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Tensile Properties of Cotton Treated with Alkali Metal Hydroxides

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Synopsis

The tenacity of cotton fibers treated without tension with 5*N* KOH at 0°C or 21°C is greater than that of fibers treated with either 5*N* LiOH or NaOH. In studying the effect of changes in fine structure on the strength of cotton fibers, we suggest that strength should be given as breaking load rather than as tenacity. Evidence is given indicating that crystallite length is an important factor and should be considered with degree of crystallinity and degree of orientation when explaining the increase in breaking load of cotton fibers brought about by treatment with 5*N* alkali metal hydroxides.

INTRODUCTION

We have previously presented evidence indicating that for a given concentration and given reaction temperature, the swelling and fine structure of the product obtained by treating cotton with an aqueous solution of an alkali metal hydroxide is affected by the size of the cation in the reagent.[1,2] In the study reported here, we extended our investigation to compare the effect of different alkali metal hydroxides on the tensile strength of cotton fibers and thus examine factors of the supramolecular structure which affect the tensile properties of mercerized cotton fibers.

EXPERIMENTAL

Materials. Deltapine Smooth Leaf cotton fibers scoured 1 hr in 2% sodium hydroxide solution were used.

Methods of Treatment. The cotton fiber was swollen in the slack state with alkali metal hydroxide and then was washed by the procedure described previously.[1,2]

Characterization of Product. Tensile tests were made using a Stelometer at a loading rate of 1 kg/sec, with the bundle size adjusted to produce the average break at about 5 kg. The tests (6 per sample) were made at 65% R.H. and 21°C. To minimize the effect of fiber structure on strength, the tests were made without a spacer and thus were nominally at zero-mm length. Tex was calculated from the weight of a bundle of fibers cut to a known length and the number of fibers in the bundle. Samples were conditioned at

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65% R.H. and 21°C. Orientation of the crystallites in the fibers was measured by the 50% x-ray angle as described by Creely et al.[3]

RESULTS AND DISCUSSION

In most of the experiments reported here, the cotton had been treated with alkali metal hydroxide of 5*N* concentration. This concentration was selected because we previously had shown[2] that treatment of cotton in the slack state with 5*N* LiOH, NaOH, or KOH at 0°C will cause substantially complete conversion of the lattice structure of the fiber from cellulose I to cellulose II. The rest of the experiments were done with cotton fiber treated with 3*N* NaOH. We earlier noted that, at 0°C, the swelling of cotton in the slack state with aqueous NaOH passes through a maximum at this concentration; but only partial lattice conversion to cellulose II is obtained by the treatment.[2] Neither LiOH nor KOH produces a similar swelling maximum.[2]

The bundle tenacity of loose cotton fibers treated, while in the slack state, with 5*N* LiOH, NaOH, or KOH was higher when the treatment was given at 21°C rather than at 0°C (Table I). Previous work[2] had indicated that the extent of swelling of cotton as a result of alkali treatment was less at 21°C than at 0°C, as measured by 2-propanol retention. Thus, there appears to be an inverse relation between tenacity and extent of swelling for cotton fibers treated with a given alkali metal hydroxide at 5*N* concentration. Such a hypothesis is supported by the fact that at either 0°C or 21°C, the tenacity of cotton fibers treated with 5*N* KOH was higher than that of fibers treated with 5*N* LiOH or NaOH (Table I), while for a given temperature the extent of swelling of cotton by 5*N* KOH is less than that caused by 5*N* LiOH or NaOH.[1,2]

The tenacity of cotton treated with 5*N* LiOH or 5*N* NaOH was lower than that of the starting nonmercerized sample (Table I). Depending on the treatment temperature, the tenacity of cotton treated with 5*N* KOH was approximately the same, or slightly higher, than that of the starting cotton. The question arises as to why the tenacities of the LiOH- or NaOH-treated samples were lower than that of the untreated cotton. Warwicker and coworkers[4] have reviewed the effect of slack mercerization with aqueous NaOH solution on the tenacity of cotton at "zero" test length. Their summary of the results indicate a 10% to 40% decrease in tenacity. Slack mercerization using NaOH appears to increase the crystallite orientation of the cotton fiber.[5] Recently, it has been suggested that the increase in orientation, as measured by x-rays, may be a function of deconvolution and not necessarily a change in crystallite orientation within the fibrillar structure of the cotton fiber.[10] Decrystallization has been suggested as a factor that may be involved in the loss in tenacity of the fiber during mercerization.[4]

When relating changes in tensile strength to fine structure of alkali-treated cotton fibers, we suggest it is more appropriate to consider results as breaking load rather than as tenacity, since it is generally accepted that chain rupture is primarily involved in the tensile failure of cotton fibers.[6,7] Thus, if we assume that the number of chain molecules per fiber cross section is not affected by fiber shrinkage, then breaking loads will be affected directly by changes in fine structure which alter the distribution of stress among the chain molecules when a tensile force is applied.

