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Behavior of Untreated and Crosslinked Cotton Fibers. I. Contribution of Maturity

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Synopsis

The physical and structural properties of American Upland cottons of different maturities have been examined carefully in order to identify fiber parameters which make significant contributions to easy-care properties. The cottons studied possess widely different degrees of overall orientation as determined from birefringence measurements and also exhibit slightly different crease recovery angles. These cottons also respond differently to swelling in sodium hydroxide solution of different concentrations. The physical and mechanical properties of these cottons modified by formaldehyde crosslinking are also compared. The increase in crease recovery angles and the concomitant tensile losses vary from cotton to cotton but are not dependent on maturity or fineness. The stiffness of crosslinked fibers decreases rather than increases with increasing bound formaldehyde. A comparison of the changes in the mechanical properties of formaldehyde-crosslinked cottons differing widely in orientation suggests that cottons with very high orientation are not suitable for chemical modification to impart durable press properties.

INTRODUCTION

Characterization of cotton has long been the objective in an attempt to predict fiber performance during mill processing and the end-use value of the fiber. A mature cotton fiber is a heterogeneous structure with a number of convolutions and structural reversals along its length. In addition, the fibrillar orientation as well as cell wall thickness varies from cotton to cotton.[1,2] The role played by structural features, such as fibrillar orientation, convolution angle, and cell wall thickness, in determining the mechanical properties of native cotton fibers is widely recognized.[3-7] The strength and recovery properties of cotton fibers removed from yarns and fabrics chemically modified to improve their wash-and-wear performance have also been investigated.[8-12] It is believed that the changes in the mechanical properties of yarns and fabrics caused by chemical treatments are due, at least in part, to the changes in the individual fibers themselves.[13] However, because of interactions among fibers in yarn and fabrics, the mechanical properties of individual fibers and the magnitude of each can vary with the type of treatment and also with fabric construction.[14,15] Consequently, direct measurements of fiber properties, such as tenacity, extension at break, and crease recovery, which are of technological importance, are highly desirable.

There is an increasing demand from the modern consumer for easy-care cotton textile products. However, the available chemical finishes, in addition to conferring excellent easy-care properties, produce undesirable strength losses and reduce the wear life of the finished goods significantly. The limited data available provide evidence for the differential response of cottons to crosslinking.[16,17] However, more data are required to identify, at least qualitatively, fiber parameters which make significant contributions to the crease recovery, tensile strength, and extension of crosslinked fibers. In this paper, the influence of fiber maturity and orientation on the properties of untreated, alkali-swollen, and formaldehyde-crosslinked fibers is considered.

EXPERIMENTAL

Materials

American Upland cottons differing widely in their initial properties, such as per cent maturity (P_m) and tenacity, were selected for this study. All the cottons were Soxhlet extracted in a 1:2 mixture of ethyl alcohol and benzene for 18 hr to remove the waxes present.

Crosslinking

The extracted cotton fibers were crosslinked with formaldehyde by the Form-D process[18] for 1, 2.5, 5, and 10 min in a constant-temperature water bath maintained at 35°C. The crosslinked cottons were neutralized in 2% sodium carbonate solution, washed, and air dried. The per cent bound formaldehyde content of crosslinked fibers was determined by the chromotrophic acid method.[19] The crosslinked as well as untreated cotton samples were conditioned at 27°C and 65% R.H. before subsequent testing was carried out.

Swelling

The six American cottons were slack swollen in sodium hydroxide solutions of 10%, 16%, 24%, and 30% w/w concentration at 29°C for 30 min, washed, and air dried.

Test Methods

The crease recovery angles of the untreated as well as crosslinked fibers were measured by the method developed in this laboratory.[20] The load–extension curves of untreated, alkali-swollen, and crosslinked cottons were determined using an Instron tester employing Stelometer jaws with a 3.2-mm nominal test length.[21a] The values of the breaking tenacity, per cent extension at break, initial modulus, work of rupture (area under the stress–strain curve measured with a planimeter), and secant modulus were calculated from these curves. The breaking tenacity values were also determined using a Stelometer at 0 and 3.2 mm nominal test lengths. The per cent maturity (P_m) was determined using the sodium hydroxide method.[21b] The linear density was determined according to the ASTM method.[21c] The fiber length distribution was obtained by the Baer Sorter method.[22]

The refractive indices of the extracted, sodium hydroxide-swollen, and crosslinked cottons were measured by the refractometric method developed in this laboratory.[23] The birefringence data were calculated from the difference between the parallel and perpendicular refractive indices.

