A Comparative Study of the Stress-Relaxation Behavior of Untreated and Alkali-Treated Jute Fibers

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ABSTRACT: In this study, we examined the effect of alkali treatment and its variables, namely, the time (2, 4, 6, or 8 h) and the concentration of alkali (1, 5, or 17.5% w/w), on the linear density, strength, and stress-relaxation properties of jute fiber. It was demonstrated that this kind of treatment led to the creation of several voids and fiber fibrillation. Properties were measured for the alkali-treated and dewaxed fibers. The linear density and tenacity of the fibers were reduced at higher alkali concentration and at longer dipping times. The strength increased with treatment with mild alkali and decreased with treatment with the strong alkali. A very low alkali treatment (1%) rendered low relaxation. At a 5% alkali concentration, interfibrillar matrix softening also played an important role and was prominent in the stress-relaxation behavior. The stress-relaxation value was much higher in the fibers treated with 17.5% NaOH compared to the dewaxed fibers; this was probably due to a loss in the fibrillar arrangement. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 123: 1348–1358, 2012

Key words: density; fibers; mechanical properties; orientation; relaxation

INTRODUCTION

The prominent advantages of natural fibers include acceptable specific strength properties, low cost, low density, high toughness, good thermal properties, and so on.[1] Their low specific weight, which results in a higher specific strength and stiffness than glass, is a benefit, especially in parts designed for bending stiffness. In the fields of the automotive industry, the reduction of energy consumption in the production of motor vehicles and the improvement of their day-to-day fuel economy are growing upward because of the accelerating use of natural-fiber composites. Among all of the natural fibers, jute fibers are one of the most important abundant fibers with a high cellulose content. Jute fibers have been subjected to various types of chemical treatments in several studies to improve their suitability as textile materials and reinforcing materials in composites. Among these chemical treatments, alkali treatment is the economically most viable one. The available literature reveals that alkali treatment removes one of jute fiber's cementing materials, hemicellulose, depending on the concentration of the alkali used, time and temperature of treatment, liquor ratio, and so on. Ray et al.[2] reported a 41% loss of the hemicellulose content of jute fibers on treatment with a 5% NaOH solution at 30°C for 2 h.

Nowadays, jute fiber is used extensively for various applications, such as textile materials and reinforcements for composites, and they need to be investigated in detail for their viscoelastic properties, such as relaxation and creep. When a fiber is held stretched, the stress in it gradually decays with time. It may drop to a limiting value when the fiber undergoes permanent set to the stretched length. This phenomenon is known as relaxation. Any change that affects the fine or molecular structure of the jute fiber will affect the viscoelastic properties of the fiber. In jute fibers, the cellulose chains lie roughly parallel to the long axis of the fiber. In localized regions of the fiber, the chains are oriented with respect to each other in such a way so as to form a crystalline lattice. When a load is applied to a fiber, the stress is transmitted from one chain to its neighbor by means of intermolecular forces. These forces are strongest in the crystalline regions where the chains lie in the closest proximity to each other, whereas in the noncrystalline portions, the application of stress causes the rearrangement of the localized cellulose chains with respect to each other. This rearrangement is believed to be responsible for stress-relaxation behavior under the conditions of the constant deformation of the fiber.

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Different theories have been proposed by many authors to explain the molecular concept of the stress-relaxation behavior of fibers.[3] Tobolsky and Andrews[4] postulated that motion due to the decay of the primary structural elements of the fiber network, the relaxation of the secondary cross bonds permitting units of the network structure to slip on the application of stress and motion of the mobile segments of the long molecules or group of molecules, were responsible for the relaxation behavior of fibers.

Some studies have also been done on the stress-relaxation behavior of textile fibers, such as cotton, viscose rayon, cellulose acetate, silk, nylon, and wool.[3,5–10] The stress relaxation of cotton and rayon cords at a constant length was measured by G'Burleigh and Wakeham.[3] They used a metaelectric strain gauge for the intermittent measurement of stress over a period of time. After suitable conditioning, the cords were stretched and clamped at zero time, and the galvanometer began its rapid deflection, corresponding to stress decay in the cords. Meredith[5] described an experimental procedure for investigating the stress-relaxation behavior in stretched cellulose fibers. He observed the decay of stress in cotton, flax, viscose, and cellulose acetate yarns. The yarns were rapidly stretched at 65% relative humidity and 25°C, and the relaxation of stress was followed for 24 h. The stress relaxation of viscose rayon, cellulose acetate, and silk were studied by Smith and Eisenschitz.[6] The creep and stress-relaxation behavior of nylon fibers, polyester fibers, wool, and so on have also been studied by other workers.[9,11–13]

Although some experiments have been carried out on different textile fibers, as mentioned previously, such investigations on the viscoelastic behavior of jute fibers have been few. Sett et a.[13] measured the creep and stress decay behavior of rotor- and friction-spun jute blended yarn. Mukherjee et al.[14] measured the stress decay behavior of NaOH-treated and $NaClO_2$-treated jute fibers. They observed that the stress decay was unaffected by the gradual lignin removal, even up to the case of the almost complete delignification on $NaClO_2$ treatment. On the other hand, the removal of hemicellulose on alkali treatment was found to have a distinct effect on the stress decay behavior. They also reported a similar trend in the variation of the compliance ratio with the progressive removal of hemicellulose and lignin. Because the stress decay and compliance ratio, both being related to the fibrillar rearrangement, were affected only by hemicellulose removal, they concluded that the hemicelluloses might be considered more intimately adhered to the fibrils. Hence, they proposed that most of the hemicelluloses were located in the interfibrillar regions within each ultimate cell and that lignin was mainly located in the middle lamella, the cementing layer between the ultimate cells.

The main objectives of this study were the following:

1. To study the effect of different concentrations of alkali on the stress-relaxation behavior, which would show the nature of the fibrillar rearrangement on the removal of the hemicellulose.
2. To observe the effect of the treatment time on the fibrillar rearrangement, which would influence the stress-relaxation behavior of the fibers.

EXPERIMENTAL

Material

Jute fibers of grade Td4 purified through dewaxing with an alcohol and benzene mixture (2 : 1) were used for this study. The fibers were wrapped in black paper and kept in polyethene bags at 25°C and 65% relative humidity. NaOH pellets were used for the treatment.

Alkali treatment

The fibers (50 g) were dewaxed with a methanol–benzene solution (2 : 1) to prepare the control sample. Three bundles of fibers, each containing 50 g of fibers and cut to a 20-cm length, were dipped in three separate beakers containing NaOH solutions at concentrations of 1, 5, and 17.5% w/w, respectively, at room temperature with a liquor ratio of 15 : 1 maintained. After 20 min, the fibers were taken out of the beakers and washed thoroughly with water to remove any NaOH sticking to the surface. The fibers were then treated with dilute acetic acid to remove any trace of NaOH remaining and were finally washed again with water and dried.

