

Effect of Resin Chemisorption and Crosslinking on Various Fiber Properties of Cotton

S. B. PATEL, A. K. KULSHRESHTHA, and P. C. MEHTA, *Ahmedabad
Textile Industry's Research Association (ATIRA), Ahmedabad-380
015, Gujarat, India*

Synopsis

Chemisorption and crosslinking of cotton cellulose has been carried out with DMEU, DMPU, DHEU, and DMDHEU. Various physicochemical properties of resin-treated samples have been studied and the data subjected to a linear regression analysis. Using the techniques of liquid retention and optical microscopy it has been found that the chemisorbed cotton is characterized by a lower level of bound resin, greater amount of methylol HCHO, and higher swellability of structure in comparison to the crosslinked cotton. This difference of behavior between the two cottons is attributed to greater rigidification and a collapse of porous structure in crosslinked cotton as a result of catalytic activity at the curing temperature. For various resin-treated samples there exists a linear relationship between the strength and recovery characteristics of single fibers and those of fiber bundles. The losses in fiber strength and extensibility are found to be proportional to the level of bound resin in various samples. Crosslinked fibers show appreciably higher magnitudes of elastic recovery and bundle crease recovery than chemisorbed fibers. The significance of these results is discussed.

INTRODUCTION

When the sorption of resins on cellulose is carried out under acidic conditions, the adsorbed resin molecules are so tightly held by cellulose that even a severe wash with water cannot remove them from the substrate. Such an adsorption is often referred to¹ as "chemisorption" in order to distinguish it from pure "physical adsorption" wherein the resin molecules are held to cellulose by the very weak van der Waal's forces.² Chemisorption is an exothermic reaction, which precedes the catalytic crosslinking of cotton cellulose with resins by the usual methods of textile finishing.

In most of the published works³⁻⁶ on the effect of crosslinking on mechanical properties of cotton, fibers are extracted from yarns or fabrics which have been subjected to resin treatment. The mechanical properties of these fibers are evaluated and correlated with those of the corresponding resin treated yarns or fabrics.

The scope of the present work is different from earlier studies on the effects of crosslinking on mechanical properties of cotton. Chemisorption and crosslinking reactions of cotton cellulose with certain resins have been carried out on fibers. The resins employed are dimethylolethyleneurea

(DMEU), dimethylolpropyleneurea (DMPU), dihydroxyethyleneurea (DHEU), and dimethyldihydroxyethyleneurea (DMDHEU). The chemically treated fibers have been analyzed for various physicochemical properties such as water retention value (WRV), alkali centrifuge value (ACV), per cent nitrogen, per cent bound formaldehyde (HCHO), wall thickness of water-swollen cross sections, etc., and subjected to infrared spectral examination. The mechanical properties of various treated samples and those of the untreated cotton (control) are evaluated on single fibers as well as on fiber bundles. In this context, a method⁷ recently developed in ATIRA for determining the crease recovery angles of fiber bundles has been found to be very useful.

The present paper highlights the changes in fiber properties of cotton which result upon chemisorption and crosslinking with resins having different reactivity and structure. Particular emphasis is laid on properties which pertain to improvement in resiliency and loss in strength of cotton upon the application of resins. Correlations have been worked out between (a) physicochemical properties and mechanical properties of fibers and (b) mechanical properties of single fibers and those of fiber bundles, for various samples. The differences in the physical structure of chemisorbed and crosslinked cotton samples have been studied by means of infrared spectroscopy and optical microscopy of fiber cell wall swollen in water. These observed structural differences between two types of resin treated cotton correlate well with the measurements of WRV and ACV. It is hoped that the results obtained in the present work are of fundamental importance to resin finishing treatments on cotton textiles.

