A Comparative Study of the Response of Cottons to Zinc Chloride and Sodium Hydroxide

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Synopsis

Different varieties of cotton drawn from various botanical species were subjected to swelling and stretching in 64.5% (w/w) aqueous zinc chloride at 32° ± 1°C for different intervals ranging from 10 to 50 min. Swelling was almost complete in 30 min. Samples stretched to original length after this interval showed tremendous improvement in orientation and strength both at zero and 3 mm gauge lengths. In several cases, the improvement was higher than that obtained with similar treatment in sodium hydroxide of mercerizing concentration at room temperature. Swelling in zinc chloride was found to be independent of the variety of cotton as evidenced by the 50% X-ray angle for the stretched fibers, which was about 9° for all the six cottons included in this study.

INTRODUCTION

Aqueous zinc chloride ($ZnCl_2$) is one of the best known swelling and decrystallizing agents.[1] Though this reagent was included by Mercer[2] in the list of mercerizing agents, very little is known about the mechanical and fine structural behavior of cotton fibers swollen and stretched in $ZnCl_2$ under controlled conditions of concentration, temperature, and time.

Unlike sodium hydroxide (NaOH), the swelling in $ZnCl_2$ is highly dependent on the duration of treatment besides concentration and temperature. Previous workers[3,4] have identified two swelling maxima for cotton fibers treated with various concentrations of $ZnCl_2$. Given sufficient time, both these concentrations produce highly decrystallized fibers, the relative effectiveness of one concentration (≃ 65%) over the other (≃ 70%) in producing decrystallization being a function of temperature, when the time of swelling is kept the same. Extensive data[3-7] are available on the decrystallization and other aspects like LODP, moisture regain, dye absorption, etc. as a function of concentration and temperature for fibers swollen in $ZnCl_2$.

Studies on the effect of $ZnCl_2$ on cotton referred to above are, however, limited to free swelling unaccompanied by stretching. It is well known[8-12] that swelling and subsequent stretching in NaOH brings about varying extents of change in fiber properties in different varieties of cotton. The only work in this line with $ZnCl_2$ is that reported by Joshi et al.[13] They used 65% $ZnCl_2$ at 35°C and studied a particular variety of cotton. It was noted that while single fiber strength, static modulus, and X-ray orientation factor registered consid-

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erable increase, lateral order showed very little change. The present investigation was undertaken with a view to gather detailed information on the effect of $ZnCl_2$ swelling on the mechanical and structural properties of different varieties of cotton and to make a critical comparison of their response to the action by the two swelling agents, $ZnCl_2$ and NaOH.

EXPERIMENTAL

Samples

Six varieties of cotton drawn from different botanical species and having widely different initial fiber properties were selected for the study. Fibers were kier-boiled before the swelling treatments.

Swelling and Stretching Treatments

Flat bundles of parallelized fibers were prepared and mounted on jaws by means of devices similar to those described in Ref. 8. The bundles gripped in the jaws were swollen slack. After the predetermined interval, the jaws were placed on a stretching frame and the fibers were stretched to the original length. For washing, the entire frame was kept in running water for 30 min with the bundle remaining stretched. The fibers were then removed from the jaws, washed again, and dried in air. For preparing slack swollen fibers all the above steps except stretching were followed.

In the case of $ZnCl_2$ the concentration and temperature were fixed at 64.5% (w/w, specific gravity at 30°C = 1.8336) and 32° ± 1°C, respectively, while three swelling times viz. 10 min, 30 min, and 50 min were employed. The above conditions were selected after analyzing the results of preliminary experiments which showed that both higher temperature and higher concentration were detrimental to strength even when the swelling time was limited to 10 min. For NaOH, a concentration of 21% (w/w) and temperature of about 31°C were chosen besides a swelling time of 10 min except for one particular variety of cotton which was swollen for additional timings of 1, 2, 5, 30, and 50 min so as to see the time effect with NaOH.

Strength Tests

Bundle tenacity was determined at two gauge lengths viz. zero and 3 mm using a Stelometer employing the standard procedure.[14] The fibers were conditioned at 65% relative humidity and 27°C before the strength tests.