The alkali centrifuge values, which are a measure of fiber porosity, were obtained from centrifuge determinations.[24] In these studies, the extracted cotton fibers were treated at 27°C in distilled water, high-boiling-point silicone oil, or in 15% w/w sodium hydroxide solution for 15 min and then centrifuged for 20 min at 1200 rpm. The per cent increase in weight in each case was determined.

Two hundred fibers of each of the extracted cottons were examined microscopically and the number of convolutions counted[6] over a length of 1 cm in the central region of individual fibers. The average convolution angle was calculated for each cotton by the method described by Meredith.[25]

RESULTS AND DISCUSSION

The data on the structural and physical properties of purified and cross-linked cottons are given in Tables I to IV, and some of the relationships are shown in Figures 1 to 7.

It is evident from Table I that the six American cottons selected for this study differ widely in maturity, fineness, and tenacity. The birefringence, which is a measure of the overall orientation of the fibrils, is different for different cottons. With increasing test length, the strength is found to decrease. The strength–uniformity ratio, which is an expression of the uniformity of strength along the fiber length, increases as the maturity of the cotton fibers increases.

The fiber porosity as determined by the centrifuge method is seen to have an inverse relationship with fiber maturity.

The curves in Figure 1 illustrate the range in the stress–strain properties for fibers of different maturities, tested in the form of fiber bundles. The

TABLE I
Properties of Untreated American Upland Cottons of Different Maturities

Cotton	Maturity, %	Fineness, μg/in	Effective length, mm	Mean length mm	ACV	Refractive index		
						$n_{\parallel}$	$n_{\perp}$	Δn
947	30	3.1	24.5	12.2	333	1.585	1.537	0.048
940	46	3.7	25.5	15.8	276	1.582	1.537	0.045
193	48	3.8	30.4	20.8	264	1.588	1.536	0.052
805	52	4.2	26.1	20.6	248	1.587	1.535	0.052
888	77	7.4	28.0	22.1	190	1.596	1.534	0.062
875	94	6.4	26.2	21.4	160	1.596	1.533	0.063

Cotton	Bundle strength, g/tex		Strength uniformity ratio	Extension, %	Work of rupture, g/tex	Initial modulus, g/tex	Secant modulus, g/tex	Crease recovery angle, deg
	0 mm	3.2 mm						
947	30.2	14.1	46	10.1	0.66	67	1.21	87
940	31.9	16.6	51	13.2	1.07	72	1.23	91
193	40.0	20.2	50	11.1	1.19	113	1.86	86
805	37.8	18.3	48	11.1	0.89	78	1.65	92
888	36.8	20.0	52	10.8	1.01	47	1.81	84
875	53.1	32.1	60	10.4	1.19	103	2.75	84

TABLE II
Mechanical Properties of Slack Swollen Fibers

Cotton no.	Maturity, %	Breaking tenacity, g/tex, slack swollen in alkali					Extension at break, %, slack swollen in alkali				
		0.0[a]	10.0	16.0	24.0	30.0	0.0[a]	10.0	16.0	24.0	30.0
875	94	29.2	31.7	29.0	27.7	27.8	10.4	12.3	18.0	21.0	20.9
888	77	19.0	19.6	18.3	18.8	18.8	10.8	13.2	21.7	22.6	22.0
805	52	18.3	20.1	21.2	21.4	21.2	11.0	13.2	21.8	21.2	21.7
193	48	20.6	21.6	21.9	21.6	21.7	11.1	13.3	20.8	21.3	21.5
940	46	16.2	17.6	18.1	17.7	17.8	13.2	13.9	19.0	19.2	20.2
947	30	13.3	15.5	16.5	16.8	16.7	10.1	12.8	17.2	17.9	17.9

[a] NaOH concentration.