EXPERIMENTAL

Preparation of Resin-Treated Samples

The work reported in this paper was carried out on Gujarat-67 cotton. The details about the effect of pH, temperature, and catalyst concentration on the equilibrium and kinetics of chemisorption of DMEU, DMPU, DHEU, and DMDHEU on this cotton will be published elsewhere.^{8,9} Cotton slivers were cut into short lengths and Soxhlet extracted first with chloroform and then with ethanol, each for a period of 18 hr. Extracted sliver pieces were then boiled in an atmosphere of nitrogen with 1% NaOH solution, washed in excess water, neutralized by dipping in 1% acetic acid, subsequently washed in distilled water and air dried in a dust-free room. The dried, cut slivers were opened in a Shirley analyzer and opened fibers were stored in an atmosphere conditioned at 65% R.H. and 27°C.

The method of preparation of chemisorbed and crosslinked samples is briefly stated as follows: The chemisorption of various resins on cotton tufts was carried out at pH 2.5, in the presence of magnesium chloride ($MgCl_2$). The experimental solutions were therefore prepared which consisted of varying amounts of resin, 1M $MgCl_2 \cdot 6H_2O$, and hydrochloric acid just sufficient to adjust the pH to 2.5. About 5 g conditioned cotton was reacted at 50°C for 48 hr with 500 ml of the experimental solution contained

in a flask. The flask was immersed in a water bath maintained at the required temperature ($\pm 0.1^\circ\text{C}$) and kept continuously shaken during the period of reaction. After completion of the reaction, fibers were removed from the flask and divided into two portions. One portion of fibers was immediately washed with water until the complete removal of unreacted resin, as checked by the absence of formaldehyde in the wash liquor by the chromotropic acid method. The other portion was dried at 110°C for 8 min and cured at 160°C for 3 min, after adjusting the wet pickup to about 100% on the weight of fibers by centrifuging. The cured samples were thoroughly washed first with a soap-soda solution and subsequently with distilled water. Thus, the method of preparation of chemisorbed and cross-linked samples differs only in that, while the former were directly washed with water after completion of reaction with experimental solution containing resin, the latter were subjected to drying and curing treatments prior to wash.

Selection of Samples for Various Analyses

In order to assess the effect of different resins on fiber properties, it is necessary to compare treated samples with roughly the same molar concentration of bound resin per unit weight of dry fibers. Therefore, for each resin (DMEU, DMPU, DHEU, and DMDHEU), two samples, one chemisorbed and the other its crosslinked counterpart, are selected. The selection of cotton samples treated with different resins for various studies is made in such a way that all chemisorbed products have nearly equal levels of bound resin.

Table I gives the chemical analysis of samples selected for various analyses reported in this paper. For any given resin, the chemisorbed and cross-linked samples are prepared from cotton using identical method of resin application. As the data given in Table I show, crosslinked samples show much higher bound resin on fibers than the corresponding chemisorbed samples. This higher fixation of resin in the former is due to the occurrence of the catalytic activity of MgCl_2 at relatively higher temperature of cure, leading to the formation of crosslinks.

TABLE I
Chemical Analysis of Resin-Treated Samples

Sample	Designation	Bound nitrogen per unit weight of dry fibers, g/g	Resin bound on fibers, moles/kg
DMEU-Chemisorbed Cotton	A	25.6×10^{-4}	71.5×10^{-3}
DMEU-Crosslinked Cotton	B	75.2×10^{-4}	246.0×10^{-3}
DMPU-Chemisorbed Cotton	C	20.7×10^{-4}	74.0×10^{-3}
DMPU-Crosslinked Cotton	D	59.3×10^{-4}	212.0×10^{-3}
DHEU-Chemisorbed Cotton	E	21.8×10^{-4}	71.2×10^{-3}
DHEU-Crosslinked Cotton	F	33.6×10^{-4}	126.7×10^{-3}
DMDHEU-Chemisorbed Cotton	G	17.8×10^{-4}	63.6×10^{-3}
DMDHEU-Crosslinked Cotton	H	66.3×10^{-4}	236.6×10^{-3}

