Studies on Swelling of Cotton Fibers in Alkali Metal Hydroxides. II. Influence of Morphology and Fine Structure on Tensile Behavior

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Synopsis

Cotton fibers varying widely in gravimetric fineness but having nearly same percentage mature fibers have been subjected to swelling in 4.5*N* LiOH, NaOH, and KOH at room temperature and 0°C. The resultant changes in fine structure are analyzed by X-ray diffraction and infrared absorption methods while variations in surface morphology are followed by scanning electron microscopy. Extent of swelling measured by changes in gravimetric fineness follows the order LiOH ≥ NaOH ≥ KOH, the exact gradation being dependent on the variety. Analysis of tensile data shows that whereas moderate swelling leads to an increase in tenacity at 3.2 mm gauge length, excessive swelling leads to a decrease of the same, the extent of decrease being a function of swelling. KOH treatment produced uniform swelling and gave better retention of T_0 and T_3 for all varieties. Tensile behavior after slack swelling in the various reagents could be interpreted on the basis of fine structural variations produced by them. However, the differential response of cottons to a swelling agent is explained by postulating variations in the packing of structural elements along the radii.

INTRODUCTION

It has been reported[1-4] that swelling power of the alkali metal hydroxides depends on the cationic size in addition to concentration and temperature of the reagent. In an earlier communication,[5] we noted that the structure–property relations of the treated samples, though showing the same general trend, had some specificity to the swelling agent as well. It was also observed that treatment with potassium hydroxide (KOH) resulted in better retention of zero gauge tenacity (T_0) for the same 3.2 mm gauge tenacity (T_3) under identical conditions. This finding, we feel, has a long range technological potential as most of the fiber properties related to structure of the swollen fibers are comparable. However, earlier studies[6-8] on cotton fibers treated with sodium hydroxide (NaOH) showed that the fiber fineness and maturity play an important role in swelling and resultant structure and properties. Hence in the present investigation we have selected cotton fibers belonging to different species, having widely different initial fineness but nearly the same maturity, and swollen these samples in an optimum concentration of KOH at two different temperatures viz., ambient temperature (30 ± 1°C) and 0° (−2 ± 2°C).

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For comparison, the treatments were carried out in NaOH, the commercial mercerizing agent, and in LiOH, the swelling agent with the smallest cationic size. Tensile, physical, and fine structural properties of the treated fibers have been measured for all the samples. Surface morphology of the fibers has been examined for a few typical samples as it is likely that changes in the external form might be related to changes in internal structure. Results are presented, and the variations in tensile properties of the swollen samples have been suitably explained based on fine structural and morphological changes.

EXPERIMENTAL

Materials

Cotton fibers selected from different genetic varieties and kiered with 1% alkali solution have been used in the study. The hydroxides of lithium, sodium, and potassium used were of analytical grade.

Treatment with Alkali Metal Hydroxides

Normalities of the alkali solutions prepared by dissolving the appropriate hydroxides in distilled water were adjusted by standard titration techniques. About 2 g each of the fibers were treated slack with 4.5N solution of the hydroxides both at room temperature (30°C) and at 0°C (precooled alkali solutions were used here) for about 10 min. At the end of the period, the fibers were washed free of alkali, scoured in 2% (v/v) acetic acid, washed further, and air-dried.

Measurements

All the samples were conditioned at 65% RH and 27°C prior to measurements.

Tensile Properties. Bundle tenacities both at nominal zero gauge length (T_0) and 3.2 mm gauge length (T_3) as well as extension ($E\%$) for the fibers were measured with an Instron tensile tester using Pressley fiber clamps, and the values were corrected based on data for standard calibration cotton tested simultaneously as per standard procedure.[9]

Physical Properties. Gravimetric fineness and moisture regain values for the samples were measured as per usual methods.[9]

Crystallinity and Crystallite Dimensions. Radial intensity scans from powder samples were obtained as per established methods described earlier.[10] Cellulose II (C II) and amorphous (Am) contents were computed by following the procedure of Chidambareswaran et al.[11] The half-maximum breadth of the strong (200) reflection (conventions used for indexing of planes is the same as that given in Ref. 5) was computed for the treated samples using the breadth of (200) reflection measured from the higher angle (2θ) side in order to avoid the interference from the strong (110) reflection. Meridional scans were obtained using a specimen holder specially fabricated for the purpose. Highly cohesive bundles of fibers were packed into the rectangular array of pinholes (1 mm diameter) in the holder and cut flush with the surface of the holder. This method is essentially the same as that used by the earlier workers,[12,13]

except for some minor modifications. The meridional (004) reflection was obtained employing wider slit geometry and better counting statistics, and the half-maximum breadth was computed.

Crystallite Orientation. From the orientation scan obtained using well-parallelized bundle of fibers at 2θ corresponding to (200) reflection, the orientation parameter $1/\phi_{1/2}$ proposed by Warwicker et al.[14] was computed. Here $\phi_{1/2}$ is the 50% X-ray angle measured as the half-maximum breadth from the azimuthal intensity distribution of the (200) reflection.