TABLE III
Mechanical Properties of Crosslinked Fibers

Cotton no.	Crease recovery angle, deg			Per cent tensile loss, %	
	0.0%[a]	0.2%	0.5%	0.2%[a]	0.5%
875	84	93	110	42	73
888	84	104	115	42	72
805	92	103	113	33	65
193	86	105	119	55	81
940	91	108	121	42	74
947	87	99	111	35	63

[a] Bound formaldehyde level.

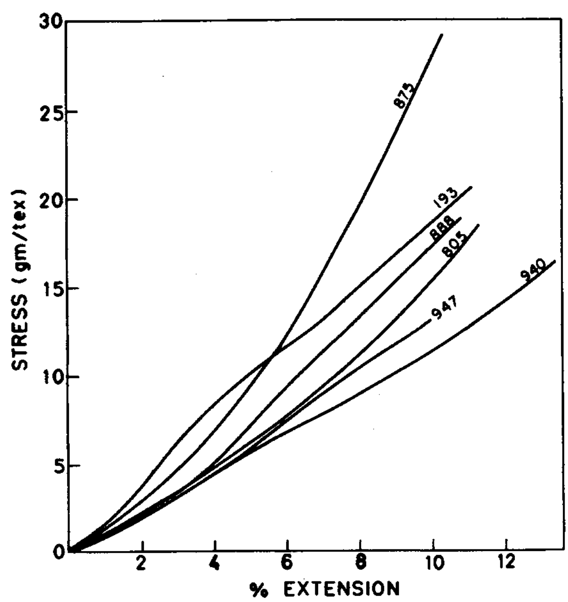

Fig. 1. Stress–strain curves of cottons of different maturities.

TABLE IV
Properties of Crosslinked Fibers

Property	Cotton no. 940				Karnak				Cotton no. 888			Cotton no. 875			Ramie		
	0.0%[a]	0.2%	0.5%	0.8%	0.0%[a]	0.2%	0.5%	0.8%	0.0%[a]	0.2%	0.5%	0.0%[a]	0.2%	0.5%	0.0%[a]	0.2%	0.5%
Crease recovery angle, deg	91	108	122	127	81	110	125	135	84	104	115	84	93	113	64	70	78
ΔCR, deg	0	17	31	36	0	29	44	54	0	20	31	0	9	29	0	6	14
Tenacity, g/tex	16.6	9.1	4.1	2.6	27.0	18.4	9.5	6.6	19.0	8.8	4.6	29.2	16.9	7.9	52.5	20.0	9.5
Tensile loss, %	0	42	74	84	0	32	65	76	0	42	72	0	42	73	0	55	82
Extension, %	13.2	10.6	7.6	5.5	10.0	18.5	7.2	6.6	10.8	7.9	5.4	10.4	7.4	5.2	5.1	3.3	2.2
Loss in extension, %	0	20	42	58	0	16	28	34	0	29	50	0	29	50	0	36	58
Loss in work of rupture, %	0	48	81	92	0	39	80	89	0	54	84	0	48	79	0	66	94
Loss in secant modulus, %	0	32	58	69	0	16	42	56	0	35	55	0	21	45	0	42	67

[a] Bound formaldehyde content.