Test Methods

Mechanical Properties. Various mechanical properties of resin-treated cotton were measured according to ASTM procedures,¹⁰ after preconditioning them at 65% R.H. and 27°C. Various mechanical properties of single fibers were obtained^{10a} from a mean of 100 tests which were performed on the Instron tensile tester using a gauge length of 1 cm. Elastic recovery was determined^{10a} at 2% extension of single fibers. Bundle strength was obtained^{10b} at 0 gauge length from a mean of tests on ten fiber bundles, employing the Stelometer. Bundle crease recovery angle was measured according to the method⁷ developed recently in ATIRA, a brief description of which is as follows:

A wide, flat bundle of fibers with a high degree of fiber parallelization was made and sandwiched between two narrow strips of cellophane tapes. Protruding fibers and extra width of the pad were cut off with a razor blade after placing an aluminium plate (2 cm × 1.5 cm) over the fiber pad. One end of the fiber pad was placed between the thick bottom plate and the thin upper metal leaf of the sample holder of the Monsanto crease recovery tester. The free end of the pad was then folded over the top of the metal leaf of thickness 0.16 mm at 5 mm from the fixed end, so that the creased edge was 15 mm long. The pad was creased under a load of 550 g for 3 mins. The bundle crease recovery angle was then measured as is usually done in a Monsanto wrinkle recovery test.^{10c} Ten fiber pads were prepared and tested in this way for each sample of resin-treated cotton.

Chemical Analysis. Bound nitrogen in the resin-treated samples was estimated by Kjeldahl's method.¹¹ Total bound formaldehyde content of treated samples was determined using the chromotropic acid method.¹² Amount of formaldehyde due to >N·CH₂OH groups in chemisorbed and crosslinked samples was obtained using the method of Jong.¹³

Infrared Spectroscopy. Infrared spectra of various resin-treated samples were obtained on a Perken-Elmer Infracord 137 using potassium bromide pellets of cut fibers. Using the absorbances of bands at wavelengths of 3μ, 3.4 μ, and 6.1 μ, the following band ratio was calculated:

IR band ratio

$$= \frac{(\text{absorbance of C=O stretching}) \times (\text{absorbance of CH}_2 \text{ stretching})}{(\text{absorbance of OH stretching})}$$

Liquid Retention Measurements. WRV of various samples were obtained according to the ASTM procedure.¹⁴ For determining ACV, Honold's procedure¹⁵ was followed.

Optical Microscopy of Swollen Cross-Sections. The following technique, which has been developed recently in ATIRA to observe the cross sections of never-dried cotton, is applied here to study the swellability of resin-treated cotton. Resin-treated samples were subjected to swelling in distilled water. Embedding of swollen fibers in a resin was not found necessary because in the wet state fibers adhere to each other without any difficulty. Thus, fiber cross sections were obtained by slicing a bundle of

wet fibers with a hand microtome. These cross sections were mounted on a slide (water being the only mounting medium) and examined under a projectina at a magnification of 1000 \times . A tracing of fiber cross sections was prepared from which 100 cross sections were used for a measurement of the wall thickness of water-swollen cross sections.^{10d} In crosslinked cotton, resin molecules form interlamellar crosslinks, which reduces the tendency of the cotton cell wall to swell in water thereby resulting in a value of wall thickness lower than that of the control or chemisorbed cotton where there is no restraint to the swelling action of water on the cell wall.

RESULTS

Effect of Chemisorption and Crosslinking with Different Resins on Structure and Physicochemical Properties of Cotton

Table II lists the results of various physicochemical analysis on resin-treated samples. Resin molecules are in a chemisorbed state on cotton in samples A, C, E, and G, whereas crosslinking of resin with cellulose molecules is the predominant feature in samples B, D, F, and H. The latter samples are characterized by a much higher level of bound nitrogen compared with the former. However, in the former samples only one functional group of resin takes part in bond formation with cellulose, whereas in the latter both the functional groups are involved in bonding, leading to the formation of a crosslink. This view is substantiated by results on the estimation of free methylol groups for various resin-treated samples (Table II). The percentage of methylolformaldehyde is much higher in chemisorbed samples than in crosslinked samples.