Infrared Spectra. Infrared spectra of the cut powders of the treated samples embedded in the potassium bromide (KBr) matrix were recorded with a Perkin-Elmer Model 457 infrared spectrophotometer. Intensities of the bands were measured using the base line technique. Both the crystallinity indices proposed by O'Connor and co-workers,[15,16] viz., index I and index II, were computed for a few of the treated samples.

Surface Morphology. Fibers drawn from treated samples were suitably mounted on specimen stub and coated with gold using "cool" sputter coater E 5100 and were scanned under the scanning electron microscope (SEM 150). Micrographs showing typical changes in fiber morphology were recorded.

RESULTS AND DISCUSSION

Initial fiber properties of the different cottons selected for the study are contained in Table I. It may be noted that the cottons vary widely in gravimetric fineness, orientation, and strength; but they have nearly the same percentage mature (P_m) fibers. Cottons varying in fineness were selected so as to see the effect of secondary cell-wall morphology on swelling and resultant change in properties.

The tensile properties of the treated samples belonging to the four different varieties of cottons are summarized in Table II while the physical and structural data of these samples are given in Table III.

At room temperature, while T_0 values show a decrease for all the varieties for both LiOH and NaOH swelling in the order NaOH $\geq$ LiOH, KOH swelling almost had the same T_0 except for the cotton Suvin (Table II). However, irrespective of the swelling agent used, T_3 shows increase in all the cottons except for NaOH-treated Suvin, which shows a small decrease in T_3. In general, the treatments with LiOH and KOH show greater improvement in T_3 than for NaOH swelling, especially for the finer cottons. It may be observed

TABLE I
Fiber Properties of the Various Cottons Chosen for the Study

Variety of cotton	2.5% span length (mm)	Tensile data			Gravimetric fineness (mtex)	Percentage mature fibers (P_m)	$1/\phi_{1/2}$ (deg^{-1})
		T_0 (g/tex)	T_3 (g/tex)	E(%)			
Suvin	37.3	55.3	33.2	4.5	110	72	0.045
PSH	37.6	48.8	26.2	5.0	130	67	0.038
Khandwa	25.5	46.6	18.7	4.9	169	75	0.038
AK.235	23.5	43.1	17.7	5.0	230	75	0.043

TABLE II
Bundle Tenacity (g/tex), Elongation (E%) and Crystallite Orientation, $1/\phi_{1/2}$(deg^{-1}) for Different Cottons Treated with 4.5N LiOH, NaOH, and KOH

Treatment	Suvin				PSH				Khandwa				AK.235			
	T_0	T_3	E (%)	$1/\phi_{1/2}$	T_0	T_3	E (%)	$1/\phi_{1/2}$	T_0	T_3	E (%)	$1/\phi_{1/2}$	T_0	T_3	E (%)	$1/\phi_{1/2}$
Nil	55.3	33.2	4.5	0.045	48.8	26.2	5.0	0.038	46.6	18.7	4.9	0.038	43.1	17.7	5.0	0.043
LiOH[a]	47.9	36.4	10.7	0.048	46.5	30.8	9.4	0.041	43.3	21.4	7.6	0.042	39.3	21.8	8.2	0.040
NaOH[a]	43.7	31.0	11.6	0.051	45.5	28.1	9.8	0.043	42.8	21.4	7.4	0.044	39.5	20.4	8.4	0.043
KOH[a]	49.4	37.0	10.1	0.051	50.7	31.6	8.4	0.042	44.7	21.2	8.0	0.046	42.9	20.5	7.1	0.042
LiOH[b]	37.0	24.4	9.3	0.047	35.3	18.4	12.1	0.040	37.6	19.3	9.4	0.041	33.9	18.4	9.6	0.042
NaOH[b]	40.3	28.6	10.3	0.051	41.5	27.4	12.7	0.043	38.2	20.5	9.0	0.042	34.6	20.4	9.6	0.044
KOH[b]	43.3	32.2	12.0	0.049	45.9	32.3	14.6	0.040	43.3	23.4	10.2	0.038	40.2	22.0	10.4	0.041

[a]At room temperature.
[b]At 0°C.