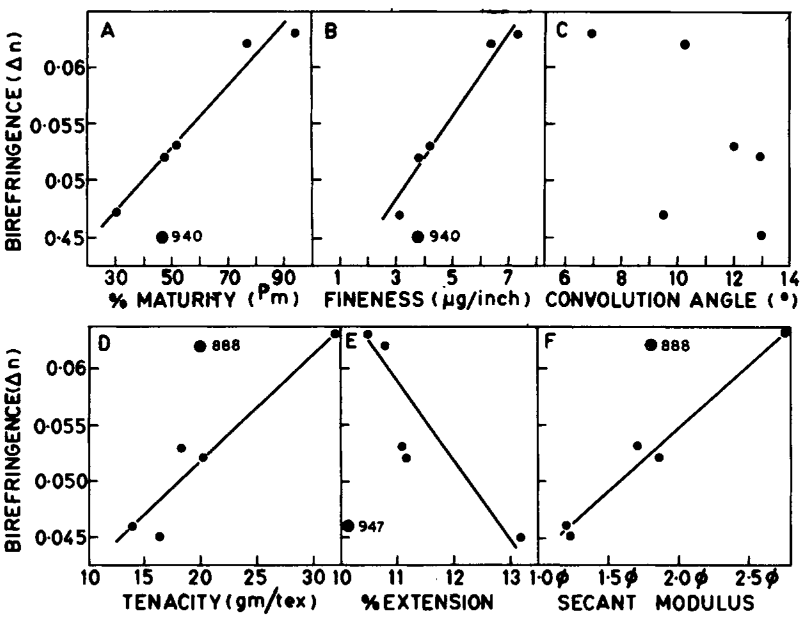

Fig. 2. Birefringence (Δn) as a function of (A) per cent maturity (P_m): (B) fineness; (C) convolution angle; (D) breaking tenacity; (E) extension at break; (F) secant modulus.

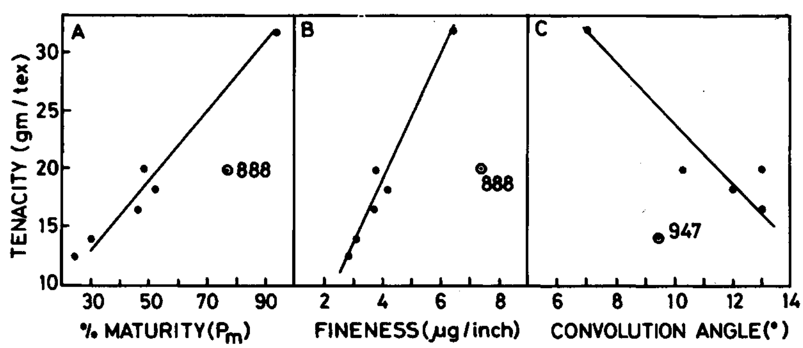

Fig. 3. Dependence of breaking tenacity on (A) per cent maturity (P_m); (B) fineness; (C) convolution angle.

curve for each cotton was selected from considerations of average tenacity as well as extension at break. Other fiber properties, such as initial modulus, work of rupture (which is a measure of the capacity of the fibers to absorb energy), and secant modulus (which denotes the stiffness of the fiber), are different for different fibers.

Relationships Between Birefringence and Fiber Properties

The effect of per cent maturity (P_m) on the birefringence as determined by the refractometer method is shown in Figure 2a. A linear relationship appears to exist, with the exception of cotton no. 940 having a maturity of 46%. Figure 2b shows the plot of the birefringence values against fineness. Here

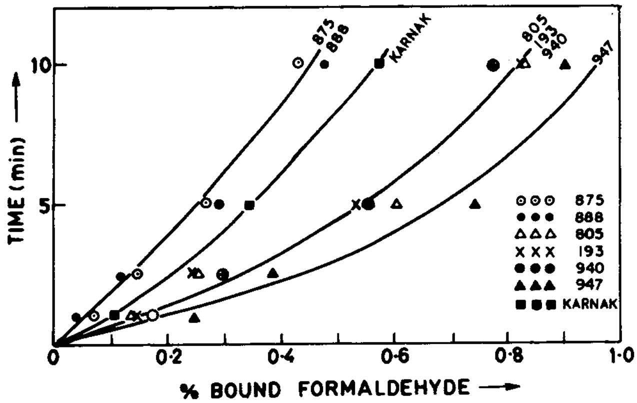

Fig. 4. Per cent bound formaldehyde as a function of time of reaction.

also, a linear relationship is seen to exist. On the other hand, there is considerable scatter in the plot of birefringence versus convolution angle (Fig. 2c).