In the second set, four bundles of fibers, each containing 50 g of fibers and cut to a 20-cm length, were treated with a 5% NaOH solution for four different time periods: 2, 4, 6, and 8 h. The washing procedure was the same as described earlier.

Testing

Fiber fineness

The fiber fineness was measured by a Lenzing vibroscope in deniers (Norwood, MA). Deniers are the weight per unit length (linear density) of a continuous filament or yarn and are used traditionally in the textile industry. They express the weight in grams of a 9-km (9000-m) length of the material.

Therefore, the lower the denier number is, the finer the material is, and the higher the denier number is, the coarser the material is. The SI unit of the fiber fineness is tex, where 1 den is equal to $\frac{1}{9}$ tex or $\frac{1}{10}$ dtex.

Tensile strength

The tensile strengths of the untreated and the alkali treated fibers were measured by an Instron 1195. A gauge length of 20 mm and a test speed of 5 mm/min were used for this study. Fifty fibers were tested for each set, and the average is reported.

Stress-relaxation behavior

The stress-relaxation behavior of the alkali-treated jute fibers were measured by an Instron 1195. The relaxations were observed for 2 min at two different strain levels, 0.6 and 0.2%, respectively. Fifty samples were tested for each set of fibers, and the average value is reported.

RESULTS AND DISCUSSION

Linear density

The variation of the linear density of the jute fibers as functions of the NaOH concentration and the treatment time are shown in Figures 1 and 2, respectively, and is given in Table I. The linear density was reduced from 26.09 den for the untreated dewaxed fibers to 16.05 den for the jute fibers treated with 17.5% NaOH and 16.99 den for the fibers treated for 8 h with 5% NaOH.

Jute fiber is polymeric in nature and is composed of three major constituents, α-cellulose, hemicellulose, and lignin. Each fiber is composed of a number of ultimate cells, which are held together by a layer called the *middle lamella.* These individual cells are connected with one another in the longitudinal direction to form a long filament. These filaments are again interconnected laterally to form a meshlike structure. Each ultimate cell is a network of very fine, long cellulosic fibrils embedded in a matrix of hemicellulose and lignin. With the stronger alkali (the 17.5% NaOH solution), the destruction of this mesh structure of the jute fibers occurred rapidly; this happened with a lowering of the linear density from 26.09 den for the untreated fibers to 16.99 den for fibers treated with 17.5% NaOH for 20 min. For the mild alkali treatments (1 and 5% NaOH solutions), the splitting was negligibly small within 20 min of treatment time, as was evident from the linear density values. However, with prolonged soaking (up to 8 h) in the 5% NaOH solution, fiber splitting occurred gradually; this increased the fineness of the fibers. Thus, a high concentration with a short treatment time and a low concentration with a long treatment time both resulted in almost the same decrease in the linear density of the fibers.

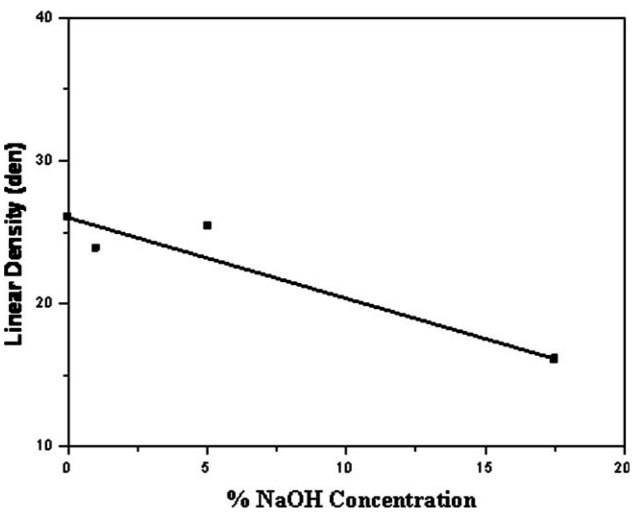

Figure 1 Variation of the linear density of the jute fibers at different alkali concentrations.

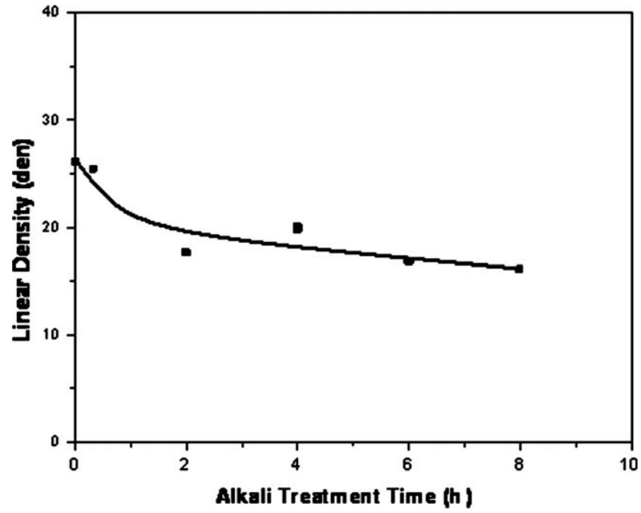

Figure 2 Variation of the linear density of the fibers with the alkali treatment time.

Strength properties

The strength properties of the untreated and alkali-treated fibers are given in Table I and are shown in Figures 3 and 4, respectively. The tenacity of the fibers increased by about 9 and 4% after treatment with 1 and 5% NaOH (for 20 min), respectively, but decreased sharply by 39% after treatment with 17.5% NaOH (Fig. 3). Such an increase in the jute fiber strength on treatment with mild alkali and the decrease in the jute fiber strength on treatment with

TABLE I
Mechanical Properties of the Untreated and Alkali-Treated Jute Fibers

Jute fiber sample	Linear density (den)	Maximum load (g)	Strain at maximum (%)	Breaking strain (%)	Tenacity at maximum (g/den)	Tenacity at break (g/den)	Modulus (g/den)
Dewaxed (20 min)	26.09	77.2	1.09	1.09	2.959	2.866	27.52
Treated with 1% NaOH (for 20 min)	23.87	81.2	1.163	1.165	3.403	3.127	29.74
Treated with 5% NaOH (for 20 min)	25.41	84.4	1.122	1.162	3.323	2.981	30.68
Treated with 17.5% NaOH (for 20 min)	16.05	28.7	3.971	4.023	1.791	1.755	6.234
Treated with 5% NaOH (for 2 h)	17.55	52.4	1.364	1.372	2.983	2.881	23.72
Treated with 5% NaOH (for 4 h)	19.83	73.4	1.263	1.266	3.701	3.352	29.05
Treated with 5% NaOH (for 6 h)	16.87	67.2	1.430	1.453	3.981	3.702	29.75
Treated with 5% NaOH (for 8 h)	16.99	81.9	1.432	1.432	3.900	3.900	29.98

the strong alkali have been reported by many authors.[15–18] As early as 1935, Sarkar[16] reported a 130% improvement in the tensile strength of jute fibers treated with 1 and 8% NaOH solutions for 48 h. Samal et al.[17] also reported 13 and 8% increases, respectively, in the tenacity and modulus of jute fibers treated with 2% NaOH (for 1 h at 35°C). Roy's study[18] showed a steady decrease in the tenacity of fibers with an increase in the alkali concentrations. He reported that the strength retained by the jute fibers after treatment with a 17.5% alkali solution was only 44% of the original. Mukherjee et al.[14] reported 0.38 and 2.67% increases in the initial moduli of jute fibers after treatments with a 1% NaOH solution for 1 h in cold and hot conditions, respectively. They also reported a 52% reduction in the initial modulus of the fibers with treatment with an 18% NaOH solution under cold conditions for 30 min.