TABLE III
Physical and Structural Data for Cottons Treated with 4.5N LiOH, NaOH, and KOH

Treatment	Suvin				PSH				Khandwa				AK.235			
			X-ray data				X-ray data				X-ray data				X-ray data	
	Fineness (mtex)	MR[a] (%)	C II (%)	Am (%)	Fineness (mtex)	MR[a] (%)	C II (%)	Am (%)	Fineness (mtex)	MR[a] (%)	C II (%)	Am (%)	Finness (mtex)	MR[a] (%)	C II (%)	Am (%)
Nil	110	6.95	0	26	130	6.95	0	28	169	7.40	0	30	230	6.90	0	29
LiOH[b]	145	9.90	40	36	147	9.86	43	35	193	9.40	43	36	272	10.40	42	37
NaOH[b]	145	10.40	55	32	149	10.23	53	35	181	10.10	58	30	269	10.70	54	33
KOH[b]	139	10.40	38	41	143	9.95	38	41	190	9.80	38	40	270	10.40	38	41
LiOH[c]	165	11.20	44	39	172	11.06	44	39	209	10.50	47	41	299	11.10	47	40
NaOH[c]	157	10.35	52	33	166	10.47	52	36	192	10.00	56	33	303	11.10	54	34
KOH[c]	155	10.95	44	38	159	10.59	45	40	200	10.30	44	40	278	11.20	44	40

[a]Moisture regain measurements at 65% RH and 27°C.
[b]At room temperature.
[c]At 0°.

from Table III that swelling as measured by gravimetric fineness followed the order LiOH ≥ NaOH ≥ KOH, the exact gradation being decided by the initial fineness of the variety. Although we are aware that tex may not be a clear indicator of the actual swelling, the same should be considered as an appropriate technique that measures the swelling retained after drying. Values of amorphous (Am) content were found to be always higher for fibers swollen in KOH, irrespective of the extent of swelling while the conversion to cellulose II (C II) remained lower for KOH treatment as compared to that with NaOH.

Reducing the temperature of swelling to 0°C increases the swelling capability of all the reagents (note from Table III the increased tex values for all the varieties after 0°C swelling). It is known that lowering of temperature results in an increase in the degree of hydration of the alkaline reagents. In the case of a fully hydrated cation, at a given normality, LiOH has the maximum degree of hydration. Since swelling is closely related to the degree of hydration (which in turn depends on the concentration and temperature of swelling agent) at 0°C, LiOH can be expected to produce the maximum swelling at a given normality. This excessive swelling results in considerable reduction in tenacities, both T_0 and T_3 the maximum effect being observed for cottons belonging to *G. barbadense* species (PSH and Suvin). Swelling in NaOH and KOH also reduces T_3 in the case of Suvin, the decrease during NaOH swelling being much higher than in KOH.

For the other cottons, T_3, after swelling in NaOH, remains almost the same as that for the respective controls or shows a marginal improvement. On the other hand, after KOH swelling, T_3 always recorded an increase. Extension ($E\%$) also increased considerably especially after KOH swelling at 0°C. C II content registered an increase during LiOH and KOH treatment which was closely followed by moisture regain. Swelling, as measured by tex, retained almost the same order as that obtained at room temperature. Am values, which generally remained high during KOH swelling, showed only marginal changes on reducing the swelling temperature. $1/\phi_{1/2}$ showed very little variation throughout, indicating minimal variations in crystallite orientation, during slack swelling.

From the above results, it is apparent that fibers treated with KOH at 0°C, retain or show an increased T_3 strength in spite of increased swelling on going from ambient temperature to 0°C. Neither the change in crystallite orientation nor that in the Am content can account for this increased retention of strength at 0°C. An interesting observation made with KOH swelling is that even at room temperature, the Am values were the highest despite lower or equal swelling. This probably implies that KOH swelling results in creation of more lattice imperfections and number of sites of attack along the length of the fibrils. These combined with greater disruption of fibrillar elements at 0°C (due to increased swelling as made explicit by the higher tex values) should lead to reduced crystallite length. The half-maximum width (HMW) of the (004) meridional reflection (inversely proportional to crystallite length) offers a method of measuring the crystallite length. This measurement, although cumbersome, was attempted for one variety of cotton swollen both at 0°C and at room temperature. The values, along with other fine structural parameters, are presented in Table IV. The HMW of the equitorial (200) reflection, which

TABLE IV
Fine Structural Data for Cotton Fibers Swollen in Alkali Metal Hydroxides (Variety:PSH)

	X-ray data					IR Data	
				Half-maximum breadth 2θ (deg)			
Treatment	C I	C II	Am	(200)	(004)	Index I	Index II
Nil	72	0	28	1.40	0.31	3.022	0.717
LiOH[a]	22	43	35	1.60	0.33	1.115	0.643
NaOH[a]	12	53	35	2.00	0.35	0.738	0.665
KOH[a]	21	38	41	2.00	0.36	1.083	0.650
LiOH[b]	17	44	39	1.80	0.34	0.601	0.631
NaOH[b]	12	52	36	2.00	0.36	0.517	0.644
KOH[b]	15	45	40	2.20	0.41	0.840	0.614

[a] With 4.5N solution at room temperature.
[b] With 4.5N solution at 0°C.

is inversely proportional to the crystallite breadth, is also included in the table. Note that the width for both the reflections shows maximum values for fibers swollen in KOH at 0°C, indicating that KOH treatment results in maximum decrease in crystallite breadth and length under identical conditions of swelling. A similar conclusion was made by Zeronian and Cabradilla[3] regarding the length of crystallites, but by an indirect method (measurement of leveling of degree of polymerization).