Orientation and Crystallinity Measurements

The azimuthal intensity profiles of the 002 diffraction arcs from parallel fiber bundles were obtained by using a Philips stabilized X-ray generator fitted with a bundle rotating device and recording accessories. The 50% X-ray angle (ψ), computed from the intensity profile, was used as a measure of orientation as suggested by Creely et al.[15] The 002 profile has been used for estimating orientation in $ZnCl_2$ and NaOH-treated fibers since it has been shown[16] that sufficient accuracy can be achieved in this procedure even in case of a mixed lattice.

Radial intensity scans of the powdered samples (cut with a Wiley mill to pass a 40 mesh screen) were obtained in the Bragg angle range 10°–40° (2θ). The total crystallinity values were obtained using Segal's method.[17] It may be noted that Segal's index was originally suggested for cellulose I lattice; however, recent work at CTRL[18] has confirmed its applicability to samples containing both cellulose I and cellulose II lattices as in the present case.

Infrared (IR) crystallinities of the samples were obtained from the infrared spectra, of the cut powder in KBr matrix, recorded with a Perkin Elmer model 457 IR spectrophotometer and using the ratio of the absorbances of the 1372 cm^{-1} and 2900 cm^{-1} bands as suggested by Nelson and O'Connor.[19]

RESULTS

Table I gives the major fiber properties of the six cotton samples used in the present study. All the samples have nearly the same level of fiber maturity except Hybrid 4(2) which was specially included in the study to test the effect of swelling on a poorly mature sample. Strength, fineness, and X-ray angle show a wide spread as samples were selected from different genetic varieties.

Time Effects

The influence of the duration of swelling on the properties of the treated fibers is evident from Figure 1(A–C) in which the bundle tenacity and ψ are plotted against swelling time for one particular sample of cotton [Hybrid 4(1)] stretched to original length. With $ZnCl_2$, swelling proceeds slowly. However, the fiber properties characterized by bundle tenacity and X-ray angle do not show any further change after 30 min indicating completeness of swelling in this interval. With NaOH, on the other hand, swelling is almost complete in about one minute and increase in the swelling time had very little effect on the fiber properties.

Lateral Order

The IR and X-ray crystallinity indices for the swollen and swollen and stretched samples for the variety Hybrid 4(1) are given in Table II. Swelling causes decrease in the lateral order, and it is more for the $ZnCL_2$ sample swollen slack for 30 min. Stretching improves the lateral order only marginally at the crystallite level (note the very little or no charge in the X-ray crystallinity). However, the order improvement at the molecular level seems to be slightly higher (see the infrared index values) especially for the fibers swollen and stretched in $ZnCl_2$.

Bundle Tenacity

The data in Table III show that slack swelling in $ZnCl_2$ (10 min) produces very little change in bundle tenacity (zero and 3 mm gauge lengths) and bundle extension in different cottons. However, Suvin cotton is an exception, showing a fall in tenacity at both gauge lengths with no change in extension. Samples swollen in slack conditions for 30 min showed tremendous decrease in both zero and 3 mm gauge strengths, the decrease in the latter being very high. In NaOH, the cottons behave quite differently. The tenacity at zero

TABLE I
Fiber Quality Data on Cotton Samples Chosen for the Study

Cotton	2.5% Span length (mm)	Fineness millitex	Percentage of mature fibers	Bundle Tenacity (g/tex)		Strength uniformity ratio (SUR)	Extension E (%)	50% X-ray angle (ψ) degrees
				Zero gauge	3 mm gauge			
Supriya	30.5	143	81	42.2	21.4	0.51	5.0	29.1
Hybrid 4(1)	29.9	154	70	36.4	19.3	0.53	6.2	32.7
Hybrid 4(2)	27.2	142	55	38.6	21.6	0.56	5.7	32.7
Varalaxmi	33.1	126	73	41.1	23.1	0.56	4.0	32.4
Jayadhar	24.2	181	78	45.2	24.5	0.54	4.0	23.7
Suvin	37.3	110	72	55.3	39.3	0.71	4.5	21.0

