Characterization of Jute Fibers Treated with Soap–Glycerol Micelles

KH. M. MANNAN, MD. S. MUNIR

Department of Physics, Dhaka University, Dhaka, Bangladesh

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ABSTRACT: A tossa variety of jute fiber (*Corchorus olitorious*) treated with soap–glycerol micelles is characterized by infrared (IR) spectroscopy, X-ray diffraction method, and tensilometry. The IR spectra for jute fibers treated with soap–glycerol micelles show a reduced absorption band due to O—H stretching at a frequency of 3420 cm^{-1} with almost absent OH bending frequencies, prominent CH_2 stretching and bending frequencies at 2915 and 1440 cm^{-1} and reduced skeletal vibration at 1060 cm^{-1}. The percentage crystallinity measured by the X-ray diffraction method increases from 45 to 53% on treated jute fibers. The tensile strength and strain percent at maximum load, Young's modulus, and work done per unit volume within an elastic limit (resilience) for treated fibers increased from 1.8 ± 0.2 to 3.43 ± 0.2 GPa, from 3.98 ± 0.1 to 4.75 ± 0.1, from 75 ± 2 to 113 ± 5 GPa, and from 26 ± 2 to 74 ± 3 MJ m^{-3}, respectively. Using a stabilizing agent (2%) and a swelling agent (2% KOH), the tensile strength, strain percent, Young's modulus, and resilience increase to 4.02 ± 0.2 GPa, 4.85 ± 0.3, 154 ± 5 GPa, and 95 ± 4 MJ m^{-3}, respectively. Under natural weathering at 12–30°C and 30–80% relative humidity over a prolonged period of 8 weeks, all the tensile properties for micelle-treated fibers increase during the first 2 weeks of exposure and then decrease exponentially to the starting values. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 77: 852–856, 2000

Key words: jute fibers; micelle; IR spectra; tensilometry

INTRODUCTION

Jute is an ecofriendly and biodegradable natural fiber. Carpets made from jute, however, are flattened due to low resilience and shredding makes the carpets unattractive to the users. Researchers exploited the techniques of radiation- and photo-induced grafting to modify the properties of natural fibers to obtain fibers for improved textile performance.[1]

Natural jute fibers contain mainly about 60% crystalline cellulose, 26% amorphous hemicellulose, 14% amorphous lignin, and some waxes, traces of proteins, and pectin.[2] The amorphous materials act as the cementing agent. Chemical treatments such as bleaching and delignification and exposure to γ-radiation produce scission and damage the secondary bonds between α-cellulose and the amorphous contents.[3,4] Mercerization of virgin jute fibers by NaOH showed that although there was a considerable amount of cellulose I–cellulose II transformation as revealed by X-ray diffraction studies cellulose I reappeared when alkali is removed by washing.[5–7] These experiments suggest that the secondary bonds between α-cellulose and lignin prevent reversal of polarity of the α-cellulose chains.

This article describes a simple method for treating jute fibers with soap–glycerol micelles in an aqueous solution. The treated jute fibers have

Correspondence to: Kh. M. Mannan.
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improved tensile strength, strain percent, Young's modulus, and resilience. These improvements in the mechanical properties are retained even after prolonged natural weathering. The method can be incorporated into the jute carpet industries without many modifications in the existing industrial setups. The micelle-treated fibers are characterized by infrared (IR) spectroscopy, the X-ray diffraction method, and tensilometry.

EXPERIMENTAL

Chemical Treatments

A virgin tossa variety of jute fibers (*Corchorus olitorius*) purchased from the market near Dhaka were used for this study. Specimens from the central portion of the fibers were selected for treating them with soap–glycerol micelles in an aqueous solution.

A surfactant lowers the surface tension of water and forms micelles by entrapping oily substances. The concentration at which the surface tension ceases to fall is called critical micelle concentration (cmc). Micelles below the cmc make the surfactant solution of a colloidal nature.[8] An aqueous solution of soap–glycerol micelles was prepared by heating and stirring the solution by a magnetic stirrer hot plate. A 90 ± 5°C temperature and a stirring rate of 200–250/min for more than 20 min gave the best result. The fibers were directly boiled for 10–15 min in this aqueous mixture of micelles formed by glycerol (Robinson Wagner Co., New York, NY) and soap (Pears soap). The fibers thus treated were throughly washed in running water for about 30 min and then dried by an IR lamp (Philips, Infraphil, 150 W). In another treatment, glycerol and soap were prepared in a conical flask by saponification of Malaysian palm oil in an aqueous solution of KOH, heating and stirring the solution by a magnetic stirrer hot plate at the rate of 200–250/min for about 30 min at a temperature of 90 ± 5°C. An aqueous medium with dispersed surfactant–glycerol micelles was formed. Cut pieces of the fibers were boiled for 10–15 min in this medium at the same temperature and stirring rate. Then, the specimens were thoroughly washed in running water for about 30 min. For some specimens, a stabilizer was added at the stage of preparing the micelles.