From a study on structure–property relations for the alkali-treated samples at room temperature, we found[17] that T_3 is highly dependent on both Am and C II in the material, although changes in other fine structural parameters, such as amorphous orientation or DP, cannot be altogether neglected. However, when we consider samples having nearly the same (C II + Am) values, the differences in tensile behavior have to be accounted for by factors like change in crystallite length or any other possible variations in the fiber structure taking place at the molecular or fibrillar level.

In order to see whether the fine structural changes taking place at the molecular level show any differences among the swelling agents, apart from those observed at the crystallite level, relative intensity changes of a few infrared bands associated with decrystallization, conversion to C II and also to changes in intramolecular H bonding (index I) and total crystallinity (C I + C II) index II, were measured and the relationship of index I with the X-ray results is shown in Figure 1. Index I, which measures total effect of swelling, shows a good linear relation with (C II + Am) as measured by X-ray techniques. However, the values of index I, pertaining to room temperature treatment of all the swelling agents as well as that of KOH 0°C falls on one line while those due to LiOH and NaOH treatment at 0°C lie distinctly away from this line. This indicates that for the same change measured by the X-ray method, i.e., at the crystallite level, changes to the structure at molecular level including disturbance to the extent of H bonding is much higher. Thus a higher molecular order combined with various factors such as lower swelling, yet more uniform disruption of structural elements and lower crystallite

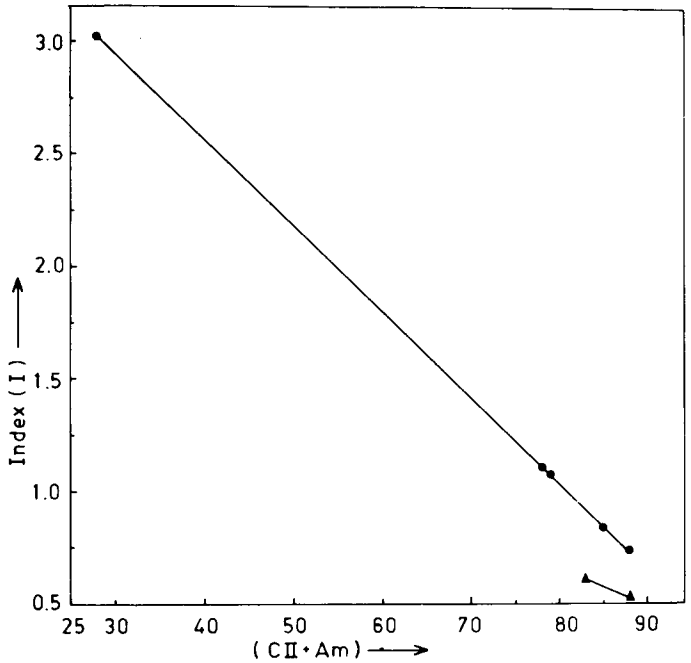

Fig. 1. Relationship of infrared index (I) with (C II + Am) from X-ray measurements.

dimensions, might be responsible for the increased retention of T_3 during swelling in KOH at 0°C.

Another interesting observation (Table II) is the very high decrease in T_3 for the cottons belonging to *G. barbadense* species, swollen in LiOH, on going from room temperature to 0°C, while for the cultivar of *G. arboreum* species, AK.235, the decrease is only marginal and the actual T_3 is close to that observed for the control. Fine structural parameters, especially Am, varied only marginally between 0 and 30°C swelling and also between varieties (Table II). Gravimetric fineness increased to the same extent for both PSH (*G. barbadense*) and AK.235 (*G. arboreum*). Infrared index I showed identical variation for both the varieties. Thus none of the measured properties differ between PSH and AK.235 so as to be able to explain the difference in tensile behavior between them.

The surface morphology of the fibers belonging to the varieties PSH and AK.235 before and after swelling as revealed by the scanning electron microscope are shown in Figures 2 and 3. A few things become obvious from these micrographs. (i) For the variety PSH, swelling in alkalies at 0°C, especially LiOH and NaOH, produces inhomogeneities in surface appearance. (ii) These inhomogeneities and surface ridges are more evident after LiOH swelling than NaOH while after KOH swelling fiber surface appears somewhat smooth and uniform. (iii) However, after swelling in all three reagents at room temperature, though the fiber weight/unit length has increased to different extents, surface appearance is almost identical. (iv) On the contrary for, the variety AK.235, even though the tex values are different for the two temperatures as well as at the same temperature, for all the three reagents, there is not much