The determinations of WRV and ACV for various resin-treated samples (Table II) indicate that the structural porosity of the cell wall is much higher in the chemisorbed samples than in crosslinked ones, as seen by higher magnitudes of both WRV and ACV for the former. These differences in wall porosity may be explained in terms of the nature and extent of reaction of resins with cotton cellulose. Thus, chemisorption of cotton with resins causes a reduction in accessibility of cell wall which becomes enhanced upon drying and curing as in crosslinked samples. For all resin-treated samples, ACV decreases linearly with increasing level of bound nitrogen (Fig. 1). Figure 2 shows water-swollen cross sections of cotton chemisorbed and crosslinked with DMPU. The swellability of chemisorbed cotton is higher relative to crosslinked cotton as seen from a measurement of wall thicknesses of cross sections presented in Figure 2 (Table II).

The infrared spectra of untreated and DMPU treated cotton are shown in Figure 3. The analysis shows that chemisorbed samples have a lower band ratio than corresponding crosslinked samples. In fact, a plot of IR band ratio against bound nitrogen for various samples is fairly linear (Fig. 4). This demonstrates that with increase in the extent of bonding of resins with cotton cellulose, there is an increase in absorption of band at wavelength 6.1 μ corresponding to the stretching of C=O bonds.

TABLE II
Various Physicochemical Properties of Cotton Treated with Different Resins

Sample designation	Bound nitrogen per unit weight of dry fibers, g/g	Total bound formaldehyde per unit weight of dry fibers, g/g	Formaldehyde due to >N. CH ₂ OH groups, % based on total bound HCHO	Wall thickness of water-swollen cross sections, μ	IR band ratio	Water retention value (WRV), %	Alkali centrifuge Value (ACV), g/100 g
Control	0	0	0	6.45	9.1	11.7	254
A (DMEU)	25.6×10^{-4}	37.1×10^{-4}	33	—	9.2	10.0	215
B (DMEU)	75.2×10^{-4}	112.3×10^{-4}	10	—	18.2	6.7	150
C (DMPU)	20.7×10^{-4}	34.3×10^{-4}	36	6.47	9.1	10.9	218
D (DMPU)	59.3×10^{-4}	91.2×10^{-4}	13	6.02	15.5	8.1	172
E (DHEU)	21.8×10^{-4}	0	—	—	14.6	7.4	216
F (DHEU)	33.6×10^{-4}	0	—	—	13.3	8.0	214
G (DMDHEU)	17.8×10^{-4}	33.8×10^{-4}	37	—	8.8	8.6	220
H (DMDHEU)	66.3×10^{-4}	93.1×10^{-4}	15	—	16.3	7.5	153

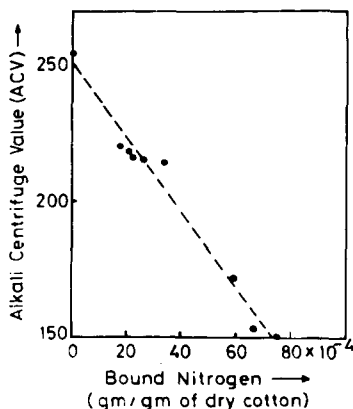


Fig. 1. Alkali centrifuge values vs. bound nitrogen for various resin-treated fibers.