The birefringence of cotton fibers is correlated with the fiber tenacity in Figure 2d. A linear relationship is again seen to exist, with the exception of cotton no. 888 having a maturity of 77%, which exhibits a high degree of orientation. Earlier workers have also observed a linear relationship between the tenacity values and the orientation as represented by the x-ray angle[3,5,7] or the birefringence.[4,6] The present study has extended these correlations to the case of cottons of different maturities. A similar trend is exhibited in the relationship between the birefringence and the fiber stiffness (Fig. 2f). Figure 2e shows the relationship between the birefringence and the extension at break. It is seen that as the orientation improves, the extensibility of the fiber decreases.

Mechanical Properties

Figure 3a shows the plot of the breaking tenacity against per cent maturity, while the relationship between the tenacity and fineness is shown in Figure 3b. Linear relationships are observed in both cases, with the exception of one cotton, no. 888. A linear equation has been found to fit the data on the tensile properties and the convolution angle shown in Figure 3c. The correlation between the tenacity and the convolution angle is much better than that between birefringence and convolution angle. Thus it is likely that linear relationships exist among the fiber properties so far considered. The high scatter in some cases is mainly due to the limited data available.

Mechanical Properties of Slack Swollen Fibers

The reaction between cotton fibers and sodium hydroxide solution is of technological significance.[26] The amounts of swelling produced and the consequent changes in fiber properties depend on the concentration of alkali. The tensile properties of the six cottons slack swollen in 10%, 16%, 24%, and 30% w/w sodium hydroxide at 29°C have been investigated. The tenacity values at 0 mm test length of the swollen fibers have been found to be lower than the untreated control in all cases, in agreement with earlier results.[26,27] The changes in tenacity at 3.2 mm test length for different cottons are given in Table II. Swelling in 10% alkali results in an increase in tenacity for low and intermediate maturity cottons. This increase in tenacity can be attributed mainly to the deconvolution that takes place when the fibers are swollen in 10% alkali, since a decrease in convolution angle should result in an increase in tenacity. Hebert et al.[28] have studied the dependence of the extent of deconvolution on the alkali concentration, and their results show that a significant decrease in the convolution angle takes place in 10% sodium hydroxide.

The swelling treatment in 16%, 24%, and 30% sodium hydroxide solution produces changes in breaking tenacity of different magnitude and also in either direction. These changes appear to be characteristic of each cotton. The highly mature cotton no. 875 shows the maximum decrease in tenacity, while the low-maturity cotton no. 947 shows the maximum gain in tenacity. Further, the strength–uniformity ratio has been found to improve on mercerization in all instances, mainly because of the removal of weak places. These wide differences in the effects of mercerization on tenacity have been earlier explained[27] as due to the predominance of the factors which produce increases in tenacity (such as removal of strains, deconvolution, and increase in crystallite orientation) over those which produce decreases in tenacity (such as increase in linear density, disorientation of the amorphous phase, and decrystallization), or vice versa.

The extension at break of cottons of different maturities progressively increases for fibers swollen in NaOH solution of increasing concentration. The process of mercerization tends to equalize the extension at break of different cottons. The extension of fibers having an initial low elongation increases much more than those which have a high extension to start with. The maximum increase in extension is about 90% of the initial value.

The secant modulus, which measures the stiffness of fibers, progressively decreases for fibers treated in alkali of increasing concentration. Cotton no. 875 shows the maximum decrease, while cotton no. 947 shows the minimum decrease in secant modulus on mercerization.

Properties of Crosslinked Fibers

Figure 4 shows the plots of bound formaldehyde against the time of reaction for the six American cottons. The data for Karnak, a long-staple, fine cotton, are also included for comparison. The relationship is observed to be nonlinear in all cases. The bound formaldehyde in any given time is found to depend on the maturity of cotton fibers and decreases with increasing maturity. This is understandable because the accessibility decreases with increasing maturity.