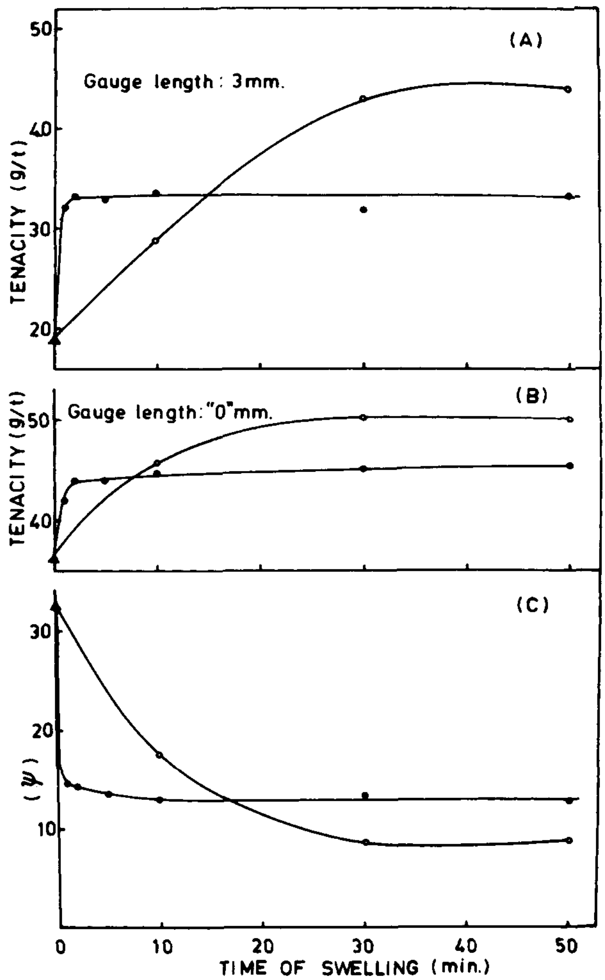

Fig. 1. Effect of swelling time on the orientation (ψ) and tenacity of swollen and stretched cotton fibers from Hybrid 4(1) (▲ control, ○ $ZnCl_2$ treatment, ● NaOH treatment).

TABLE II
Infrared and X-Ray Crystallinity Values of Swollen and Stretched Samples
Cotton: Hybrid 4(1)

Treatment	Crystallinity data: Total X-ray crystallinity	Crystallinity data: Infrared index
1. Nil	83	0.71
2. 64.5% (w/w) $ZnCl_2$, 32° ± 1°		
(a) Swollen slack, 10 min	74	0.61
(b) Swollen slack, 30 min	56	0.53
(c) Sample (a) Stretched to OL,	73	0.63
(d) Sample (b) Stretched to OL,	57	0.58
3. 21% (w/w) NaOH 31°C		
(a) Swollen slack, 10 min	67	0.57
(b) Sample (a) Stretched to OL	67	0.57

OL = Original length.

TABLE III
Bundle Tenacity and Orientation Data for the Swollen and Stretched Fibers from different Varieties of Cotton