The changes in the tenacity, modulus, and breaking strain of the jute fibers subjected to alkali treatment might be possibly due to some interacting factors, such as the following:[18]

1. The rupture of the alkali-sensitive bonds existing between the different components of the fiber due to the partial removal of hemicellulose and the swelling of the fibers.
2. The formation of new hydrogen bonds between certain cellulose chains due to the removal of the hemicelluloses; these bonds normally separate the cellulose chains. This could have been due to the release of the initial strains and the subsequent readjustments of the chains.

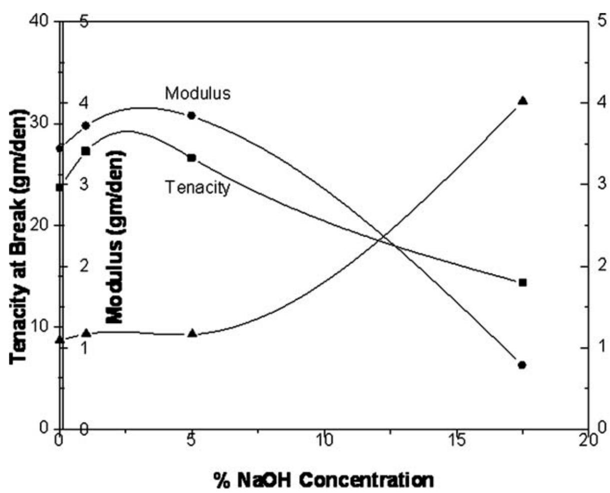

Figure 3 Variation of the strength properties of the jute fibers with the alkali concentration.

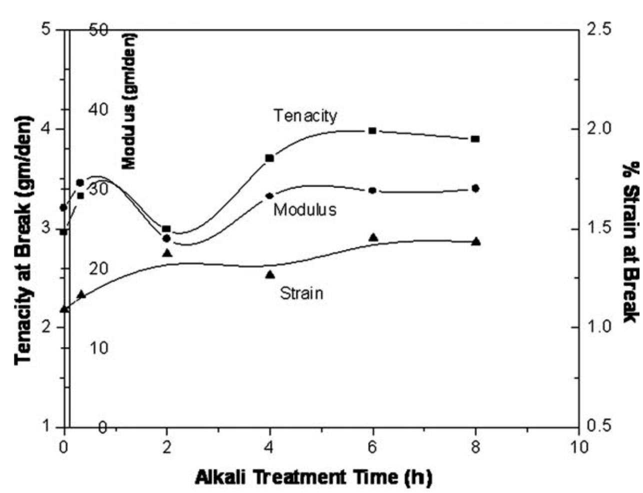

Figure 4 Variation of the strength properties of the jute fibers at different alkali treatment times.

TABLE II
Stress-Relaxation Behavior of the Dewaxed and Alkali-Treated Jute Fibers at 0.6 and 0.2% Constant Strains

Sample	Strain (%)	Maximum load (g)		Load at 0 s (g)		Load at 15 s (g)		Load at 30 s (g)		Load at 60 s (g)		Load at 90 s (g)	
		M	*SD*	*M*	*SD*	*M*	*SD*	*M*	*SD*	*M*	*SD*	*M*	*SD*
Dewaxed	0.2	12.6	.0036	12.6	.0036	12.0	.0034	11.8	.0034	11.7	.0034	11.7	.0034
Td4 jute fiber	0.6	47.8	.0016	47.8	.0016	46.0	.0015	45.5	.0015	45.1	.0015	44.9	.0015
Jute fiber	0.2	10.1	.0041	10.1	.0034	9.6	.0039	9.5	.0038	9.4	.0038	9.4	.0038
treated with 1% NaOH	0.6	35.1	.010	35.1	.010	33.9	.010	33.65	.010	33.4	.010	33.2	.010
Jute fiber	0.2	6.2	.0037	6.2	.0037	5.7	.0033	5.7	.0033	5.6	.0032	5.5	.0032
treated with 5% NaOH	0.6	51.7	.0170	51.7	.0170	48.6	.0169	48.0	.0169	47.4	.0169	47.1	.0168
Jute fiber	0.2	4.65	.0020	4.65	.0020	4.0	.0020	3.85	.0020	3.7	.0020	3.55	.0020
treated with 17.5% NaOH	0.6	15.7	.0053	15.7	.0053	12.9	.0046	12.3	.0044	11.7	.0043	11.4	.0042
Low severity	0.2	8.2	.0028	8.2	.0028	7.3	.0026	7.2	.0026	7.1	.0025	6.9	.0025
Steam-exploded	0.6	47.6	.0137	47.6	.0137	44.2	.0132	43.6	.0131	43.0	.013	42.7	.0129
High severity	0.2	5.0	.0019	5.0	.0019	4.4	.0017	4.2	.0016	4.1	.0016	6.9	.0025
Steam-exploded	0.6	22.8	.0137	22.8	.0137	19.5	.0132	19.0	.0131	18.5	.013	18.2	.0129

M = mean; *SD* = standard deviation. Crosshead speed = 5 mm/min. Hold time = 2 min.

3. The changes in the proportion of the crystalline cellulose.
4. The decrease in the orientation of the molecular chains.

After treatment with low-concentration alkali (1–7%), the disorientation effect was negligible, and the lowering in the strength was mainly due to the cleavage of the alkali-sensitive bonds; this resulted in a decrease in the force of cohesion of the molecular chains. With a strong alkali solution (>9%), the crystalline regions were drastically affected; this led to a severe breakage of bonds and produced a pronounced disorientation effect, which contributed toward a loss in the strength of the fibers.

Stress-relaxation behavior

The stress-relaxation behavior of the dewaxed untreated Td4 (control) fibers and the alkali-treated jute fibers (treated with 1, 5, and 17.5% NaOH for 20 min) at 0.2 and 0.6% strain levels is given in Table II. The variation in σ/σ_0 (where σ is the load in the fiber at time t and σ_0 is the load in the fiber at time $t = 0$) against time (s) at 0.2 and 0.6% strain levels is given in Table III and is shown in Figures 5 and 6, respectively.