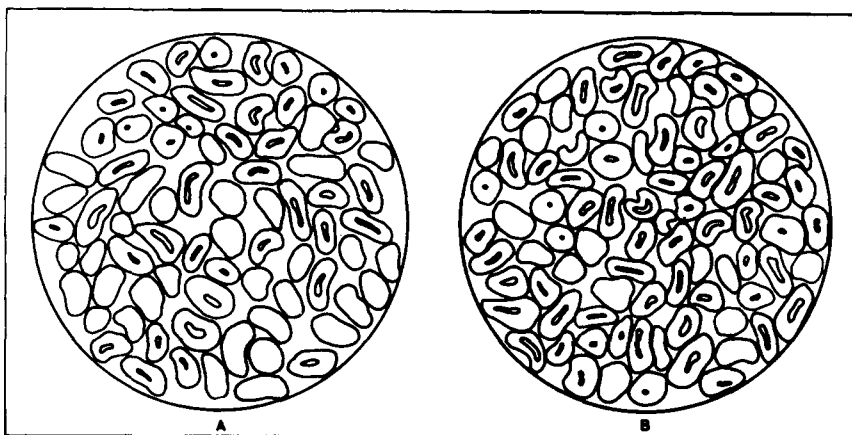


Fig. 2. Water-swollen cross sections of DMPU-treated cotton: (A) chemisorbed; (B) crosslinked.

Effect of Resin Treatment on Mechanical Properties of Cotton

Table III presents the test results on various mechanical properties of single fibers and fiber bundles of resin treated (both chemisorbed and crosslinked) cotton samples. Figures 5a and 5b show the plots of single fiber strength and bundle strength against bound nitrogen on treated fibers. Both these plots show that the strength of resin-treated samples decreases linearly with the increasing level of bound resin on fibers. Figure 6 shows the relationship between loss in bundle strength and crease recovery angles of fiber bundles. It can be seen that the increase in bundle crease recovery takes place at the expense of bundle strength, indeed as one would expect.

A very significant feature of the test results presented in Table III is the existence of a large correspondence between the mechanical properties of single fibers and those of fiber bundles for various resin-treated samples. Figures 7a and 7b illustrate the relationship which exists between bundle

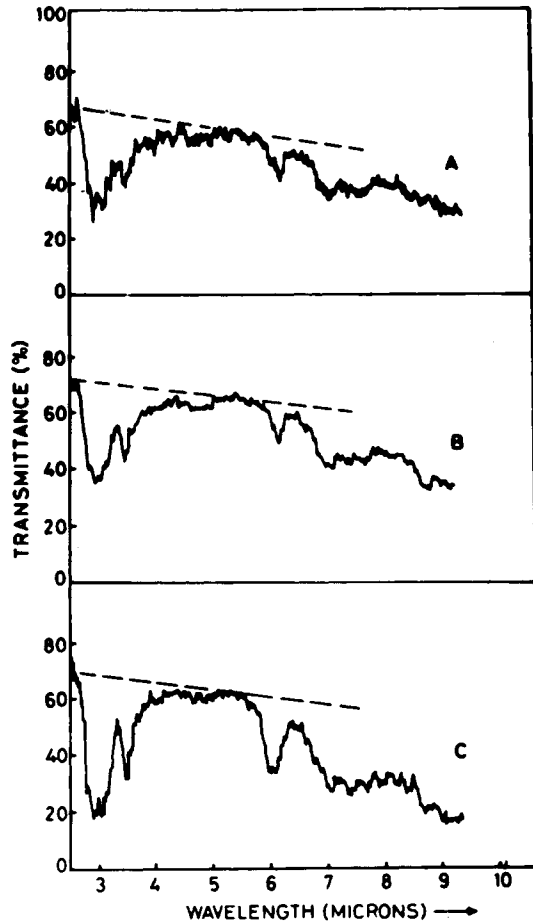


Fig. 3. Infrared absorption spectra of (A) untreated cotton; (B) cotton chemisorbed with DMPU; (C) cotton crosslinked with DMPU.

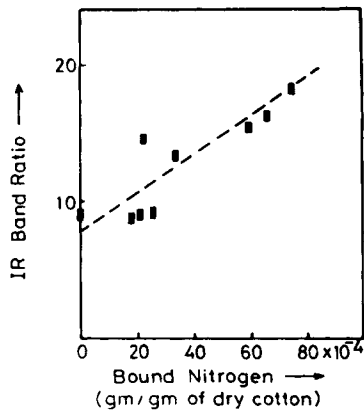


Fig. 4. IR band ratio vs. bound nitrogen.

