	
2-h alkali-treated	2.750	1.1	0.9797	0.35	308.80	80	
4-h alkali-treated	3.132	1.2	0.9491	0.30	347.50	130	
6-h alkali-treated	4.503	1.6	0.9609	0.30	520.30	160	
8-h alkali-treated	4.705	1.6	0.8974	0.30	553.50	144	

^a S.D., standard deviation.



Figure 5 Variation of the rate of change of tenacity, modulus, and percent breaking strain with alkali-treatment times.

The observations were further substantiated by FTIR studies, shown in Figure 7. The exact position and the probable assignments of the peaks are given in Table III.

In raw jute fiber, the peak in the area of $3100-3800 \text{ cm}^{-1}$ arose due to O—H stretching vibrations. The peaks at 2970.8 and 2923.9 cm⁻¹ are due to the C—H stretching vibration in cellulose and hemicellulose. Two sharp peaks are evident at 1735 and 1715 cm⁻¹ due to the C—O stretching vibration of carboxylic acid and ester groups of hemicellulose. A shoulder and a medium sharp peak arose at 1650 and 1630 cm⁻¹ due to absorbed water. The peaks in the region 1440 to 1530 cm⁻¹ are due to various lignin components. The peak at 890 cm⁻¹ is due to β -glucosidic linkage. The other peaks are due to various characteristics of specific groups as indicated in Table III.^{17,33,34}

It was observed that maximum changes occurred within 2 h of treatment. The peak area at 3100-3800 cm⁻¹ increased considerably, indicating the presence of an increased number of OH groups arising due to the cleavage of alkali-sensitive bonds. The sharp peak at 2970.81 cm^{-1} in raw jute fiber became a little shoulder in 2-h alkali-treated fibers and the peak at 2923.9 cm^{-1} shifted to lower values at 2919.2 cm⁻¹. Removal of hemicellulose caused these changes in the peak positions. The peak intensities at 1735 and 1715 cm^{-1} were considerably reduced in the 2-h treated fibers as some amount of C=O groups were killed by the removal of hemicellulose. The little shoulder at 1630 cm^{-1} in raw jute due to absorbed water became more prominent in the







Figure 6 SEM surface studies of jute fibers: (a) raw jute; (b) 2-h alkali-treated; (c) 4-h alkali-treated.



Figure 7 FTIR spectra of (a) raw jute fibers and (b) 2-h alkali-treated jute fibers.

2-h alkali-treated fibers, indicating a higher amount of absorbed water. The peaks in the region 1440–1530 cm^{-1} remained unchanged, indicating no change of the lignin component. A small sharp peak at 890 cm⁻¹ due to β -glucosidic linkage in raw jute underwent a shift to higher wavenumbers in the 2-h alkali-treated fibers. This might be due to the rotation of the glucose residue around the glucosidic bond, indicating transformation of the raw jute to the chemically modified one. A weak shoulder at 675 cm⁻¹ in raw jute due to an out-of-place bending vibration of an intermolecular H-bonded OH group shifted to lower wavenumbers. From 4-8 h, the changes in the FTIR spectra of the alkali-treated fibers were relatively small.

Jute is a multicellular fiber. Each unit cell of jute fiber is composed of small particles of cellulose surrounded and cemented together with lignin and hemicellulose. Although the length of the individual cell is very small, they are attached with one another in the longitudinal direction, thereby producing a long, continuous fiber. These filaments of the fibers are not entirely separated from those of the neighboring units, but there exists several loose attachments among them, giving rise to a meshlike structure. Alkali treatment tends to react with the cementing material hemicellulose, in particular, leading to destruction of the mesh structure and splitting of the fibers into finer filaments. Splitting of the cemented fibers causes a reduction in the fiber diameter. This fiber fibrillation, which is the breaking down the composite fiber bundle into smaller ones, increases the effective surface area available for wetting by the resin. Thus, bonding between the fiber and the matrix at the interface was improved.

On alkali treatment, the number of free hydroxyl groups on the surface of the jute fibers increased, thus providing a greater number of active sites on the fiber surface. An interesting observation was reported by Sarkar et al.³⁵ in 1947: In an attempt to extract the acid constituents of jute fibers by a cold and dilute NaOH solution, they observed that the acid value of the alkali-treated jute fibers were doubled. Hydrolysis of alkali-sensitive bonds produced a higher number of free hydroxyl groups and enhanced the accessibility of hydroxyl groups on the surface of the fibers, thereby increasing the chemical reactivity.

The strength of the jute fibers also increased on alkali treatment. In untreated jute fibers, hemicellulose remains dispersed in the interfibrillar region separating the cellulose chains from one another. The cellulose chains are therefore always in a state of strain. 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 a close packing of the cellulose chains takes place, leading to a higher strength of the fiber.