TABLE III
Variation of σ/σ_0 with the Treatment Time of the Dewaxed and Alkali-Treated Jute Fibers

Sample	Strain (%)	Maximum load (g)	σ/σ_0 at 0 s	σ/σ_0 at 15 s	σ/σ_0 at 30 s	σ/σ_0 at 60 s	σ/σ_0 at 90 s
Dewaxed	0.2	12.6	1	0.9523	0.9365	0.9285	0.9285
jute fiber	0.6	47.8	1	0.9623	0.9529	0.9445	0.9393
Jute fiber	0.2	10.1	1	0.9504	0.9405	0.9306	0.9285
treated with 1% NaOH (for 20 min)	0.6	35.05	1	0.9671	0.9600	0.9529	0.9472
Jute fiber treated	0.2	6.2	1	0.9193	0.9193	0.9032	0.8870
with 5% NaOH (for 20 min)	0.6	51.7	1	0.9400	0.9284	0.9168	0.9110
Jute fiber treated	0.2	4.65	1	0.8602	0.8279	0.7956	0.7634
with 17.5% NaOH (for 20 min)	0.6	15.7	1	0.8216	0.7834	0.7452	0.7261

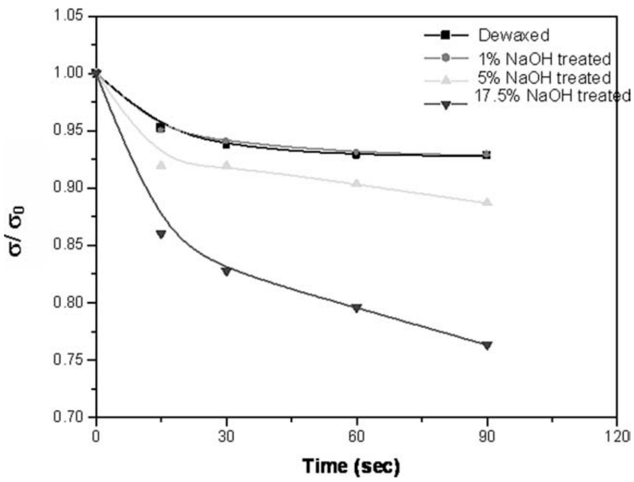

Figure 5 Stress-relaxation behavior of the dewaxed jute fibers and jute fibers treated with 1, 5, and 17.5% NaOH at a 0.2% strain level.

At a 0.2% constant strain (Fig. 5), the relaxation behavior of the dewaxed fibers and the fibers treated with 1% NaOH were almost similar, whereas the relaxation was more prominent in the fibers treated with 5% NaOH. For the jute fibers treated with 17.5% NaOH, a very sharp stress relaxation was observed. At a higher strain level (0.6%; Fig. 6), the stress-relaxation behavior was more pronounced. The fibers treated with 1% NaOH showed a decrease in the stress-relaxation behavior.

In case of the fibers treated with 5% NaOH for different times (20 min and 2, 4, 6, and 8 h), the stress relaxation at 0.2 and 0.6% strain levels are given in Table IV. The variation of σ/σ_0 at 0.2 and 0.6% strain levels are given in Table V and are shown in Figures 7 and 8, respectively. At the 0.2% strain level, an increase in the stress relaxation was observed for the fibers treated for 20 min and 2 h, but the relaxation decreased abruptly in the fibers treated for 4 h and then showed a gradual increasing trend in the fibers treated for 6 and 8 h (Fig. 7). At a 0.6% strain level, the trends were slightly different, as shown in Figure 8.

When a jute fiber is stretched, there is rearrangement among the fibrils so that they share the load among themselves.[14] With the removal of the hemicellulose, there is a greater ease of rearrangement; this results in a higher stress development in the fiber. At the same time, an opposing phenomenon is operative in the fibers on alkali treatment. The removal of the hemicellulose causes interfibrillar matrix softening; this adversely affects the stress transfer among the fibrils and lowers the stress development in the fibers under tensile deformation.

With the low-concentration NaOH (1%) treatment, the first phenomenon was highly predominant. It was apparent that the vacant space created by the removal of a small amount of hemicellulose allowed a rearrangement among the fibrils on the application of stress, such that a very high stress was developed in the fibers. It increased the strength properties but, on the other hand, decreased the slippage of the chains with respect to each other and lowered its stress-relaxation behavior. This effect was more prominent at a higher (0.6%) strain level (Fig. 6).

In the fibers treated with 5% NaOH, more than 30% hemicellulose was removed.[2] Thus, here, along with the first phenomenon, the second phenomenon, that is, interfibrillar matrix softening, was also partly operative. Hence, there was little increase in the strength properties, and the stress-relaxation behavior was more prominent compared to the dewaxed fibers at both high (0.6%) and low (0.2%) strain levels.

When the jute was treated with the 17.5% NaOH solution, the removal of the hemicellulose was accompanied by the swelling and shrinkage of the ultimate cells;[19] this resulted in some disorientation of the fibrils. Here, the second phenomenon was highly predominant. A loss in the fibrillar arrangement[14] was responsible for the low strength properties and very high stress-relaxation behavior.

At the 0.2% strain level, the rate of the fall of stress relaxation was quite sharp up to 30 s in all of the fibers (dewaxed fibers and fibers treated with 1, 5, and 17.5% NaOH), as shown in Figure 9. However, between 30 and 90 s, the rate became shallower in the case of the dewaxed fibers and the fibers treated with 1 and 17.5% but continued to be same in the fibers treated with 17.5% NaOH, which was imperative of the continuing rearrangement of the cellulose chains, resulting in stress relaxation, even at low stress levels.

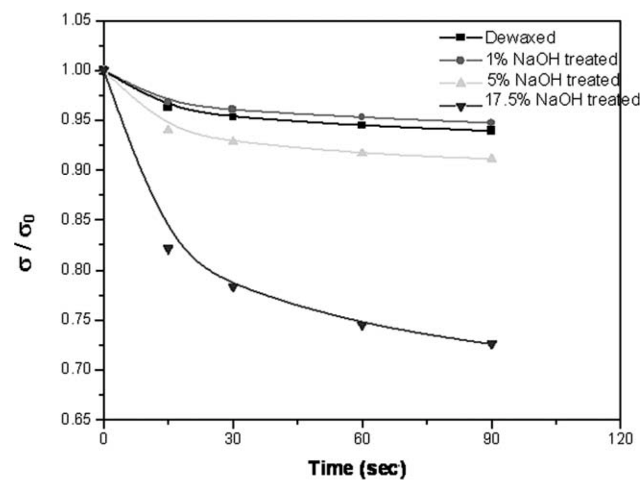

Figure 6 Stress-relaxation behavior of the dewaxed jute fibers and jute fibers treated with 1, 5, and 17.5% NaOH at a 0.6% strain level.