TABLE III
Mechanical Properties of Single Fibers and Fiber Bundles of Cotton Chemically Modified with Different Resins

Sample designation	Single fiber properties*				Elastic tensile recovery, %	Bundle strength at 0" gauge length, g/tex	Bundle recovery angle, deg.
	Breaking load, g		Breaking extension, %				
	Mean	C.V.	Mean	C.V.			
Control	5.22	39.3	9.5	34.0	75.0	40.7	95
A (DMEU)	4.96	31.4	7.2	30.0	70.7	37.6	94
B (DMEU)	3.79	25.5	4.9	23.6	75.4	28.6	111
C (DMPU)	4.82	38.6	8.6	38.8	73.5	38.6	98
D (DMPU)	3.38	30.0	6.3	27.9	77.5	28.3	104
E (DHEU)	4.96	37.5	9.1	33.2	71.1	40.0	95
F (DHEU)	3.61	35.2	7.8	30.0	73.4	30.4	97
G (DMDHEU)	5.17	25.9	9.6	24.8	72.7	37.9	96
H (DMDHEU)	2.52	34.7	4.6	32.9	83.3	22.8	134

* Mean of measurements on 100 single fibers.

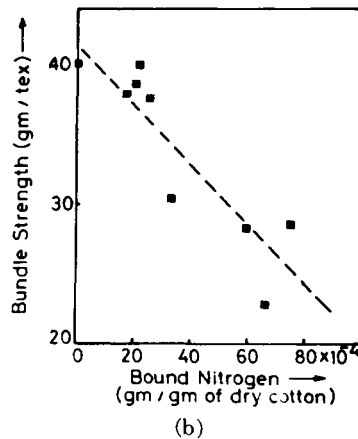
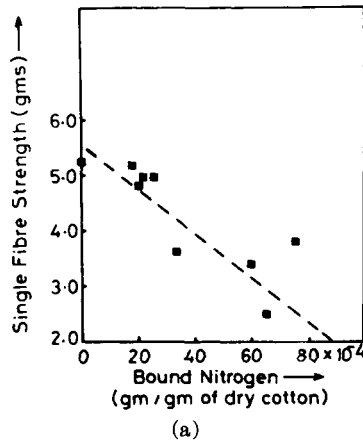


Fig. 5. Plots of strength characteristics of fibers vs. bound nitrogen for various resin-treated samples: (a) single fiber strength; (b) bundle strength.

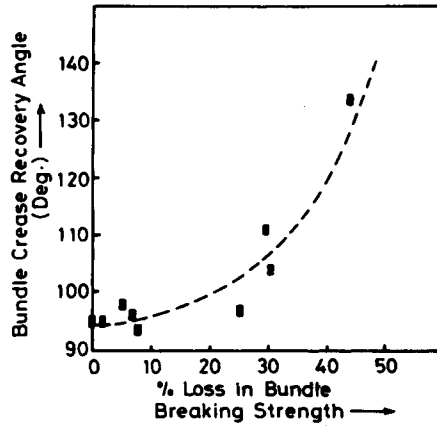


Fig. 6. Loss in bundle strength (expressed as percentage of control) in relation to crease recovery angle of fiber bundles.

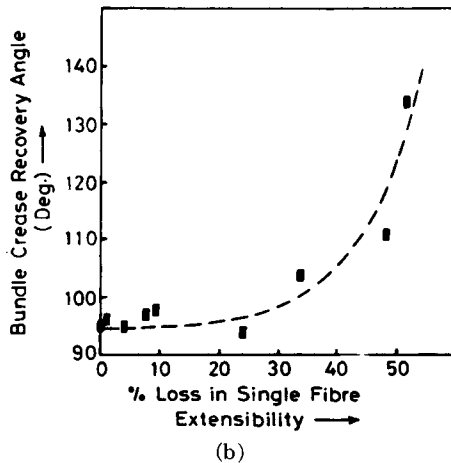
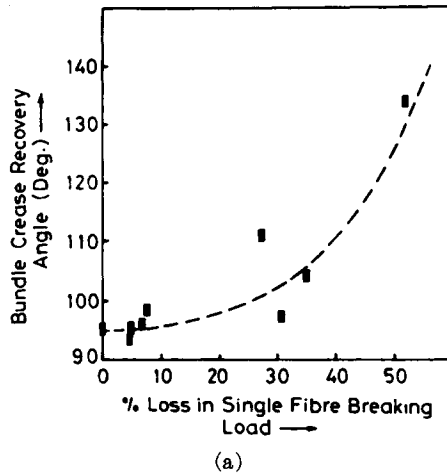