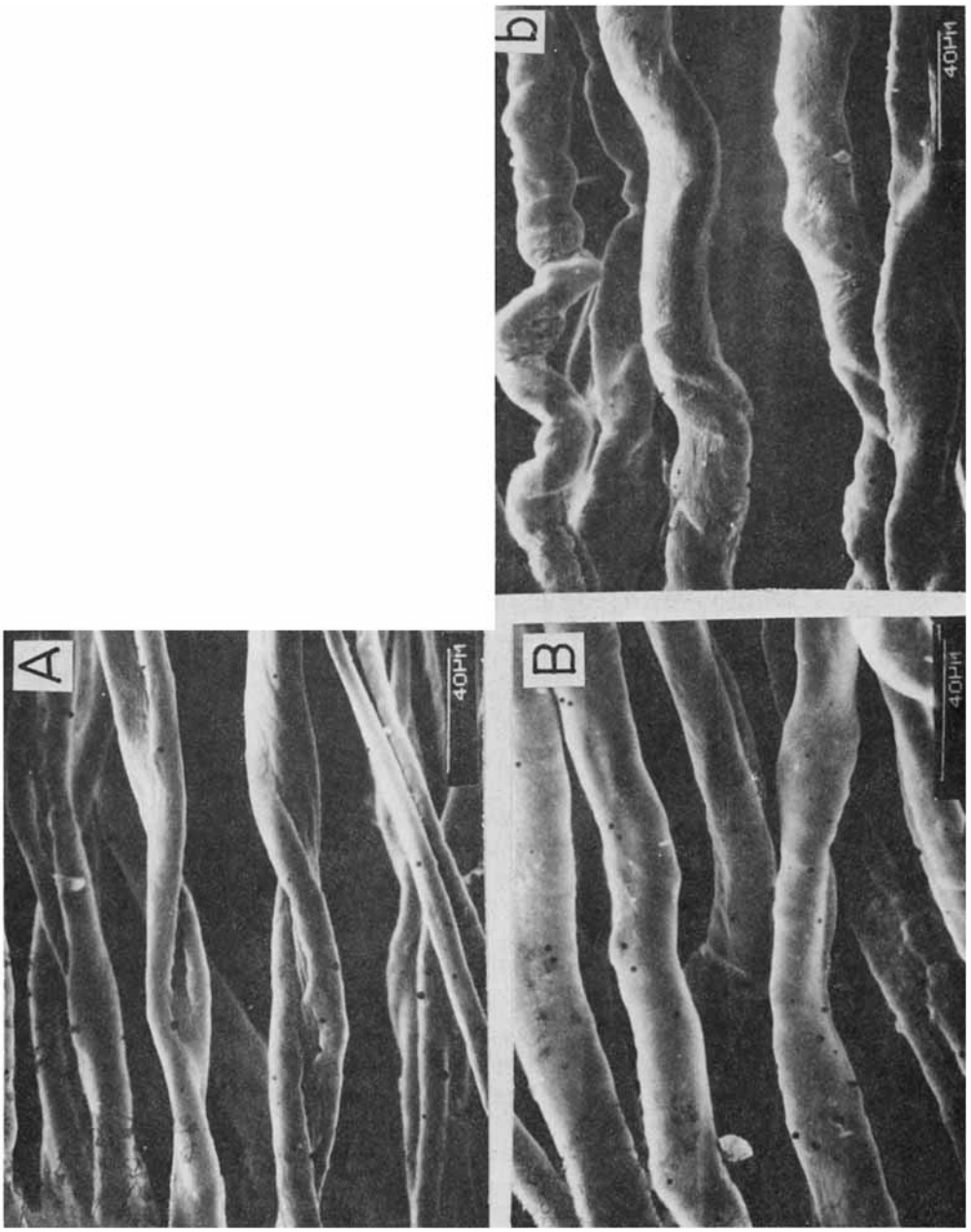

Fig. 2. SEM micrographs of PSH cotton: (A) control; (B, C, D) swollen at 30°C; (b, c, d) swollen at 0°C; (B, b, C, c, D, d) in 4.5*N* LiOH, NaOH, and KOH, respectively.