TABLE IV
Stress-Relaxation Behavior of the 5% Alkali-Treated Jute Fibers (Treatment Times = 2, 4, 6, and 8 h) at 0.6 and 0.2% Constant Strains

Sample	Strain (%)	Maximum load (g)		Load at 0 s (g)		Load at 15 s (g)		Load at 30 s (g)		Load at 60 s (g)		Load at 90 s (g)	
		M	*SD*	*M*	*SD*	*M*	*SD*	*M*	*SD*	*M*	*SD*	*M*	*SD*
Dewaxed	0.2	12.6	.0036	12.6	.0036	12.0	.0034	11.8	.0034	11.7	.0034	11.7	.0034
Td4 jute fiber	0.6	47.8	.0016	47.8	.0016	46.0	.0015	45.5	.0015	45.1	.0015	44.9	.0015
Jute fiber	0.2	6.2	.0037	6.2	.0037	5.7	.0033	5.7	.0033	5.6	.0032	5.5	.0032
treated with 5%	0.6	51.7	.0170	51.7	.0170	48.6	.0169	48.0	.0169	47.4	.0169	47.1	.0168
NaOH for 20 min													
Jute fiber	0.2	5.1	.0021	5.1	.0021	4.7	.0021	4.6	.0020	4.5	.0020	4.5	.0020
treated with 5%	0.6	30.0	.0087	30.0	.0087	28.2	.0083	27.9	.0082	27.5	.0082	27.4	.0081
NaOH for 2 h													
Jute fiber	0.2	6.0	.0027	6.0	.0027	5.7	.0025	5.7	.0025	5.6	.0025	5.6	.0025
treated with 5%	0.6	34.7	.0130	34.7	.0130	33.0	.0125	32.6	.0124	32.3	.0123	32.1	.0123
NaOH for 4 h													
Jute fiber	0.2	5.3	.0028	5.3	.0028	5.0	.0027	5.0	.0026	4.9	.0026	4.9	.0026
treated with 5%	0.6	21.5	.0075	21.5	.0075	19.9	.0073	19.6	.0072	19.3	.0072	19.2	.0071
NaOH for 6 h													
Jute fiber	0.2	9.5	.0041	9.5	.0041	8.9	.0038	8.9	.0037	8.8	.0037	8.7	.0037
treated with 5%	0.6	32.7	.0138	32.7	.0138	30.7	.0132	30.4	.0131	30.0	.0130	29.8	.0129
NaOH for 8 h													

M = mean; *SD* = standard deviation. Crosshead speed = 5 mm/min. Hold time = 2 min.

At the 0.6% strain level, the rate of the fall of stress relaxation was quite sharp up to 30 s in all of the fibers (dewaxed fibers and fibers treated with 1, 5, and 17.5% NaOH), as shown in Figure 10. However, between 30 and 90 s, the rate became slower in the dewaxed fibers and the fibers treated with 1 and 17.5% NaOH but continued to be nearly the same in the 5% treated fibers. The interplay between the two opposing phenomena, the rearrangement among the fibrils, and the interfibrillar matrix softening, which was more pronounced in the 5% treated fibers, could have been responsible for such an increased rate in the stress-relaxation behavior at the high stress level. However, in the fibers treated with 1 and 17.5% NaOH, one of the two phenomena was more predominant (the rearrangement among the fibrils in the 1% treated fibers and interfibrillar matrix softening in the 17.5% treated fibers); this resulted in a steady decay in the relaxation rate.

The variation of the rate of stress relaxation at two different strain levels (0.2 and 0.6%) for the dewaxed fibers and the jute fibers treated with 1, 5, and 17.5%

TABLE V
Variation of σ/σ_0 with the Treatment Time of the 5% Alkali-Treated Jute Fibers

Sample	Strain (%)	Maximum load (g)	σ/σ_0 at 0 s	σ/σ_0 at 15 s	σ/σ_0 at 30 s	σ/σ_0 at 60 s	σ/σ_0 at 90 s
Dewaxed jute fiber	0.2	12.6	1	0.9523	0.9365	0.9285	0.9285
	0.6	47.8	1	0.9623	0.9529	0.9445	0.9393
Jute fiber	0.2	6.2	1	0.9193	0.9193	0.9032	0.8870
treated with 5%	0.6	51.7	1	0.9400	0.9284	0.9168	0.9110
NaOH for 20 min							
Jute fiber	0.2	5.1	1	0.9215	0.9019	0.8823	0.8823
treated with 5%	0.6	30.0	1	0.9400	0.9300	0.9167	0.9133
NaOH for 2 h							
Jute fiber	0.2	6.0	1	0.9500	0.9500	0.9333	0.9333
treated with 5%	0.6	34.7	1	0.9510	0.9394	0.9308	0.9250
NaOH for 4 h							
Jute fiber	0.2	5.3	1	0.9433	0.9433	0.9245	0.9245
treated with 5%	0.6	21.5	1	0.9255	0.9116	0.8976	0.8930
NaOH for 6 h							
Jute fiber	0.2	9.5	1	0.9368	0.9368	0.9263	0.9157
treated with 5%	0.6	32.7	1	0.9388	0.9296	0.9174	0.9113
NaOH for 8 h							

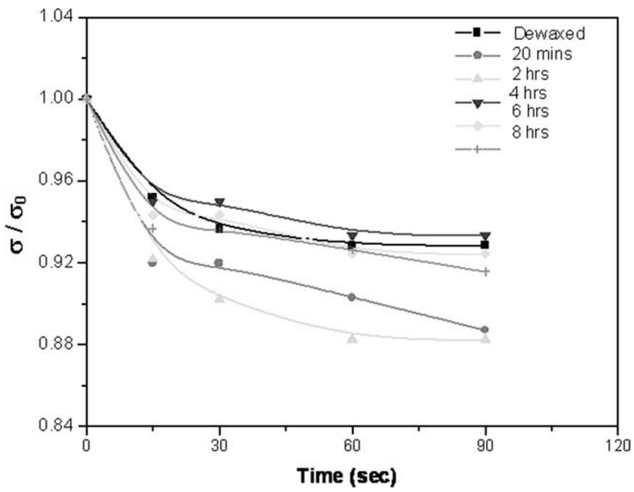

Figure 7 Stress-relaxation behavior of the jute fibers treated with 5% NaOH (for 20 min and 2, 4, 6, and 8 h) at a 0.2% strain level.

