# Behavior of Untreated and Crosslinked Cotton Fibers. II. Contribution of Intrinsic Fiber Properties

G. M. VENKATESH and N. E. DWELTZ, Ahmedabad Textile Industry's Research Association (ATIRA), Ahmedabad 380015, India

#### **Synopsis**

The mechanical properties of extracted and formaldehyde-crosslinked cotton fibers are presented. The crease recovery angles of different cotton fibers are more or less the same. As the per cent bound formaldehyde increases, the crease recovery angle of the treated fibers increases while the tensile strength decreases. Crease recovery and tensile loss factors appear to be sensitive indices of the improvement in crease recovery angles and the concomitant losses in tenacity of the fibers modified by any crosslinking process. The crease recovery angles for any cotton modified by formaldehyde crosslinking depend on the pretreatment it has received.

#### **INTRODUCTION**

The influence of maturity and fineness on the improvement in the crease recovery and the concomitant tensile losses as a result of formaldehyde crosslinking of fibers has been described earlier.<sup>1</sup> Other fiber properties such as tenacity and extension at break of the untreated fibers are also known to influence the improvement in crease recovery and the resultant loss in tenacity after crosslinking with formaldehyde. The role played by these properties will now be discussed.

### **EXPERIMENTAL**

#### Materials

Cottons differing widely in fiber properties such as tenacity, and extension at break but having more or less the same fineness and maturity (with the exception of one cotton, ISC-67) were selected for this study. These cottons were Soxhlet extracted using a 2:1 mixture of benzene and ethyl alcohol for 18 hr, and Karnak and Giza-45 were also kier boiled using a 2% NaOH solution for 6 hr.

#### Crosslinking

The extracted cotton fibers were crosslinked by the formaldehyde Form-D process<sup>2</sup> for 1, 2.5, 5, 15, and 60 min in a constant-temperature water bath maintained at 35°C. The crosslinked fibers were neutralized with a 2% sodium carbonate solution, washed in water, and air dried.

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## **Test Methods**

The mechanical properties of the fibers conditioned at  $27^{\circ}$ C and 65% R.H. for 24 hr were studied as follows: The crease recovery angles of the control as well as crosslinked cotton fibers were measured in the form of highly parallelized flat bundles by the method developed by Venkatesh et al.<sup>3</sup>

The breaking tenacity was determined using a Stelometer at 0 and 3.2 mm test lengths (ASTM D1445-67). The strength uniformity ratio was calculated as the ratio of the strength at 3.2 mm test length to the strength at 0 mm test length.

The tensile loss factor is defined as the ratio of the per cent tensile loss to the per cent bound formaldehyde and was determined by drawing a tangent at the origin to the curve of per cent loss in tensile strength versus per cent bound formaldehyde. Similarly, the crease recovery factor is defined as the ratio of the per cent improvement in the dry crease recovery angle to the per cent bound formaldehyde and was determined by drawing a tangent at the origin to the curve of per cent improvement in dry crease recovery versus the per cent bound formal-dehyde.

The per cent maturity  $(P_m)$  values were obtained by the sodium hydroxide swelling method (ASTM D1442-64T). The linear density was determined by the ASTM method D1769-60.

The effective and mean fiber lengths were determined by the Baer Sorter method.<sup>4</sup> The alkali centrifuge values were obtained by the method developed by Marsh et al.<sup>5</sup> and Honold and Grant.<sup>6</sup>

#### **RESULTS AND DISCUSSION**

The various properties of Soxhlet-extracted and crosslinked cottons are given in Tables I and II, and some of the interrelationships are shown in Figures 1-5.

From Table I it is seen that the cottons differ widely in tenacity, strength uniformity, and length. However, the initial crease recovery angles are not very different for all the cottons. The fiber porosity as determined from the alkali centrifuge measurements are different for different cottons.

Figure 1 shows the per cent bound formaldehyde plotted against the time of reaction. It is seen that the amount of bound formaldehyde in any time interval is different for different cottons. It has been shown that when the cottons of the same species but of different maturities are considered, the amount of bound



Fig. 1. Per cent bound formaldehyde as a function of time of reaction.

TABLE I Properties of Soxhlet-Extracted Cottons

	Crease recovery angle, degrees		85	80	85	85	88	85	86	82
	Strength uniformity ratio		0.70	0.63	0.66	0.63	0.48	0.56	0.61	0.60
	strength, tex	3.2 mm	36.0	27.0	31.2	29.1	20.9	23.3	30.4	22.4
	Bundle g/	0 mm	51.6	43.0	47.3	46.0	42.2	41.8	49.9	37.3
	Alkali centrifuge value		199	180	198	187	182	212	203	244
	Fineness, mg/in.		3.0	3.0	3.4	3.6	3.5	3.8	3.6	3.3
	Maturity, P., %		77	84	77	84	81	68	77	59
	Mean length, mm		27.0	27.0	28.0	27.0	23.4	23.2	24.5	16.8
	Effective	Effective length, mm		34.2	37.5	38.5	31.8	32.5	33.7	30.5
		Cotton	Giza-45	Karnak	Sudan	Sudan	HopiAcala	Shankar-4	Sujata	<b>ISC-67</b>

		ss factor	<b>3.2</b> mm	300	200	170	165	170	165	165	200
		Tensile lo	0 mm	170	165	135	125	135	100	110	120
	Tensile losses, $\%$ , at formaldehyde levels	1.0	3.2 mm	82	81	82	83	80	62	80	84
			0 mm	11	68	72	67	68	60	69	99
		0.5	3.2 mm	74	63	61	64	59	60	59	67
			0 mm	57	50	50	48	49	44	51	45
		0.2	3.2 mm	46	37	34	31	32	32	32	38
			0 mm	30	28	25	53	26	19	23	22
	Crease recovery factor			171	153	135	130	135	71	60	100
	ase recovery es, degree, at aldehyde levels		1.0	140	135	134	137	133	126	129	129
			0.5	130	123	122	123	120	109	112	115
	Cre	form	0.2	112	103	105	101	102	95	67	96
			Cotton	Giza-45	Karnak	Sudan	Sudan	HopiAcala	Shankar-4	Sujata	ISC-67