Cotton	Treatment	T_0 (g/tex)	T_3 (g/tex)	SUR	E%	ψ°
Supriya	1. Nil	42.2	21.4	0.51	5.0	29.1
	2. 64.5% (w/w) $ZnCl_2$ 32° ± 1°C					
	(a) Swollen slack, 10 min	40.4	21.0	0.52	4.6	25.8
	(b) Swollen slack, 30 min	21.6	13.2	0.61	7.6	20.1
	(c) Sample (a) Stretched to OL	41.2	29.7	0.72	3.5	15.2
	(d) Sample (b) Stretched to OL	44.2	39.4	0.89	4.0	8.4
	3. 21% (w/w) NaOH, 31°C					
	(a) Swollen slack, 10 min	32.1	24.1	0.75	13.4	23.4
	(b) Sample (a) Stretched to OL	36.8	29.9	0.81	5.0	14.0
Hybrid 4(1)	1. Nil	36.4	19.3	0.53	6.2	32.7
	2. 64.5% (w/w) $ZnCl_2$ 32° ± 1°C					
	(a) Swollen slack, 10 min	36.8	20.4	0.55	5.5	27.6
	(b) Swollen slack, 30 min	25.8	14.6	0.57	7.7	21.3
	(c) Sample (a) Stretched to OL	45.9	28.9	0.63	3.4	18.0
	(d) Sample (b) Stretched to OL	50.2	43.0	0.86	4.0	8.6
	(e) Swollen slack, 50 min and Stretched to OL	50.2	44.0	0.88	4.3	9.0
	3. 21% (w/w) NaOH, 31°C					
	(a) Swollen slack, 10 min	34.9	24.4	0.70	12.5	23.6
	(b) Sample (a) Stretched to OL	44.5	34.1	0.77	5.2	13.1
Hybrid 4(2)	1. Nil	38.6	21.6	0.56	5.7	32.7
	2. 64.5% (w/w) $ZnCl_2$ 32° ± 1°C					
	(a) Swollen slack, 10 min	41.2	20.7	0.50	5.4	23.6
	(b) Sample (a) Stretched to OL	41.4	35.5	0.86	3.8	10.7
	(c) Swollen slack, 30 min and Stretched to OL	42.0	38.3	0.91	3.6	9.2
	3. 21% (w/w) NaOH, 31°C					
	(a) Swollen slack, 10 min	34.0	22.0	0.65	12.2	28.1
	(b) Sample (a) Stretched to OL	35.2	35.2	1.00	5.2	13.5
Varalaxmi	1. Nil	41.1	23.1	0.56	5.2	32.4
	2. 64.5% (w/w) $ZnCl_2$ 32° ± 1°C					

Cotton	Treatment	T_0 (g/tex)	T_3 (g/tex)	SUR	E%	$\psi°$
	(a) Swollen slack, 10 min	40.1	22.2	0.55	5.2	25.2
	(b) Swollen slack, 30 min	23.1	15.3	0.66	6.9	18.6
	(c) Sample (a) Stretched to OL	50.2	33.4	0.67	3.7	17.7
	(d) Sample (b) Stretched to OL	52.8	44.6	0.84	4.2	9.2
	3. 21% (w/w) NaOH, 31°C					
	(a) Swollen slack, 10 min	33.2	25.3	0.76	12.2	24.9
	(b) Sample (a) Stretched to OL	44.1	37.8	0.86	5.1	11.7
Jayadhar	1. Nil	45.2	24.5	0.54	4.0	23.7
	2. 64.5% (w/w) $ZnCl_2$ 32° ± 1°C					
	(a) Swollen slack, 10 min	44.3	25.8	0.58	4.0	21.2
	(b) Sample (a) Stretched to OL	48.6	25.5	0.52	4.1	19.5
	(c) Swollen slack, 30 min and Stretched to OL	50.5	41.3	0.82	2.8	9.2
	3. 21% (w/w) NaOH, 31°C					
	(a) Swollen slack, 10 min	35.1	23.5	0.67	9.9	22.1
	(b) Sample (a) Stretched to OL	45.7	35.4	0.77	4.0	10.4
Suvin	1. Nil	55.3	39.3	0.71	4.5	21.0
	2. 64.5% (w/w) $ZnCl_2$ 32° ± 1°C					
	(a) Swollen slack, 10 min	43.3	28.8	0.67	4.5	19.0
	(b) Swollen slack, 30 min	26.7	17.4	0.65	5.9	19.2
	(c) Sample (a) Stretched to OL	51.5	38.6	0.75	3.7	14.4
	(d) Sample (b) Stretched to OL	52.2	46.3	0.89	3.6	9.0
	(e) Swollen slack, 50 min, and Stretched to OL	57.9	50.1	0.87	4.5	8.4
	3. 21% (w/w) NaOH, 31°C					
	(a) Swollen slack, 10 min	45.7	38.0	0.83	14.6	19.8
	(b) Sample (a) Stretched to OL	55.7	54.5	0.98	6.0	9.2

gauge length is drastically reduced, while at 3 mm it shows either an increase or a marginal decrease. Extension values show considerable increase.