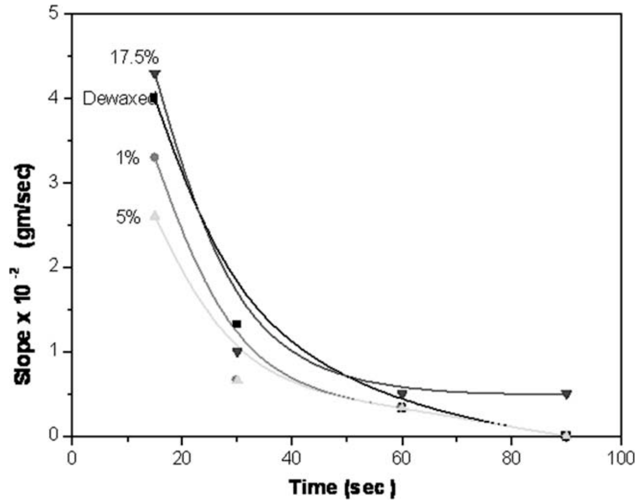

Figure 9 Rate of stress relaxation of the dewaxed jute fibers and jute fibers treated with 1, 5, and 17.5% NaOH at a 0.2% strain level.

NaOH are shown in Figure 11. The rate of relaxation was higher at higher strain levels. This might have been due to the application of greater amounts of force, which caused an increased rearrangement of the chains and more relaxation.

It was apparent from the high stress-relaxation behavior of the fibers treated for 20 min and 2 h (Fig. 12) that the dissolution of hemicellulose might have been predominant up to 2 h. of treatment and might have left vacant sites in the fiber structure and allowed the fibrils to undergo extensive rearrangement, which resulted in high stress-relaxation behavior. The sudden decrease in the stress-relaxation behavior of the fibers treated for 4 h might suggest that some fibrillar rearrangement could have played an active role in lowering the relaxation behavior. Again a gradual increase in the relaxation behavior was observed in case of the fibers treated for 6 and 8 h; this might suggest that the prolonged soaking time facilitated closer rearrangement of the cellulose chains in a more orderly fashion, increasing the relaxation behavior.

However, at the higher strain level (0.6%), when a greater force was applied on the fiber, the compactness of the cellulose chains might have increased to such an extent that the slippage of the chains with respect to each other was reduced and it lowered the relaxation behavior, as shown in Figure 13. This

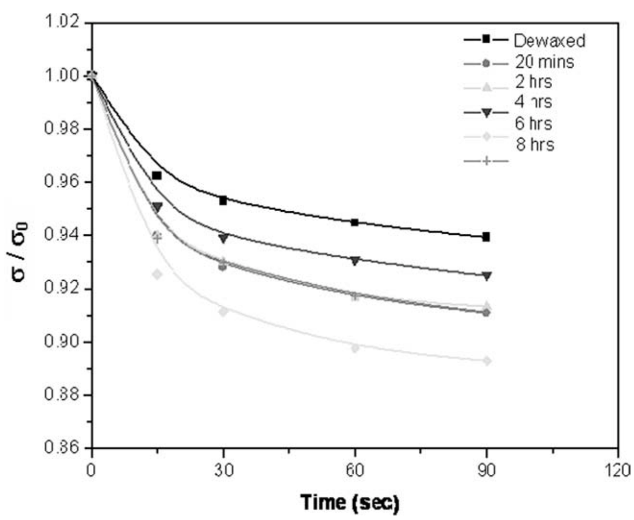

Figure 8 Stress-relaxation behavior of the jute fibers treated with 5% NaOH (for 20 min and 2, 4, 6, and 8 h) at a 0.6% strain level.

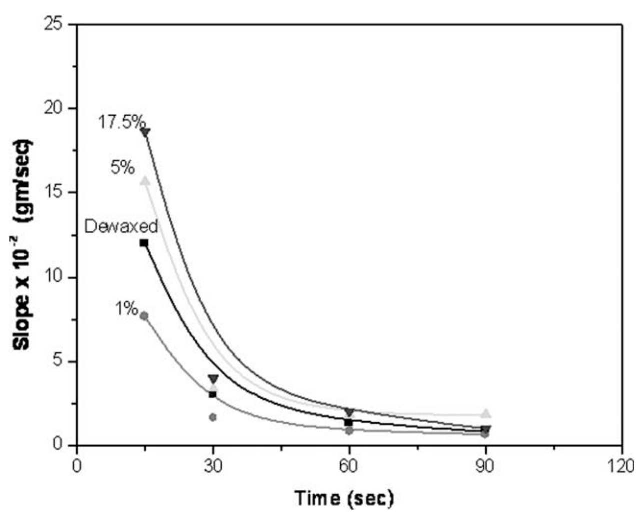

Figure 10 Rate of the stress relaxation of the dewaxed jute fibers and jute fibers treated with 1, 5, and 17.5% NaOH at a 0.6% strain level.

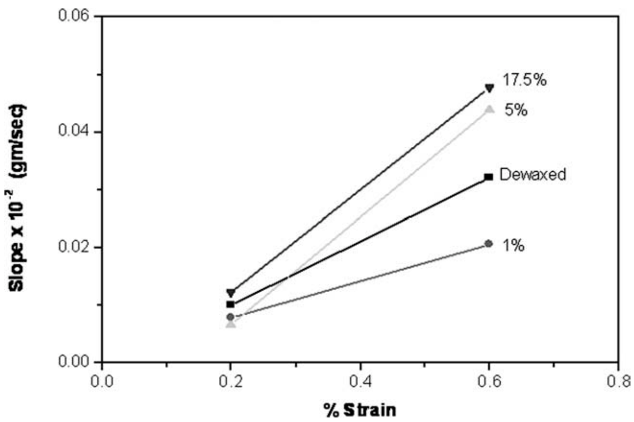

Figure 11 Rate of stress relaxation of the dewaxed jute fibers and jute fibers treated with 1, 5, and 17.5% NaOH at two different stress levels.

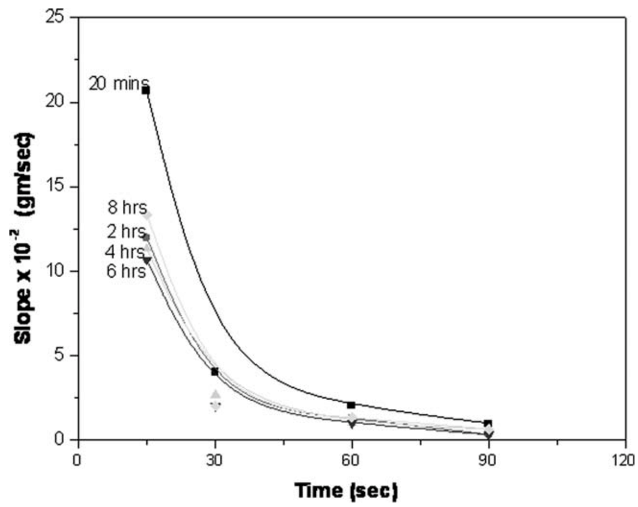

Figure 13 Rate of stress relaxation of the jute fibers treated with 5% NaOH (for 20 min and 2, 4, 6, and 8 h) at a 0.6% strain level.

was also reflected in the rate of stress relaxation of these fibers at the two strain levels, as shown in Figures 11 and 14, respectively. At the low strain level, fibers treated for 8 h showed the highest rate of stress relaxation, but at a higher strain level, the fibers treated for 20 min exhibited a much higher rate of relaxation, followed by those treated for 8 and 2, and the fibers treated for 4 and 6 h showed nearly similar rates of relaxation.