From the results given in Table III, it is seen that different cottons exhibit different crease recovery angles at comparable formaldehyde levels. Figure 5 shows the relationship between the improvement in crease recovery angle (ΔCR) and the per cent bound formaldehyde. The improvements in crease recovery angles are also slightly different at corresponding levels of bound formaldehyde. These results substantiate the observations of Grant et al.[13]

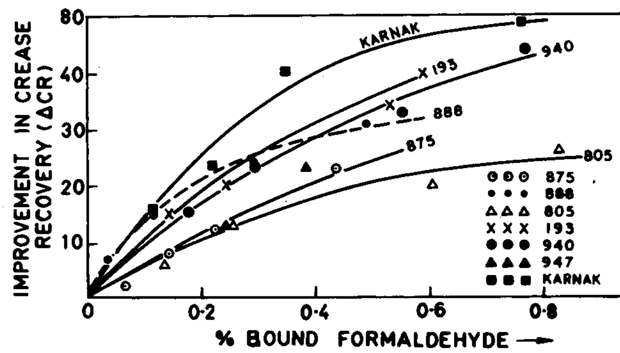

Fig. 5. Improvement in dry crease recovery angle (ΔCR) as a function of per cent bound formaldehyde.

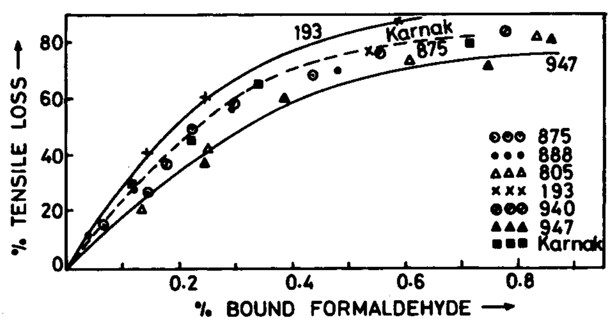

Fig. 6. Relationship between per cent bound formaldehyde and per cent tensile loss.

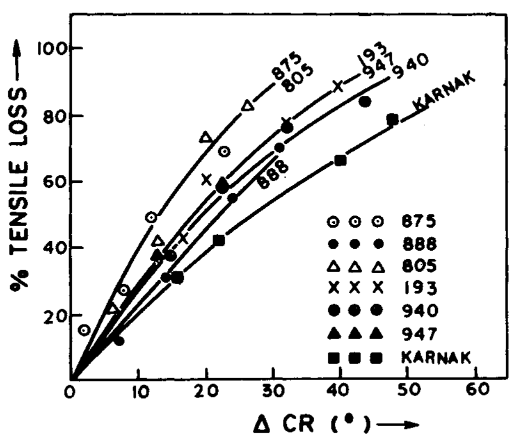

Fig. 7. Plots of tensile loss vs. improvement in crease recovery angle (ΔCR).

who have shown that resin-finished fabrics woven from cottons of different fiber properties exhibit different dry crease recovery angles. It is further seen that the crease recovery angle or the improvement in crease recovery angle (ΔCR) does not depend on the degree of wall thickening or the fineness. Among the American Upland cottons, no. 940 exhibits the highest improvements in crease recovery, almost comparable to that of Karnak. The maximum difference in crease recovery at any bound formaldehyde level is found to be about 25°.

The per cent tensile losses are plotted against the bound formaldehyde level in Figure 6. Here, differences in tensile losses for different crosslinked cottons are seen. Again, no systematic dependence of tensile loss on per cent maturity or fineness is observed (Table III). Among the cottons with more or less the same value of maturity, no. 193 shows a higher tensile loss, while no. 805 shows a lower tensile loss than no. 940 at any bound formaldehyde level.

The per cent tensile losses for the same cottons with a wide range in their initial properties are correlated with the improvements in conditioned crease recovery angle (ΔCR) in Figure 7. Distinct curves appear to exist for various cotton samples. Within a particular species, it appears that cottons with very high maturity show higher losses in tensile strength than cottons with lower maturity for the same improvement in crease recovery.

The improvement in crease recovery angle and concomitant tensile loss arise on account of crosslink embrittlement. From the technologic point of view, the actual crease recovery level and the retained strength and extension after easy-care finishing are equally important. The changes in the mechanical properties of crosslinked cottons and ramie differing widely in orientation are given in Table IV, in the order of increasing orientation.