Stretch to original length, after a 10 min swelling in $ZnCl_2$, showed considerable improvement in bundle tenacity at both the gauge lengths. When stretching was preceded by a 30 min swelling period, a spectacular increase in tenacity at 3 mm gauge could be observed for all the varieties except Hybrid 4(2), which showed only a marginal increase. This probably indicates that the predominantly immature sample could swell to the optimum level in the shorter interval of 10 min itself. It may be noted that in some cases, strength at 3 mm gauge recorded almost a 100% increase over that of the untreated samples. Equally notable is the trend in the spread of the bundle strength values. While in the untreated cottons, the spread in tenacity at 3 mm gauge length is 20 g/tex, after optimal swelling in $ZnCl_2$ it comes down to 8 g/tex. The reduction in spread of tenacity at zero test length is not so impressive. With NaOH swelling followed by stretching also, the tenacity is increased at both test lengths, the increase at 3 mm being more notable. However, the spread in tenacity at 3 mm gauge length for the different varieties does not show any decrease. Strength uniformity ratios (SUR), also given in Table III, show considerable increase after swelling and stretching in both the reagents.

Fibrillar Orientation

Swelling in $ZnCl_2$ brings about considerable reduction in the ψ values. When stretched to original length, the X-ray angle reduces further (see Table III). The normalized intensity profiles for the 002 reflection for the variety Hybrid 4(1) before and after swelling and stretching in the two reagents are shown in Figure 2. Note the very narrow profile (C in Fig. 2) obtained for the

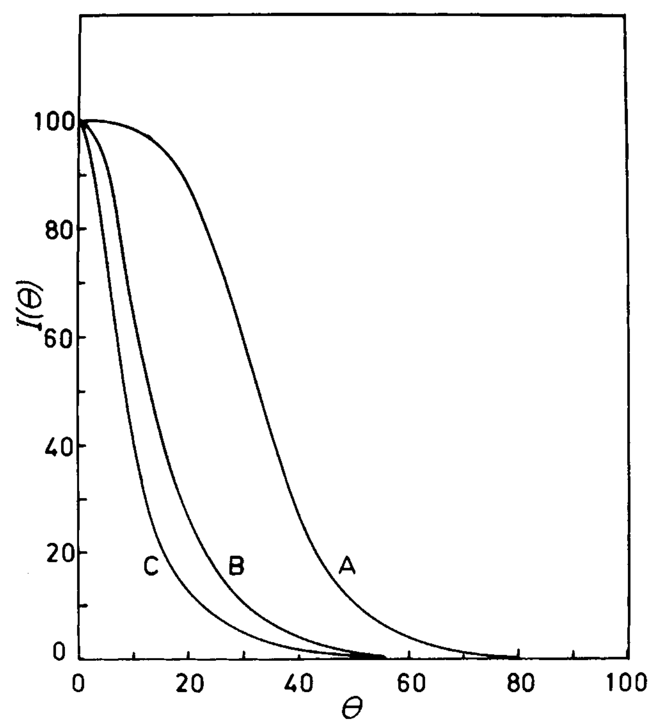

Fig. 2. Peak-normalized orientation profiles of the 002 arc from fibers of Hybrid 4(1): (A) untreated (B) swollen in NaOH and stretched to OL, and (C) swollen in $ZnCl_2$ for 30 min and stretched to OL.

TABLE IV
Correlation of X-ray Angle (ψ) with Tenacity (T_0 and T_3) and Extension (E%) for Fibers from Different Varieties of Cotton Swollen and Stretched in NaOH and $ZnCl_2$

	Correlation coefficient	
Pair of properties	After NaOH treatment	After $ZnCl_2$ treatment
ψ, T_0	−0.35	−0.58
ψ, T_3	−0.82	−0.89
ψ, E%	+0.24	+0.75

fibers after optimal swelling and stretching in $ZnCl_2$. It is also significant to note that when the fibers are stretched after 30 min swelling, the X-ray angles of all the six samples of cotton reach a nearly equal value of 8–9°, which is close to that found in ramie fiber, known for its high fibrillar orientation. In the case of NaOH treatment, even after stretching to original length, the samples show a certain range for the X-ray angles (9–14°).