CONCLUSIONS

The alkali treatment of jute fibers with a 5% NaOH solution showed that most of the changes occurred within 2-4 h of treatment. The weight loss due to dissolution of the hemicellulose was maximum at these treatment hours, leaving heavy voids in the fiber structure and separating the fibers from the strands. Thus, a sharp drop in the linear density was measured from 33.0 to 14.5 denier after 6 h of treatment. The loss of hemicellulose led to closer packing of its cellulose chains. FTIR measurements also confirmed the open structure of the fiber having a larger absorption of water as the dissolution of hemicellulose took place in the fiber structure. On further treatment with alkali from 4-8 h, the crystallinity of the fibers was found to have improved sharply to a limit. The tenacity and modulus of the fibers improved by 45 and 79%, respectively, and the percent breaking strain was decreased by 23% after 8 h of treatment, which is indicative of the fiber becoming stiffer and more brittle in accordance

Absorbance in cm^{-1}							
Raw Jute	Alkali-treated 2 h	Alkali-treated 4 h	Alkali-treated 6 h	Alkali-treated 8 h	Possible Assignments		
3100–3800	3100–3800	3100–3800	3100–3800	3100–3800	O—H stretching		
2970.8 2923.9	2919.2	2914.0	2919.6	2915.7	C—H stretching vibration in cellulose and hemicellulose		
1735	1735	1735	1735	1735	C=O stretching of		
1715					carboxylic acid or ester		
$1650.0 \\ 1630.3$	$1660.0 \\ 1629.6$	$1660.0 \\ 1630.0$	$1662.1 \\ 1629.7$	$1663.0 \\ 1629.7$	Absorbed water		
1600.0	1600.4	1603.5	1604.2	1599.0	Aromatic C=C stretching		
1465-1530	1465 - 1530	1465 - 1530	1465 - 1530	1465 - 1530	Lignin component		
1440.3	1439.1	1438.2	1439.9	1439.6	$\rm CH_2$ bending in lignin		
1410	1411	1411	1411	1411	CH_2 and CH_3 bending		
1380.0	1379.1	1377.2	1377.0	1377.0	C—H bending		
1326–1358	1326–1358	1326-1358	1326-1358	1326–1358	O—H in-plane bending		
1253.1	1250	1250	1250	1250	C—O stretching of acetyl (lignin)		
1160	1160	1160	1160	1160	Antisymmetric bridge C—O—C stretching		
1047.8	1048.5	1052.0	1051.8	1053.2	C—O/C—C stretching vibration		
890	896	896	896	896	β-Glucosidic linkage		
675.0	669.6	669.1	669.6	669.9	Out-of-place bending vibration of intermolecular H- bonded O—H group		
610.0	607.7	609.4	608.1	609.8	Out-of-plane O—H bending		
561.2	559.4	561.1	561.0	561.0	Torsional vibration of pyranose ring		

Table III FTIR Spectral Data of Jute and Alkali-Treated Jute Fibers

with the increase in the crystallinity of the fibers. The rate of change of the tenacity, modulus, and breaking strain showed the possibility of two different mechanisms before and after 4 h of treatment. The loss of hemicellulose as a cementing material in the first region of up to 4 h of treatment contributed to the closer packing of the cellulose chains and the second region beyond 4 h of treatment showed a slow improvement in crystallinity, with further closer packing of the cellulose chains, making the fibers stiffer and stronger. A transition with a predominant chemical reactivity before 4 h of treatment and a gradual development of crystallinity thereafter was apparent.

The authors are indebted to the Council of Scientific & Industrial Research, Government of India, for providing financial assistance during the course of the investigation. Sincere thanks are due to Dr. N. R. Bose, Mr. K. Banerjee, and Dr. A. Chakrabarty of Central Glass & Ceramic Research Institute for their guidance. Mr. A. K. Rana, Mrs. M. Sarkar, Mr. A. Banerjee, Dr. T. K. Guha Roy, Dr. A. K. Mukhopadhyay, Mr. A. Saha, Mr. N. G. Saha, Dr. (Mrs.) P. Debnath, Mr. S. Kundu, and Dr. A. C. Mukherjee of IJIRA are gratefully acknowledged for their support and advice in carrying out the experiments in their laboratories. The authors are grateful to Dr. C. Chakrabarty, Mr. S. Shome, and Mr. U. S. Kundu of the Geological Survey of India for their help in taking the SEM photographs. The directors of the Indian Association for the Cultivation of Science (IACS), the Indian Jute Industries' Research Association (IJIRA), and the Central Glass & Ceramic Research Institute (CG&CRI) are deeply acknowledged for their interest and facility support.

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