In the fibers treated for 20 min, the dissolution of hemicellulose might have created quite a large vacant space in the fiber structure with no time for the cellulose chains to rearrange among themselves; this resulted in the slippage of the cellulose chains on the application of stress. However, in the fibers treated for 8 h, there was sufficient time for the chains to rearrange themselves in a more compact manner and to share the load among themselves; this resulted in relaxation. In the fibers treated for 4 and 6 h, there was a transition between the dissolution and rearrangement of the chains; this resulted in a low relaxation, shown in Figures 12 and 13. The rate of stress relaxation at the two strain levels are shown in Figure 14.

CONCLUSIONS

1. The linear density was reduced from 26.09 den for the untreated dewaxed fibers to 16.05 den for the fibers treated with 17.5% NaOH and

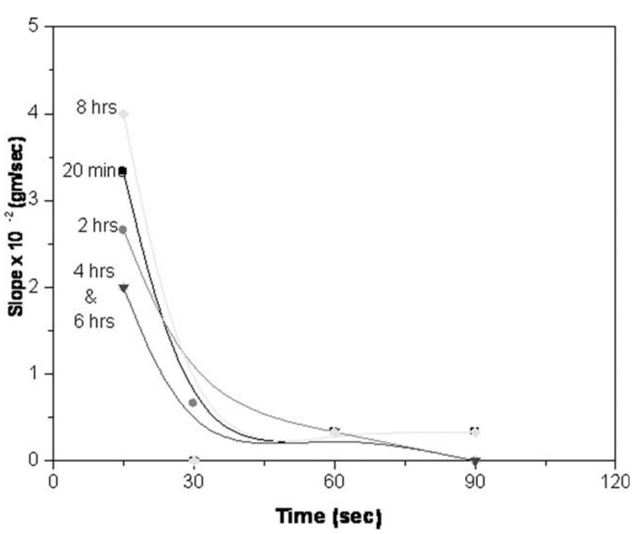

Figure 12 Rate of stress relaxation of the jute fibers treated with 5% NaOH (for 20 min and 2, 4, 6, and 8 h) at a 0.2% strain level.

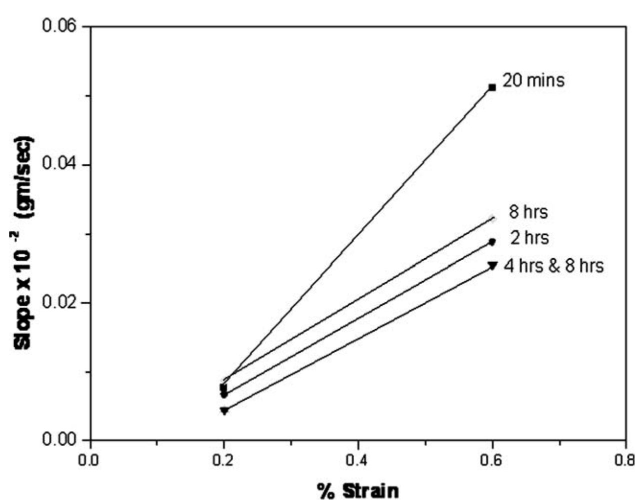

Figure 14 Rate of stress relaxation of the jute fibers treated with 5% NaOH (for 20 min and 2, 4, 6, and 8 h) at two different stress levels.

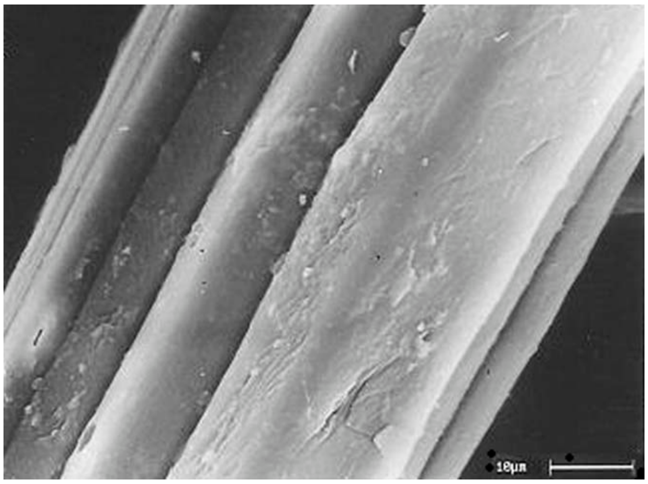

Figure 15 Scanning electron micrograph of the untreated jute fiber.

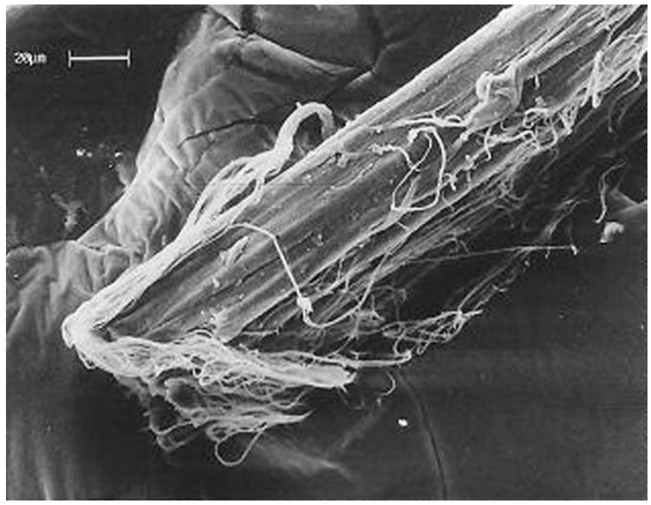

Figure 17 Scanning electron micrograph of the jute fiber treated with 17.5% NaOH (for 20 min).

16.99 den for the fibers treated with 5% NaOH for 8 h. Thus, the high concentration/short treatment time and low concentration/long treatment time combinations both resulted in almost the same decrease in the linear density of the fibers.