Correlations

Correlation coefficients of ψ with tenacity at zero gauge (T_0), tenacity at 3 mm gauge (T_3), and E% were worked out and are shown in Table IV. Control and stretched samples of all varieties are included in working out these correlations. It may be noted that with $ZnCl_2$ treatment, ψ shows a negative correlation with both T_0 and T_3, while the actual value of the correlation coefficient is much higher between ψ and T_3 (−0.89) than that between ψ and T_0 (−0.58). ψ shows a positive association with E% ($r = 0.75$). For NaOH treatment also, ψ showed a good correlation with T_3 ($r = -0.82$), while with T_0 and E% very poor correlations are obtained ($r = -0.35$ and $r = +0.24$, respectively). High correlation between ψ and T_3 suggests the uniformity introduced in the samples by the swelling and stretching treatments.

DISCUSSION

A comparison of the data (Table III) for fibers treated slack in the two reagents will show that, the wide spread in T_3 and ψ values that existed among the varieties narrowed down considerably on treatment with $ZnCl_2$. This effect is very well brought out by the set of curves shown in Figure 3 (A, B) where both tenacity and ψ values clearly show a tendency to converge after 30 min swelling. A similar graphical representation for the NaOH-treated samples is not possible as the swelling phenomenon in this case is almost independent of time. However, it is obvious from the table that for the NaOH-treated samples, the variation in strength as well as orientation among the varieties still persists.

Under optimum conditions, the fibers swell much more in $ZnCl_2$ than in NaOH, resulting in a greater loosening of the fibrillar elements and higher fiber shrinkage. Higher longitudinal contraction causes consequent increase in fiber diameter. This in turn leads to greater separation between fiber layers,

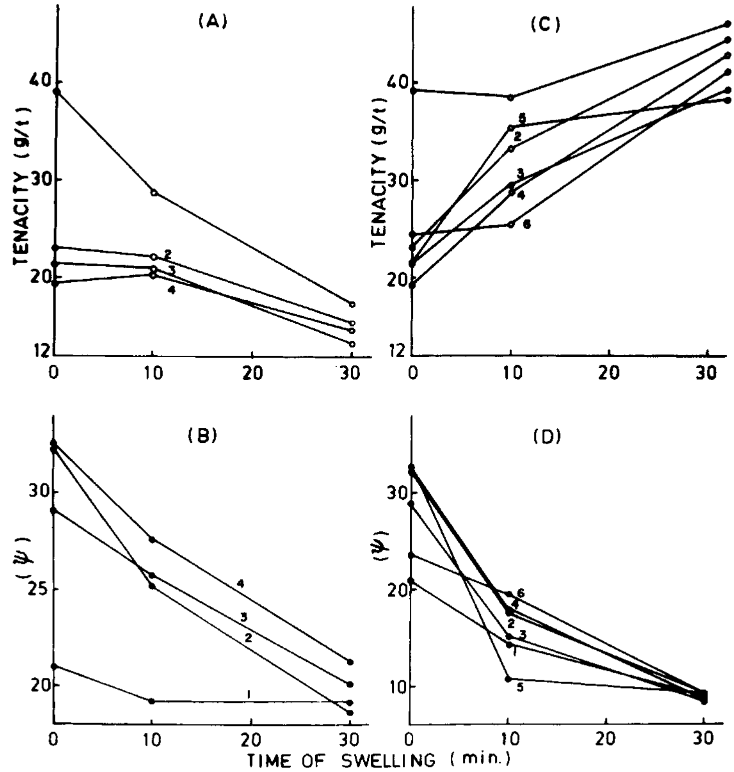

Fig. 3. Dependence of orientation (ψ) and tenacity (3 mm gauge length) on the swelling time for different varieties of cotton after (A and B) swelling and (C and D) swelling and stretching in $ZnCl_2$. [1: Suvin, 2: Varalaxmi, 3: Supriya, 4: Hybrid 4(1), 5: Hybrid 4(2) and 6: Jayadhar.]

with consequent weakening of interlamellar and interfibrillar linkages. As a result, the bundle tenacity drops considerably at higher gauge lengths, without enhancement of extension despite high shrinkage (note the lower extension in Table III for the $ZnCl_2$ swollen samples than those for NaOH).