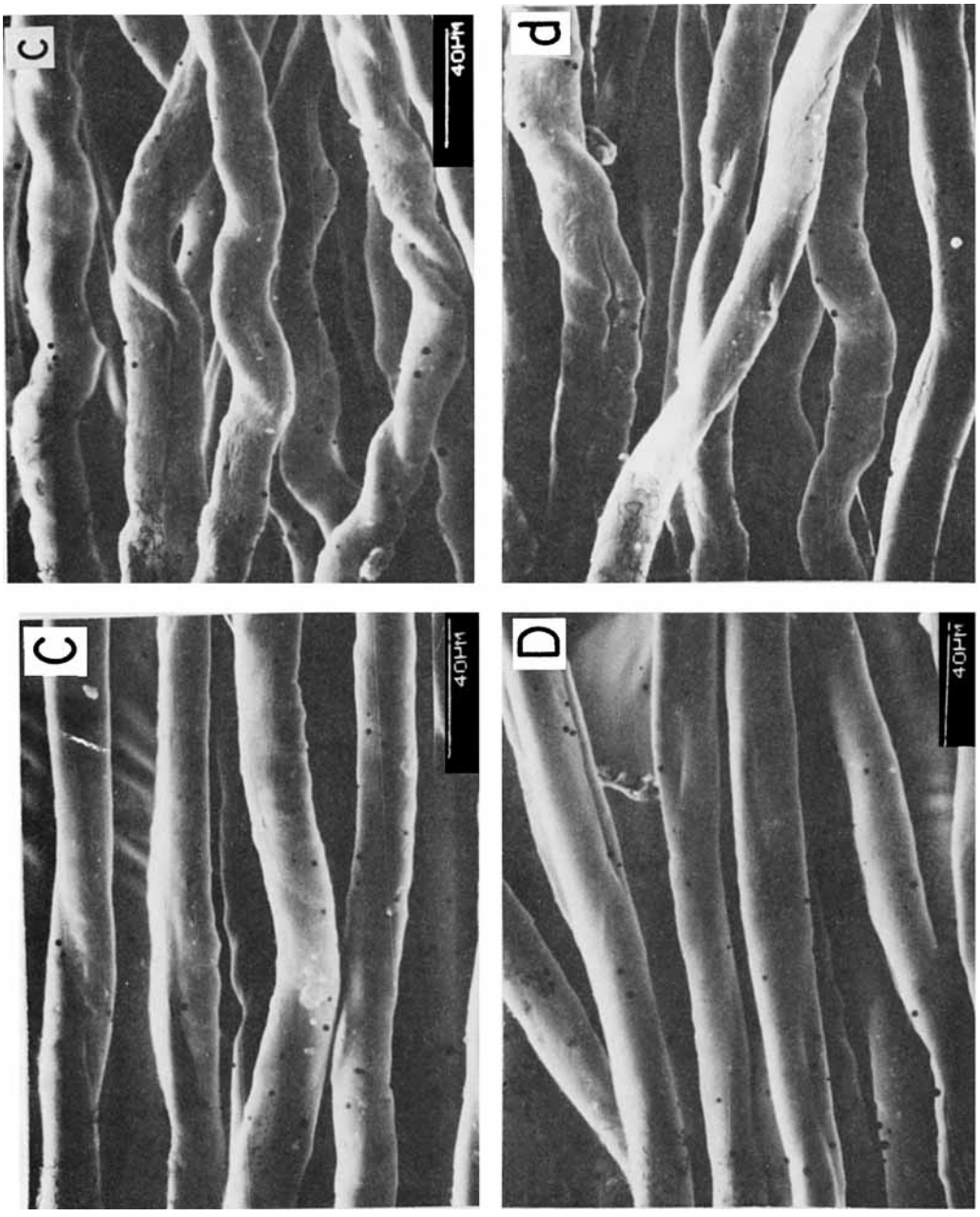

Fig. 2. *(Continued from the previous page.)*

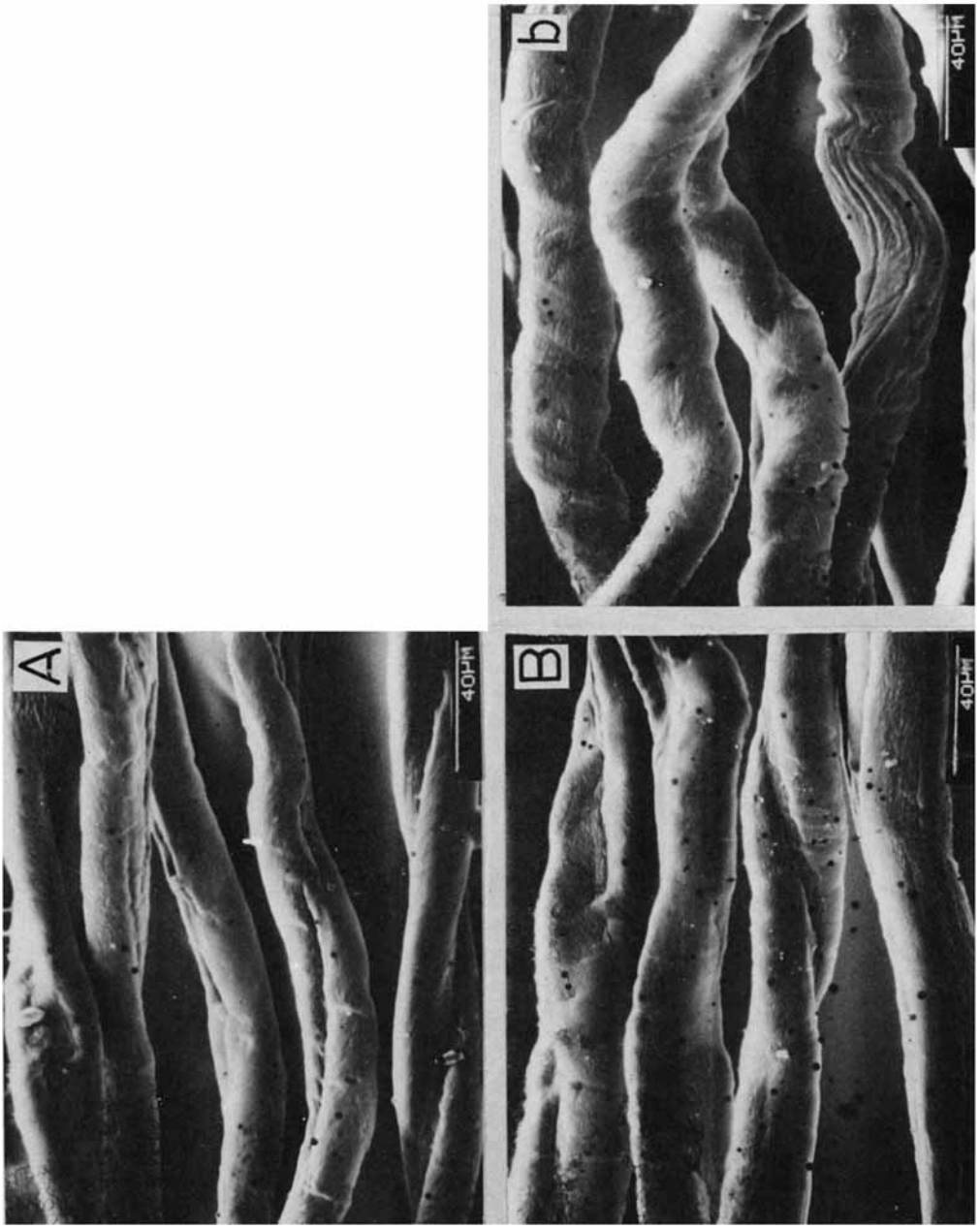

Fig. 3. SEM micrographs of AK.235 cotton: (A) control; (B, C, D) swollen at 30°C, (b, c, d) swollen at 0°C; (B, b, C, c, D, d) in 4.5*N* LiOH, NaOH, and KOH, respectively.