2. The strength properties of the fibers increased after treatment with 1 and 5% NaOH but decreased sharply after treatment with 17.5% NaOH. With prolonged treatment time (2, 4, 6, and 8 h), the strength properties increased. The change in the strength properties could have been due to the release of the initial strains and subsequent rearrangements of the cellulose chains in a more compact manner, which affected the crystallinity index of the fibers, as evident from the X-ray diffractograms.[20]
3. On alkali treatment, two opposing phenomena were operative. The removal of hemicellulose caused interfibrillar matrix softening, and also, the release of the initial strains helped the cellulose chains to arrange themselves in a compact manner, which facilitated the development of high stress in the fiber.[19] The interplay of the two factors and the predominance of one factor over the other controlled the stress relaxation and the creep behavior of the fibers.
4. At a low alkali concentration (1%), the removal of a small amount of hemicellulose might have allowed rearrangement of the fibrils in such a manner that a high stress was developed in the fiber, which improved its strength properties; on the other hand, a decrease in the slippage of

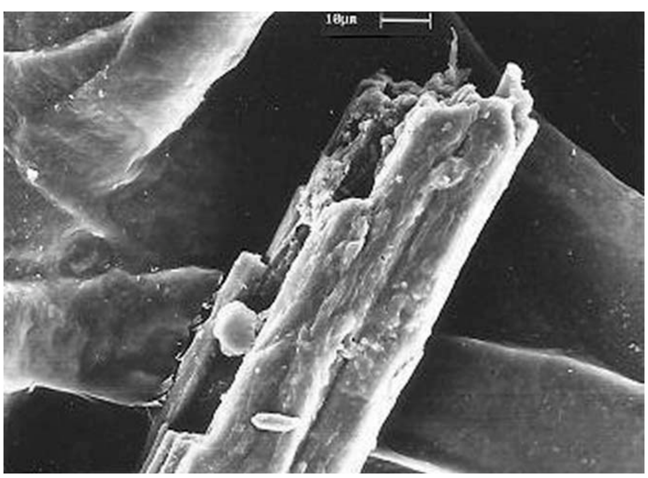

Figure 16 Scanning electron micrograph of the jute fiber treated with 5% NaOH (for 20 min).

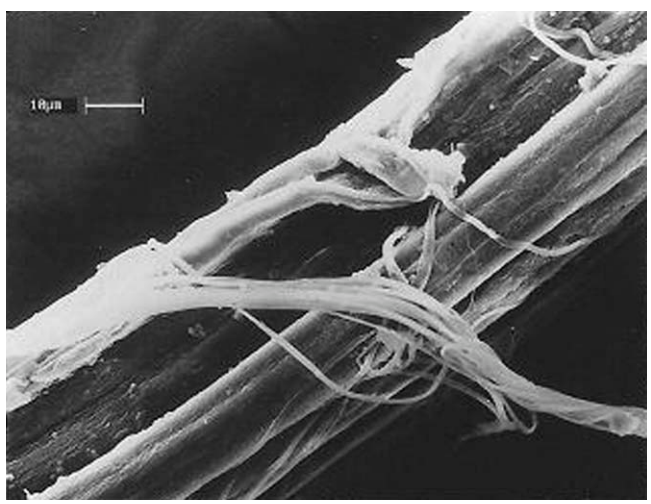

Figure 18 Scanning electron micrograph of the jute fiber treated with 5% NaOH (for 4 h).

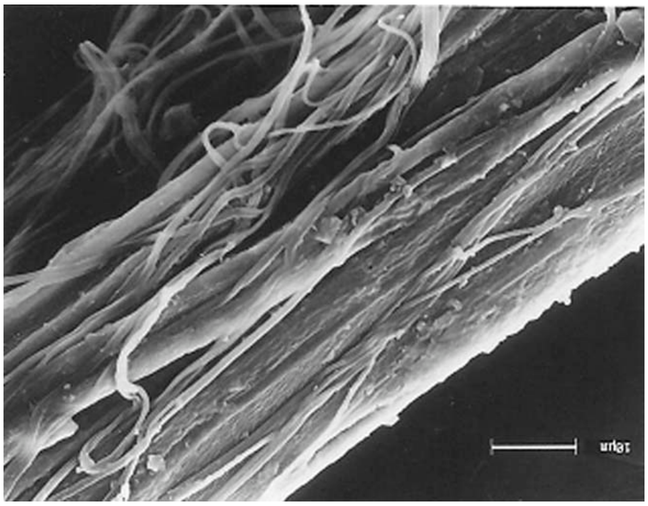

Figure 19 Scanning electron micrograph of the jute fiber treated with 5% NaOH (for 8 h).

the chains with respect to one another decreased their stress-relaxation behavior.

5. At a 5% alkali concentration, the removal of hemicellulose was significant as compared to the untreated one and here, the interfibrillar matrix softening also played an important role (Figures 15–16) and (Figures 18–19). And with time the extent of removal of hemicellulose is increasing as evident from the micrographs. In these fibers, therefore, there was little increase in the strength properties, and the stress relaxation was also more prominent.
6. When the fibers were treated with a 17.5% NaOH solution dissolution of hemicellulose is more (Fig-17) and the removal of hemicellulose was accompanied by the swelling and shrinkage of the ultimate cells;[19] this resulted in some disorientation of the fibrils. Such a loss in fibrillar arrangement might have been responsible for such low strength and high stress-relaxation properties.
7. With longer treatment times, the dissolution of hemicellulose was followed by the subsequent rearrangement of the cellulose chains in a compact manner. The dissolution of hemicellulose was predominant up to 2 h of treatment; this led to a high stress-relaxation behavior. After that, the readjustments of the fibrils among each other might have become predominant and lowered the stress-relaxation behavior.

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References

1. Bledzki, A. K.; Gassan, J. Prog Polym Sci 1999, 24, 221.
2. Physical Properties of Textile Fibers; Morton, W. E.; Hearle, J. W. S., Eds.; Heinemann: London; 1962, p 354.
3. G'Burleigh, E.; Wakeham, H. Text Res J 1947, 17, 245.
4. Tobolsky, A. V.; Andrews, R. D. J Chem Phys 1945, 13, 3.
5. Meredith, R. J Text Inst 1954, 45, T438.
6. Smith, H. D.; Eisenschitz, R. J Text Inst 1931, 22, T170.
7. O'Shaughnessy, M. T. Text Res J 1948, 18, 263.
8. Mauryama, T.; Dumbleton, J. H.; Williams, M. I. J Macromol Sci Phys 1967, 1, 1.
9. Meredith, R.; Hsu, B. J Polym Sci 1962, 61, 253.
10. Abbott, N. J. Text Res J 1951, 21, 227.
11. Catsiff, E.; Alfrey, T., Jr.; Shaughnessy, M. T. Text Res J 1951, 23, 808.
12. Feughelman, M. J Text Inst 1954, 45, T630.
13. Sett, S. K.; Mukherjee, A.; Sur, D. Text Res J 2000, 70, 723.
14. Mukherjee, A.; Ganguly, P. K.; Sur, D. J Text Inst 1993, 84, 348.
15. Macmillan, W. G.; Sengupta, A. B.; Majumdar, S. K. J Text Inst 1954, 45, T703.
16. Sarkar, P. B. Ind J Chem Soc 1935, 12, 23.
17. Samal, R. K.; Mohanty, M.; Panda, B. B. J Polym Mater 1995, 12, 235.
18. Roy, M. M. J Text Inst 1953, 44, T44.
19. Holland, H. D.; Halscy, G.; Eyring, H. Text Res J 1946, 16, 723.
20. Ray, D.; Sarkar, B. K. J Appl Polym Sci 2000, 80, 1013.