It is seen from these data that the improvement in crease recovery angle, as well as the per cent loss in tenacity, increases curvilinearly with increasing bound formaldehyde. The rate of increase is, however, different for various fibers. Ramie, which is the most oriented fiber, exhibits a minimum improvement in crease recovery angle and a maximum loss in tenacity, while no. 940, with the minimum orientation, shows a much greater improvement in crease recovery angle and a lower loss in tenacity. Karnak, a typical long-staple, fine cotton with moderate fibrillar orientation, shows the maximum improvement in crease recovery and the minimum tensile loss at any level of bound formaldehyde.

A significant decrease in the fiber extension (as compared to the untreated value) is shown by all the cottons and ramie after crosslinking. The rate of decrease in extension is different for different fibers. Ramie, the stiffest of all fibers, exhibits the highest loss in extension, followed by no. 875 and 940. It is interesting to note that Karnak, belonging to the G. barbadense variety, retains a considerably higher extension than other cottons after crosslinking. This comparatively high extension for the crosslinked cotton is most desirable from the point of view of fabric tear strength and abrasion resistance.[29]

The work of rupture, which is related to the energy absorbing capacity of the fiber, has been correlated with the durability of a fabric.[30] The per cent loss in the work of rupture after crosslinking increases curvilinearly as a function of the bound formaldehyde. The reduction in the work of rupture is maximum for ramie. However, the per cent losses are not significantly different for different American cottons at comparable levels of bound formaldehyde.

The secant modulus decreases for all the crosslinked fibers. The initial differences in the secant moduli of different cottons are maintained at different levels of bound formaldehyde. In the case of ramie, the decrease in the secant modulus is much faster. Thus, the crosslinked fibers show a decrease rather than an increase in stiffness over the untreated controls. Similar results have been obtained by Rebenfeld[31] and Grant et al.[12] for fibers extracted from yarns treated with resins and formaldehyde. In contrast, resin-finished and crosslinked fabrics exhibit an increase in stiffness over the untreated control.[32,33] However, the tensile and strain recovery moduli increase sharply with increasing bound resin or formaldehyde.[8,10,12] The initial elastic moduli do not show any systematic trend. Some cottons show a decrease in elastic modulus, while for others there is no significant change after crosslinking. The differences in the elastic moduli of different cottons are significantly reduced by crosslinking.

A comparison of the properties of crosslinked cottons given in Table IV shows that, purely from the point of view of flexibility and crease recovery, cotton no. 940 appears to be promising. However, the retained strength is too low to be acceptable. On the other hand, the highly oriented cotton no. 875 has a higher retained strength, but the actual crease recovery and retained extension at break are comparatively low. The fiber with the highest orientation, namely, ramie, is unsuitable for chemical modifications since most of its mechanical properties deteriorate rather rapidly. In addition to this, the resin or formaldehyde fixation is low for ramie. Applying the same considerations, Karnak appears to be the most suitable cotton.

The results of this study suggest that (i) fiber maturity does not have much influence on the response of cottons to crosslinking, except that the amount of formaldehyde which is bound is higher for low-maturity cottons than for high-maturity cottons in a given interval of time; and (ii) fiber orientation does not seem to have a significant effect, unless the fibers are very highly oriented. Further studies are required to determine the influence of other parameters such as length, fineness, strength, and extension, in order to prepare a cotton product suitable for the future market, increasingly dominated by chemically treated products.

CONCLUSIONS

The influence of fiber maturity and orientation of cotton fibers on their response to swelling in alkali as well as formaldehyde crosslinking to impart easy-care properties and on the consequent changes in the mechanical properties have been considered.

The birefringence, which is a measure of fibrillar orientation, has been correlated with fiber maturity, tenacity, extension at break, convolution angle, and secant modulus. The tenacity values have also been found to be related to fiber maturity, fineness, and convolution angle.

Various cottons respond differently to swelling in sodium hydroxide solution of different concentrations.

Crosslinking produces widely different effects on the fiber properties of cottons of different maturities. However, no systematic dependence on maturity or fineness has been observed.

The changes in tenacity, extension, work of rupture, secant modulus, and initial modulus do not appear to have any significant dependence on maturity or orientation. Cottons with very high orientation are not very suitable for chemical modifications which impart durable press characteristics.

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