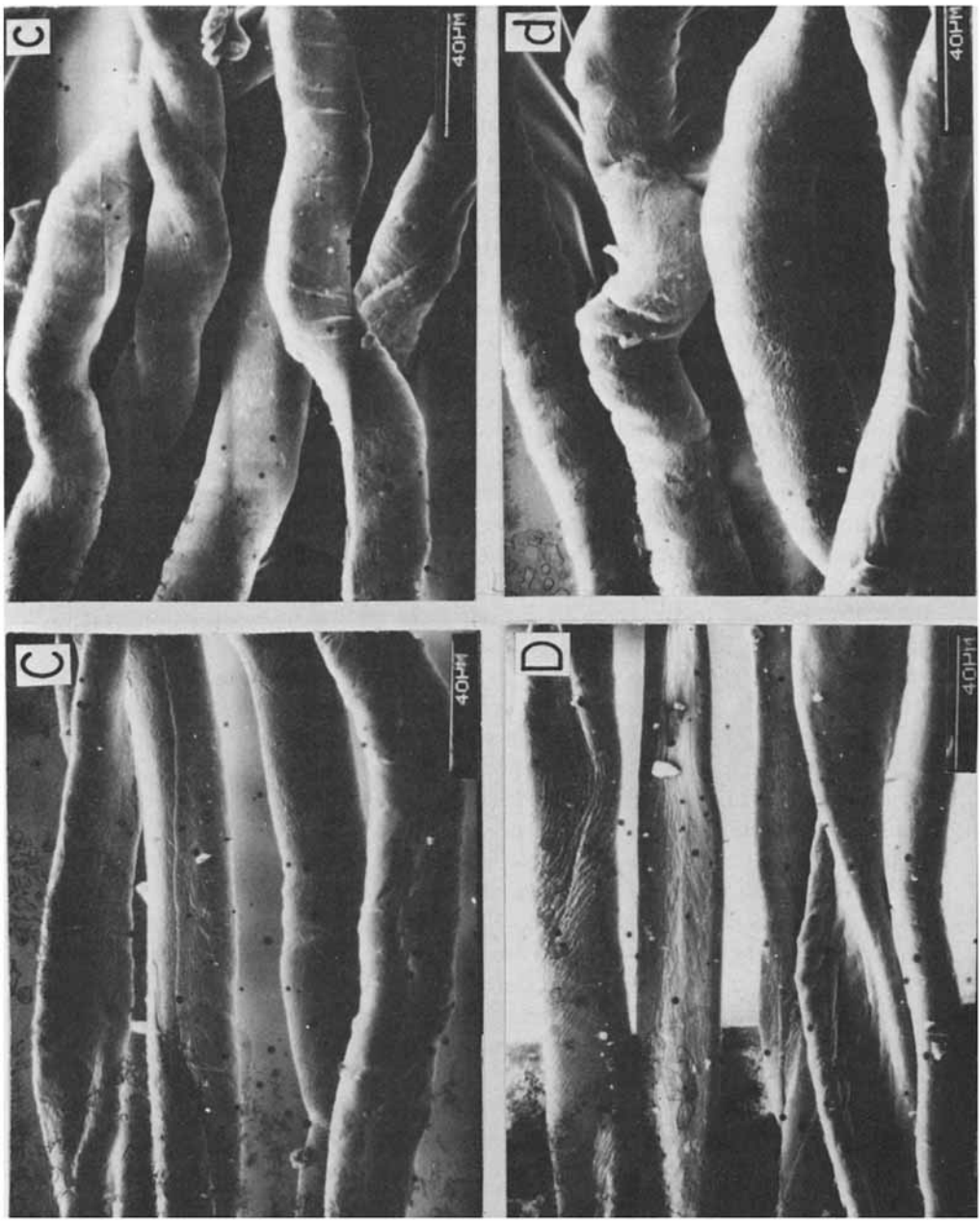

Fig. 3. *(Continued from the previous page.)*

difference in surface appearance on going from room temperature to 0°C even after LiOH treatment.

From morphological studies[18,19] of cell wall fragments carried out with a transmission electron microscope, it is well recognized that the secondary cell wall of cellulose fibers consists of macrofibrils of varying width resulting from the bundling of microfibrils arranged helically and connected radially by fibrils going from one bundle to the next. During swelling with intracrystalline reagents such as the ones used in the present study, inter- and intrafibrillar linkages are broken, leading to disturbance of fibrils from their original dispositions, depending on how drastic the penetration (decided by concentration and temperature) and swelling power (dependent on cationic size) of the reagent are. When the fibers are dried after washing out the reagent, fibrils cannot fully restore to their original state as the new linkages formed now fix them in a different disposition. However, when the same reagent under identical conditions of treatment affects differently two different varieties of cotton, as made apparent in the present study, not only by their different mechanical behavior, but also by their surface morphology (external form), one is intuitively led to think that there should exist some basic differences in the arrangement of internal structural elements along the radii.

It may be recalled[20] that differences have been reported by earlier workers in the number of growth layers among early-season and late-season cottons, in addition to the layer width (layer may be identified with macrofibrillar bundles). Studies on cross-sectional morphology[21,22] of different varieties of cotton show a higher circularity index for the *G. arboreum* than for the *G. barbadense*. For a given perimeter a circle can enclose the maximum area and hence a nearly circular cross-section will ensure more space for packing of structural elements. The characteristic bean-shaped cross section of the barbadense varieties would suggest less packing space and higher packing density. A closer packing of the structural elements can also contribute to the higher initial tenacities observed for these cottons. When the swelling pressure produced by the swelling agent is low, as in room temperature swelling, the bean-shaped cross section tends to become circular[23] and the packing becomes loose and more uniform. The reduction in strength that might arise from the increased separation among the structural elements is mostly off set by the release of inherent strain in the fibers produced during growth and drying. However, if the cross sections are nearly circular as it is the case with the *G. arboreum* cottons, during moderate swelling, the loosening of the structure will not be as high as that for the *G. barbadense* type and when the strains are released by the action of the swelling agent increase in tenacity at higher gauge length would result.

When the swelling is excessive as it happens at 0°C in LiOH, the swelling pressure developed in a closely packed structure may push the elements radially in either directions (towards the center as well as the periphery). This could lead to bulging and unevenness (and even cracking in the extreme case) of the external surface. If the changes noticeable in surface appearance can be taken to be reflections of changes occurring within the fiber, it is easy to infer that excessive swelling by LiOH at 0°C, has now introduced more nonuniformities or weak links internally as well than was originally present, resulting in a decrease in T_3. However, if the packing of the elements along the radii is

poorer or less close in a particular variety, then even during 0°C LiOH swelling, even though the elements are pushed radially, such a drastic effect would not occur on the surface as well as internally, as there is sufficient room for realignment within the cell wall. Hence, it is unlikely that in these varieties, the internal nonuniformities or "weak links" show an increase even during excessive swelling. Consequently, T_3 strength might remain unaltered.

It is thus apparent that although the tensile behavior of the alkali-swollen cotton fibers follows closely the fine structural changes that accompany swelling, performance of different varieties of cotton to a given chemical treatment is dependent on the secondary cell-wall morphology and its variations.

CONCLUSIONS

While slack swelling at moderate level helps to remove the weak links and improves T_3, excessive swelling effected by LiOH and NaOH at 0°C introduces additional nonuniformities in the fiber, causing a reduction in T_3. The decrease in T_3 is found to depend on the variety. KOH produces more uniform swelling both at 0°C and room temperature as is evident from the surface morphology and the higher retention of T_0 and T_3.

Further, the lower the gravimetric fineness of the cottons, the greater is the superiority of KOH treatment. Differences observed in the fiber surface morphology after 0°C swelling between the *G. arboreum* and *G. barbadense* varieties suggest some basic variation in the packing of structural elements between them.

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