IR Spectra

Samples were first cut into small pieces by a scissor and then these were repeatedly cut at the tip of a small scissor for about 1 h. Larger portions were picked up by forceps and a granular portion of cellulose was selected for the study. About 100 mg of potassium bromide was powdered in a mortar and then dried in an oven for about 20 min. Samples weighing 1 mg were mixed thoroughly in a mortar with 100 mg of dried KBr. The mixture of cellulose and KBr was placed in a die, which was then evacuated and subjected to a working pressure of 6 tons for 5-min pressing time. To study the effect of the free and bonded OH, the specimens were not dried at all. The spectra of jute fibers dispersed in KBr were recorded by a Shimadzu IR-470 IR spectrophotometer.

X-ray Diffraction

X-ray equatorial diffraction profiles were collected by a JEOL diffractometer, Model JDX 8P, using CuKα radiation at the operating voltage and current of 30 kV and 20 mA, respectively, from 5° to 50° of the 2θ range at the scanning speed of 1°/min. The specimens were cut into very fine pieces as in the IR study.

Tensilometry

All tensile measurements were made following ASTM D2101 and ASTM 3822 standards. A computer-controlled constant rate of an extension-type LRX materials testing machine (Lloyd Instruments Ltd.), meeting the ASTM E4 accuracy of having speed and force measurement accuracies of 0.2 and 0.5%, respectively, was used. For the optimum degree of comparability, specimens having under 8% elongation at break were extended at the same rate of elongation, which was 10% of the initial length (25 mm) of the specimen per minute.

Tensilometry data were collected and analyzed in accordance with the ASTM D2101-94 specifications. Data were analyzed by a software (DAPMAT) which calculates the initial moduli from the best straight line fitted by the least-squares method using data of the selected linear portion of the load-extension curve. This program also calculates the area under the selected portion of the load-extension curve. All specimens were tested in an air-conditioned room at about 65 ± 8% relative humidity and 25 ± 5°C temperature. The average values of tensile strength, strain percent, Young's modulus, and work done per unit volume within the elastic limit (resilience) quoted in this study were calculated by the software from 6–10 observations for each sample and these have 0.1–

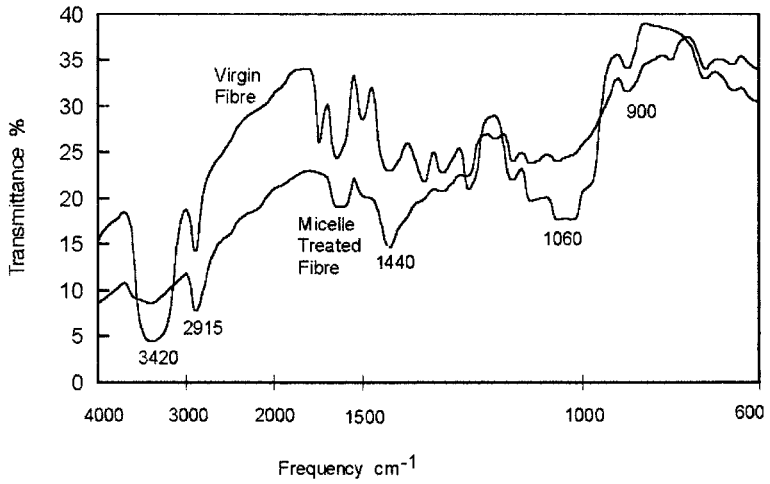

Figure 1 IR spectra for virgin and soap–glycerol micelle-treated jute fibers (*C. olitorious*).

0.2 GPa, 0.1–0.3, 2–5 GPa, and 2–5 MJ m^{-3} standard deviations, respectively.

RESULTS AND DISCUSSIONS

General

All the surfactant–glycerol micelle-treated fibers changed color from yellow to amber and became silky and absorbed 8% moisture (on dry basis), which is 2% less than that absorbed by the virgin jute fibers.

Infrared Spectra

At reactions in which the chain of the cellulose remains essentially intact, the reaction takes place with the three free OH groups.[9] Figure 1 shows the IR spectra for the virgin and treated

Table I Absorption Band for Virgin Jute Fibers

Approximate Position of Maximum		
Wavelength (microns)	Wave No. (cm^{-1})	Vibrating Groups Most Probably Giving Rise to the Observed Absorption Band
2.8–3.2	3420	Free OH and bonded OH stretching
3.44	2915	CH_2 asymmetric stretching
5.80	1724	C═O stretching (acid)
6.10	1639	Adsorbed H_2O
6.33	1580	COO^- ion stretching
6.66	1502	Aromatic skeletal ring vibration due to lignin
6.93	1440	CH_2 symmetric bending
7.35	1360	CH deformation
7.57	1320	CH deformation or CH_2 wagging
8.03	1245	CH bending
8.61	1160	Antisymmetric bridge oxygen stretching
9.02	1109	C—O—C or antisymmetric in phase ring stretching
9.43	1060	Skeletal vibration
9.78	1022	C—OH stretching
11.11	900	C_1 group frequency
15.15	660	OH out-of-plane deformation
16.53	605	OH out-of-plane deformation