TABLE II Mechanical Properties of Crosslinked Fibers

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Fig. 2. Plots of improvement in crease recovery angle  $(\Delta CR)$  as a function of per cent bound formaldehyde.

formaldehyde is strongly dependent on maturity.<sup>1</sup> It appears that the amount of bound formaldehyde is a characteristic of each cotton when cottons of similar maturities are considered. Figure 2 shows the relationship between the improvement in crease recovery angle ( $\Delta CR$ ) and the per cent bound formaldehyde. The improvements in crease recovery angles at any level of bound formaldehyde are different for various cottons. Giza-45 shows maximum improvement in crease recovery. Grant et al.<sup>7</sup> have studied the crease recovery properties of fabrics woven with cottons of different fiber properties and have observed improvements in crease recovery to different extents after resin finishing to about the same level.

The crease recovery factors are different for different cottons. It is seen that the higher the improvement in crease recovery, the higher will be the crease recovery factor. The crease recovery factor is maximum for Giza-45 and minimum for Sujata and Shankar-4.

Figure 3 shows the per cent tensile losses of the crosslinked fibers plotted against the per cent bound formaldehyde. It is observed that different cottons lose tensile strength to different extents. In the case of tensile loss also, the maximum difference is about 20% at any bound formaldehyde level for the different cottons studied. Giza-45, which shows the maximum improvement in crease recovery, is also the cotton showing the highest loss in tensile strength. Thus, it appears that the improvement in crease recovery and the concomitant tensile loss arise because of the same mechanism just as in the case of fabrics.<sup>8,9</sup> The only difference is that the tensile losses suffered by the fabric are comparatively less than the losses in tensile strength of the fibers crosslinked to have more or less similar levels of crease recovery.<sup>9,10</sup> Other studies in which the tensile losses in crosslinked fibers, yarns, and fabrics have been investigated have shown the same results.<sup>11-13</sup>

It is seen from Table II that, as the crease recovery factor increases, the tensile loss factor also increases. These factors thus appear to be sensitive indices which characterize crosslinked fibers.

Figure 4 shows the relationship between the per cent tensile loss and the corresponding improvement in crease recovery angle ( $\Delta CR$ ). The data for different



Fig. 3. Per cent tensile loss vs. per cent bound formaldehyde.



Fig. 4. Plots of improvement in crease recovery angle  $(\Delta CR)$  as a function of bound formaldehyde.

cottons appear to be within definite extreme limits, with a great deal of scatter observed between these two limits. The scatter for any individual cotton is much less, and distinct curves appear to exist. It is seen from Figure 4 that Giza-45 appears to be the best cotton from considerations of both the improvement in crease recovery and the retained tensile strength. From these studies the cottons can be graded as follows: Giza-45, Karnak, Sudan, Sujata, Shankar, and ISC-67, the latter showing the poorest performance. The final crease recovery angle and the strength retained after chemical modifications are important considerations in easy-care and durable-press finishing of cotton fabrics. From the results so far obtained, it is apparent that a stronger, finer cotton that is not highly oriented should be selected for weaving fabrics which are to be chemically processed to impart easy-care performance. However, the yarn and fabric structure may modify the physical and mechanical properties of the finished fabrics to some extent. It has been shown that the tensile losses as a result of resin finishing or crosslinking increase in the order fiber < yarn < fabric at corresponding levels of bound resin or formaldehyde. The general trend that has been observed in the case of fibers in this study can be expected to be observed in the case of fabric also. The observations of Grant et al. support this conclusion.

## **Effect of Pretreatment**

The crease recovery angles of Giza-45 fibers have been found to increase in the order raw (undewaxed) < extracted < kier boiled, suggesting that crosslinking extracted and kier-boiled cotton fibers might produce improvements in crease recovery to different extents at corresponding bound formaldehyde levels. Kier-boiled Karnak and Giza-45 fibers were crosslinked by the Form-D process. Figure 5a shows the crease recovery angles of the Soxhlet-extracted as well as kier-boiled Karnak and Giza-45 fibers plotted against the per cent bound formaldehyde. It is seen that the crease recovery angles of the kier-boiled fibers are significantly higher than those of the Soxhlet-extracted fibers at corresponding



Fig. 5. Relationship between per cent bound formaldehyde and (a) crease recovery angle and (b) per cent tensile loss.

bound formaldehyde levels. Figure 5b shows the plots of per cent tensile losses against the per cent bound formaldehyde levels. It is seen that the kier-boiled samples exhibit somewhat higher tensile losses at corresponding formaldehyde levels than the extracted samples. From these interrelationships, it is clear that the kier-boiled fibers exhibit higher crease recovery angles accompanied by marginally higher strength losses than the extracted samples at corresponding bound formaldehyde levels. Even the improvements in the crease recovery angles of kier-boiled fibers are comparatively higher than those in the case of extracted fibers after crosslinking. Thus, it appears that partial removal of wax accompanied by almost complete removal of noncellulosic matter is more desirable than partial removal of wax alone in order to impart better crease recovery properties to finished fibers or fabrics.

#### CONCLUSIONS

Cotton fibers differing widely in fiber properties have been found to exhibit different amounts of improvements in crease recovery when modified by formaldehyde crosslinking. The concomitant tensile losses are also different. The crease recovery angles exhibited by a given cotton depend on the pretreatment it has received.

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