Swelling in NaOH produces less shrinkage (about 10–15% as compared to 30–40% in $ZnCl_2$), and hence lower disruption of fibrillar elements, which contribute to strength at ultrastructural level. In other words, the disturbance to the structure produced by NaOH swelling is less drastic and acts favorably in relieving strain when stress is applied over higher gauge lengths.

On stretching to original length, $ZnCl_2$-treated samples acquired much higher bundle tenacity values and better fibrillar orientation levels than did fibers similarly treated in NaOH. Moreover, both bundle tenacity (3 mm gauge length) and ψ values converged considerably [Fig. 3 (C, D)]. The convergence of ψ values was only partial for the NaOH-treated samples, while the bundle tenacity spread still persisted.

During application of stretch to the slack swollen fibers, the disrupted structural elements tend to get oriented along the fiber axis. In addition, the lamellar separation tends to approach that in the raw sample, as the fibers have now been pulled along the axis to their original length. Since the fiber structure becomes more flexible in the swollen state in $ZnCl_2$, during stretching, the fibrillar elements are free to slide over each other and would align themselves in a better way resulting in higher orientation as indicated by the very low ψ values. This also helps to improve the lateral order at the molecular level (see the IR index for the $ZnCl_2$ stretched samples in Table II). It is basically the improvement in orientation that contributes to the increase in strength at 3 mm gauge as indicated by the high correlation (r = −0.89) between strength and orientation for the different varieties. It has been shown that slack swelling in $ZnCl_2$ results in considerable lowering of the crystallite size.[3] Since during stretching, there is very little improvement in the lateral order at the crystallite level, it may be inferred that the crystallite size too, remains more or less the same. Lowering of the crystallite size by swelling can also contribute to the ability of the fiber to withstand load as the crystalline elements will now be more flexible. Because of the above mentioned facts, swelling and stretching in $ZnCl_2$ could result in almost a 100% increase in strength at 3 mm gauge length.

All the varieties of cotton swell almost to the same extent in $ZnCl_2$ leading to the same final fibrillar orientation angle on stretching, which is lower than that obtained after a similar treatment with NaOH. Only in the case of Suvin cotton (*G. barbadense*) does NaOH produce the same swelling as in $ZnCl_2$ as revealed by the ψ value obtained after stretching ($\psi = 9°$ for both the reagents). Among the different varieties selected for study, this cotton has the highest strength to begin with. As a result, swelling and subsequent stretching produced only about 25% increase in 3 mm gauge strength compared to 75-100% in most of the other varieties. This peculiarity of the barbadense cottons has been noted by earlier workers[11] during swelling in NaOH. The variation in ψ for the different varieties swollen and stretched in NaOH clearly indicates that each cotton swells to a different extent, and hence the fibrillar elements get aligned differently on stretching, thus demonstrating the varietal response of cottons to NaOH treatment unlike in the case of $ZnCl_2$.

CONCLUSIONS

Stretching of cotton fibers after optimal swelling in $ZnCl_2$ could produce fibers with comparable strength uniformity as that obtained with a similar treatment in NaOH. However, the bundle tenacities for the $ZnCl_2$-treated fibers both at zero and 3 mm gauge lengths were substantially higher. Bundle tenacity at 3 mm gauge length and ψ were found to be strongly associated for fibers swollen and stretched in both the reagents. A nearly constant value for ψ and a narrow range for bundle tenacity at 3 mm gauge length for the swollen and stretched fibers indicate that different varieties of cotton respond identically to $ZnCl_2$ swelling unlike in NaOH where varietal response is prevalent.

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