Table II Tensile Properties of Virgin Jute Fibers (*C. olitorius*) and of Jute Fibers Treated with Soap–Glycerol Micelles Dispersed in Aqueous Solution With and Without a Stabilizer

Sample	Tensile Strength at Max Load (GPa)	Strain % at Max Load	Young's Modulus (GPa)	Resilience (MJ m^{-3})
S1: Raw Tossa	1.82 ± 0.2	3.98 ± 0.1	75 ± 2	26 ± 2
S2: Tossa boiled with 2% KOH and 2% stabilizer	1.78 ± 0.2	4.38 ± 0.2	62 ± 3	30 ± 2
S3: Tossa boiled with 8% glycerol, 2% soap, and 2% KOH	3.43 ± 0.2	4.75 ± 0.1	113 ± 5	74 ± 3
S4: Tossa boiled with 8% glycerol, 2% soap, 2% stabilizer, and 2% KOH	4.02 ± 0.2	4.85 ± 0.3	154 ± 5	95 ± 4
S5: Tossa boiled with 10% palm oil and 2% KOH	3.08 ± 0.2	4.62 ± 0.3	102 ± 3	66 ± 5
S6: Tossa boiled with 10% palm oil, 2% KOH, and 2% stabilizer	4.78 ± 0.2	4.95 ± 0.2	146 ± 3	111 ± 5
S7: Tossa boiled with 10% palm oil and 2% KOH after 8 weeks of weathering	2.97 ± 0.2	4.50 ± 0.2	86 ± 4	60 ± 3

jute fibers. The absorption bands corresponding to the vibrating groups giving rise to the most probable absorption bands are given in Table I. These assignments of the absorption bands for the virgin jute fibers are in agreement with those for cellulose obtained from sources like cotton linters, white birch, and black spruce.[10] The extra band at 1502 cm^{-1} has been assigned to the aromatic skeletal ring vibration due to lignin.[11]

The IR spectra for jute fibers treated with soap–glycerol micelles and a stabilizer show several intesting differences from the IR spectra for the virgin fibers. First, the absorption band due to O—H stretching (3420 cm^{-1}) is considerably reduced and O—H bending frequencies are almost suppressed. Second, the CH_2 stretching frequency at 2915 cm^{-1} and the CH_2 bending frequency at 1440 cm^{-11} become very prominent. Third, the absorption band due to ring vibration at 1060 cm^{-1} is greatly reduced. Fourth, the carbonyl stretching frequency at 1724 cm^{-1} disappears. The sample for virgin jute fibers was dried in an oven at 80°C for 12 h and the band at 1060 cm^{-1} is not affected, whereas all bands due to OH vibrations disappear.

O'Connor et al.[12] showed that the ratio of absorbances at 7.0 and 11.1 microns is a reasonably sensitive measure of the degree of crystallinity. Using appropriate baselines, this index of crystallinity increases from 2.97 for virgin fibers to 4.44 for the micelle-treated fibers.

X-ray Diffraction Study

The X-ray diffraction patterns for the virgin and micelle-treated jute fibers are structurally identical. There are several methods used to assess the crystallinity by X-ray diffraction techniques.[13] The classical method of Hermans and Weidinger gives a measure of the crystallinity in terms of the ratio of integrated intensity under the peaks to the integrated intensity under the complete trace.[14] Although this method has difficulty in separating the scattering of the crystalline regions from that of the background due to noncrystalline regions, it is simple to use. The crystallinity for the virgin jute fiber increases from 43 to 55% on treatment with soap–glycerol micelles.

Tensile Properties

Results of the tensile properties for different chemical treatments are given in Table II. Several specimens were prepared by boiling these with different concentrations of KOH and it is found that above 2% KOH the tensile properties deteriorate as KOH concentration increases. Specimens were also prepared by keeping the 2% stabilizer fixed and with different concentrations of KOH (sample S2). It is found that the differences in the tensile properties between the virgin fibers (sample S1) and fibers treated with KOH and the stabilizer (sample S2) are statistically insignificant. In general, under the best condi-

tions as in samples S-4 and S-6, there is improvement in tensile strength and resilience by 2.5–3 times, although strain percent at break does not show much improvement. The improved mechanical properties for samples S4 and S6 along with the increased crystallinity suggest an improved chain folding.[15]

Jute fibers treated with micelles were subjected to natural weathering conditions for 8 weeks by keeping these outside the room, and during daytime, these were directly exposed to the sunlight. The outside temperature varied from 12 to 30°C during the night and daytime, respectively, while the relative humidity varied from 30 to 80%. These improvements are retained even under prolonged natural weathering (sample S-7).

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