Fig. 7. Plots of bundle crease recovery angles vs. (a) % loss in single fiber strength; (b) % loss in single fiber extensibility.

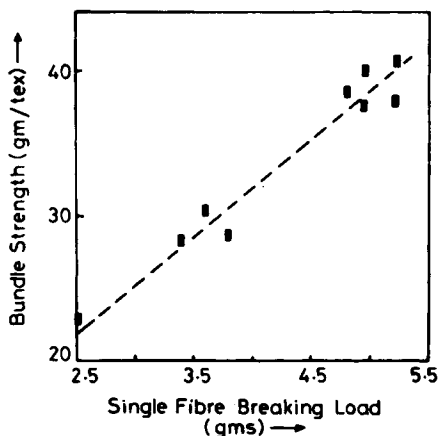


Fig. 8. Relationship between single fiber strength and bundle strength for various resin-treated samples.

crease recovery angle and per cent loss in single fiber strength and extensibility, respectively. These plots show that for any gain in crease recovery to take place upon resin treatment, there should be a loss in single fiber strength and extensibility. Figure 8 depicts a linear relationship between single fiber strength and bundle strength for various resin-treated samples. The correlation coefficient has a value of 0.95. Such a correlation has *not* been observed in case of strength properties of cotton fibers removed from boll at various intervals after flowering.¹⁶ This was attributed to the fact that in growing cotton fibers there is a systematic, progressive change in single fiber strength as well as in the surface characteristics of fibers with growth period, with the result that the bundle strength does not linearly increase with increase in single fiber strength. The existence of a very significant correlation between bundle strength and single fiber strength in the present case would therefore indicate that the surface properties (such as adhesion between adjacent fibers) of various resin-treated samples are similar in magnitude. Microscopic evidence¹⁷ has shown that the resin-treated cotton possesses a rather rough but more uniform surface in comparison to the untreated cotton. Finally, Figure 9 reveals a linear relationship between tensile recovery of single fibers and bundle crease recovery angles for various resin-treated samples. It should be noted that the DHEU-treated samples (E and F) which are devoid of HCHO do not show improvement in recovery properties (Table III).

Further examination of Table III reveals that for samples treated with DMEU and DMPU, the C.V. of single fiber properties is less for cross-linked samples (B and D) than for chemisorbed samples (A and C). This is attributed to the development of structural rigidity in crosslinked cotton. Though the C.V. of single fiber properties for DHEU-treated samples (E and F) is of the same order as that of the control, the same trend is still discernible. The reversal of this trend in DMDHEU-treated samples (G and H) is probably due to heavy strength loss in the crosslinked sample.

TABLE IV
Results of Linear Regression Analysis

Sr. no.	Properties correlated	Correlation coefficient
1.	ACV-bound nitrogen	-0.97
2.	IR band ratio-bound nitrogen	0.86
3.	% Loss in bundle strength-bound nitrogen	0.89
4.	% Loss in single fiber strength-bound nitrogen	0.83
5.	% Loss in bundle strength-bundle crease recovery	-0.84
6.	ACV-loss in bundle strength	-0.93
7.	WRV-total bound HCHO	-0.87
8.	single fiber strength-bundle strength	0.95
9.	Single fiber recovery-bundle crease recovery	0.92
10.	% Loss in single fiber strength-bundle crease recovery	0.83