TABLE I
Bundle Tenacity of Cotton Fibers Treated in the Slack State with Alkali Metal Hydroxides

Swelling agent	Swelling agent concn., N	Bundle tenacity[a] for sample treated with swelling agent at: 0°C, g/tex	0°C, S.D.[b]	0°C, RT[c]	21°C, g/tex	21°C, S.D.[b]	21°C, RT[c]
—	—	34.6	0.75	100	34.6	0.75	100
LiOH	5.0	28.2	0.47	82	31.4	0.59	91
NaOH	3.0	26.6	0.41	77	—	—	—
	5.0	28.7	0.57	83	30.6	0.38	88
KOH	5.0	34.2	0.74	99	36.0	0.58	104

[a] Measured without a spacer and thus nominally at zero mm test length.
[b] S.D. = standard deviation.
[c] RT = tenacity of sample as a percentage of the tenacity of the starting cotton fiber.

The fineness of the nonmercerized cotton fibers was 0.187 tex, and that of the fiber treated with 5N alkali at 0°C or 21°C was higher (Table II). Breaking loads can be calculated by multiplying tenacity by tex. Such calculations revealed that the breaking loads of fibers treated with 5N LiOH, NaOH, or KOH at 0°C or 21°C were higher than that of the nonmercerized cotton (Table II). KOH treatment produced fibers that had higher breaking loads than fibers treated with LiOH or NaOH at either 0°C or 21°C.

Previously, we had thought that the increase in breaking load of cotton fibers by NaOH mercerization was due to decrystallization causing a relief of strains in the fiber structure set up during the drying and crystallization of cellulose while the fibers were in the boll.[8] We now believe the increase in strength may also have been due, in part, to an increase in crystallite orientation brought about by the NaOH mercerization and to a reduction in crystallite length. The crystallite length of cotton, as measured by level-off degree of polymerization (LODP), is reduced approximately 37% by mercerization with NaOH at 21°C[1] and 43% at 0°C.[2] If the fiber crystallites are visualized as relatively inflexible rods linked together by the chains of the less ordered regions, then a reduction in length of the rods by mercerization may facilitate movement of the rods when a tensile force is applied to the fiber, and thus permit a relief and redistribution of strain.

TABLE II
Fineness and Breaking Load of Cotton Fiber Treated in the Slack State with Alkali Metal Hydroxides

Swelling agent	Swelling agent concn., N	Results for sample treated with swelling agent at 0°C, tex	0°C, Breaking load, g	21°C, tex	21°C, Breaking load, g
—	—	.187	6.47	.187	6.47
LiOH	5.0	.237	6.68	.224	7.03
NaOH	3.0	.240	6.38	—	—
	5.0	.234	6.72	.226	6.92
KOH	5.0	.214	7.32	.205	7.38

Our attention was drawn to the possible effect of crystallite length on tensile strength when we considered reasons why KOH mercerization was increasing the breaking load of cotton more than was NaOH mercerization. Measurement of fiber crystallite orientations indicated that the increase due to alkali treatment was the same, within experimental error, for both treatments with 5*N* NaOH or 5*N* KOH at 0°C. The 50% x-ray angle for the nonmercerized cotton was 31.1, and for the NaOH-treated and KOH-treated samples, it was 22.7 and 22.3, respectively. Previous work[2] has shown that for cotton treated with 5*N* NaOH or 5*N* KOH at 0°C, the sorption ratios of the products are approximately the same (about 1.55), indicating that both treatments increase the accessibility of the fiber by similar amounts. Therefore, the reduction in crystallinity caused by the two treatments is about the same. On the other hand, the LODP of the KOH-treated sample is about 15% less than that of the NaOH-treated sample, and 51% less than the nonmercerized sample.[2] Thus, of the measured parameters relating to fine structure, the only difference between the NaOH- and KOH-treated samples is the crystallite length.

The same three parameters, namely, lowered crystallinity, increased orientation, and shorter crystallite length, need to be included when considering the increased breaking load of cotton treated with 5*N* LiOH relative to that of the untreated cotton. Although degree of orientation was not measured for LiOH-treated samples, other work indicates that it would be increased.[9] Also, crystallite length and degree of crystallinity are decreased by LiOH treatment.[1,2]

Crystallite length by itself cannot explain completely differences in strength of cotton treated with various alkali metal hydroxides at different temperatures. Although the LODP of cotton treated at 21°C with 5*N* KOH[1] is roughly similar to that of cotton treated at 0°C with 5*N* LiOH or NaOH,[2] the KOH-treated sample has the higher breaking load (Table II). The LiOH or NaOH treatments cause more extensive swelling of cotton than the KOH treatment,[1,2] and this may impair the supramolecular structure of the fiber in a manner leading to a lower breaking load. Degree of orientation may vary to some extent with the treatment temperature also.

It should be noted that treatment with 3*N* NaOH at 0°C produced a fiber that had a lower tenacity (Table I) and lower breaking load (Table II) than either nonmercerized cotton or cotton mercerized with 5*N* NaOH at 0°C. The swelling of cotton passes through a maximum when fibers are treated with 3*N* NaOH at 0°C. However, the supramolecular structure has not been affected in a manner that will cause an increase in breaking load relative to the nonmercerized fiber.

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