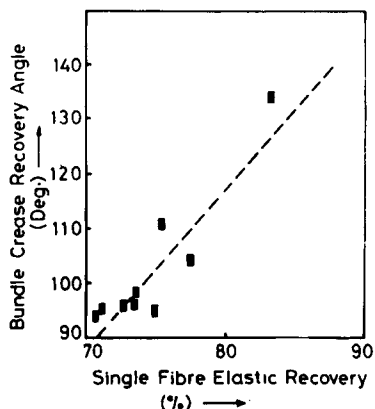


Fig. 9. Relationship between elastic recovery of single fibers and bundle crease recovery angles for various resin-treated samples.

Results of linear regression analysis on various parameters listed in Tables II and III are given in Table IV. Correlation coefficients obtained are of reasonably high magnitude. It is indeed remarkable that in various regression analyses performed, a common regression line can be fitted to the data points of both chemisorbed as well as crosslinked samples. This is attributed to the fact that in both chemisorbed as well as crosslinked products, bound resin exists partly as crosslinks and partly in monolinked state (Table II, fourth column). This would mean that various physicochemical and mechanical properties of resin-treated cotton are to a large extent independent of the manner in which resin fixation takes place on cotton or on the molecular structure of resin and depend mainly on the amount of resin bound on fibers. This conclusion need not apply to cases^{18,19,20} where cotton is subjected to swelling prior to crosslinking.

DISCUSSION

The chemisorption and crosslinking reactions with different resins play the same role on cotton fibers which the well-known resin finishing processes of wet fixation²¹ and pad-dry cure²² play on fabric. In the view of the authors, the chemisorption of resins occurs mainly on fibrillar surfaces (interfibrillar diffusion), whereas crosslinking with resins at the curing temperatures takes place within the fibrils also (inter- and intrafibrillar diffusion). This view is in accord with the results of Usmanov et al.²³ and is supported by significantly higher values of WRV and ACV for chemisorbed fibers than for crosslinked ones. Results obtained in the present work indicate that the former have a more porous, more accessible structure compared to the latter. This difference in swellability of structure is attributed to (a) difference in the level of bound resin between the two and (b) the rigidification and collapse of fiber structure upon crosslinking, due to catalytic activity at a high temperature. As stated earlier, chemisorption of resins on cotton is a one-ended reaction, whereas in crosslinking both the reactive groups of the resin molecule form a bond with cellulose molecules.

The decrease in strength as bound N increases, whether measured from fibers, yarns, or fabrics, is well recognized. Strength loss of cotton upon resin treatment arises on account of crosslink embrittlement and molecular degradation caused by the catalyst.²⁴

In order to improve smooth drying and crease recovery characteristics in a cotton fabric, a certain level of bound resin within fibers is essential. Effect of resin treatment on cotton fibers, apart from strength reduction, is an enhancement of tensile and bending recoveries of single fibers. Both these properties contribute to the crease recovery angles observed. Relationship between recovery properties of fibers and the crease recovery angles of fabric prepared from them has been discussed by Grant et al.²⁵ Recent work⁷ reported from ATIRA has shown that there exists a direct correspondence between the crease recovery angles of a treated fabric and those of bundles of warp and weft yarns extracted from the same. The only missing link which remains to be studied in correlating the recovery behavior of fibers with that of the corresponding fabric is the relationship between crease recovery angle of a yarn bundle and that of a bundle of fibers extracted from the yarn. It should be remembered, however, that fiber movement is very limited in a fabric on account of fabric structure. In a creased fabric, fibers are subjected to strains from tension, bending, and torsion. Fibers crosslinked in a fabric have superimposed on their natural configuration crimps determined by yarn and fabric geometry. These configurations significantly influence recovery.²⁶

This research has been financed in part by a grant made by the United States Department of Agriculture under P.L.480, Grant No. FG-In-398.

* This paper forms a part of S. B. Patel's Ph.D. thesis, to be submitted to Gujarat University.

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Received November 27, 1973

Revised March 5, 1974