Cross-Linking Decrystallized and Partially Acetylated Cotton Yarn with Dimethylol–Dihydroxy–Ethylene–Urea

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SYNOPSIS

Easy-care finishing of cotton fabrics invariably results in the loss of mechanical properties of the fabrics. There have been many attempts from time to time by different workers to reduce the above loss in the mechanical properties. But the problem is yet to be resolved satisfactorily. As the uniform distribution of cross-links in the resin-finished structure of cellulose is the key for achieving a better balance of mechanical properties, the present work aims at a suitable modification of the cellulose matrix to achieve the goal. The paper reports the mechanical properties of the modified and cross-linked cotton yarn. The cross-linked samples of the modification treatment involving swelling the yarn at normal stretch with 20% NaOH and partially acetylating without washing off the alkali have displayed higher tenacity, elongation, and toughness, with better easy-care properties as compared to the control.

INTRODUCTION

Although resin finishing of cotton is widely practiced, the problems accompanying this treatment such as loss in tensile strength and tear strength are yet to be satisfactorily dealt with. There has been a considerable amount of work reported[1-4] for obtaining a better balance of mechanical properties in the cross-linked cotton through various pretreatments before cross-linking. Among the various pretreatments attempted, the one involving decrystallization of cotton with sodium hydroxide followed by partial substitution as a means of preserving the decrystallized structure has attracted the attention of various workers.[5-8] In the above process, the swollen matrix of cellulose is prevented from collapsing on drying. The altered matrix of cellulose is thus more reactive with enhanced accessibility, lending itself to a better and uniform penetration of cross-linking reagent. Gigliardi and Werhner[9] have also pointed out the possibility of obtaining a better distribution of cross-links in such modified cotton. Recently, Iyer et al.[10,11] have shown that dye uptake and the resin uptake of the modified matrix is much higher than that of the unmodified one. However, enough details are not available on the resin uptake characteristics and its effect on the physical properties of the modified cellulose matrix. It is the purpose of this paper to report the effect on the tenacity retention and the wrinkle-recovery property of modified cotton yarn treated with different concentrations of resin.

MATERIALS AND METHODS

Dewaxed and kier boiled, doubled cotton yarn (2/30^s) of the MCU-7 variety was used in the study. The above yarn was treated as a control for comparison of various treatments.

Modification Treatments

Treatment T_1

The dewaxed and kier boiled yarn in the lea form was cross-linked with the resin dimethylol–dihydroxy–ethylene–urea (DMDHEU) under the following conditions: The samples were immersed for 8 min in the cross-linking bath containing the desired concentration of the resin using $MgCl_2$, $6H_2O$

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as catalyst and polyethanol as a softener and wetting agent and then passed through a padding mangle for a 70% wet pickup. The padded samples were mounted taut on a specially designed SS frame and dried at 80°C for 8 min and cured at 160°C for 3 min. The cured samples were given a mild detergent wash and air-dried.

Treatment T_2

In this treatment, yarn samples were given preswelling treatments employing 12, 15, and 20% NaOH (w/w) in the following manner: The samples were swollen in the alkali bath (M : L = 1 : 30) for 15 min under a slack condition followed by stretching to its normal length and retention in the stretched condition for 10 min. The samples were washed with hot water followed by cold water in the stretched condition. All the above operations were carried out on a specially fabricated mercerizing machine capable of stretching the yarn to different levels. After washing, the samples were given a souring treatment with 0.25% acetic acid for 0.5 h, followed by washing with tap water and air-drying. The dried samples were cross-linked as in treatment T_1.

Treatment T_3

The samples were swollen as in treatment T_2 under the stretched condition. Instead of washing off the alkali, the alkali pickup of the yarn was adjusted approximately to 100%. The yarn under the stretched condition was then acetylated using the procedure of Kulshreshtha and Dweltz[7] to get 4–5% acetyl content. The mercerized and acetylated samples were washed with tap water and air-dried. The dried samples were cross-linked as described in treatment T_1.

Yarn Properties

The acetyl content of the modified samples was determined by a standard chemical method[12] and the nitrogen percentage of the cross-linked samples were determined by the Kjeldahl method.

The yarn tensile strength and elongation were measured on an Uster automatic yarn tester, and the crease recovery angle, on a Metrimpex crease recovery tester using the method of Sitaram et al.[13] All the above measurements were carried out at 65% RH and 27°C.

Moisture regain determination was made from duplicate samples of about 0.5 g, each of which were predried over $CaCl_2$ and conditioned at 65% RH till constant weight was attained. They were later dried for 5 h at 110°C for determining the bone-dry weight. The regain is expressed as the percentage of the dry weight of the sample.

RESULTS AND DISCUSSION

Reactivity

The reactivity of the modified cotton yarn is presented through the following two parameters, namely, the resin uptake and moisture regain.

The nitrogen percentage of the cross-linked samples is taken as a measure of the resin uptake. Figure 1 presents the same. It is clearly seen that the mercerized and acetylated samples show increased reactivity toward the resin as compared to the unmodified yarn except in the case of samples swollen with 12% NaOH. It can also be seen that, in general, the reactivity of the substrate increases with the increase in the concentration of the alkali used for preswelling.

The moisture regain values of the samples swollen in 20% NaOH of treatments, T_1, T_2, and T_3 are shown in Table I. It is seen that the moisture regain of the modified yarn (T_3) is far superior to that of T_2 as well as of T_1. The above trend is observed at all resin concentrations. As moisture regain bears a direct relationship to accessibility of the structure, it can be concluded that the modification treatment has resulted in a matrix with enhanced accessibility.

Tenacity Retention

Table II shows the response of the modified cotton to tenacity at various concentrations of resin treatment. The dewaxed and kiered sample after mercerization with 12% NaOH showed a tenacity retention of 102% over the control sample, whereas the same sample after acetylation showed a marginal fall of 3% (99.2%). In case of treatment T_2 at 12% NaOH, the tenacity retention varied from 78.4% (1% resin) to 70.3% (3% resin), whereas at 4% resin the retention was 59.9% and showed a continuous fall in tenacity thereafter. In the case of treatment T_3, the tenacity retention varied from 79.0% at 1% resin to 71.0% at 3% resin and a steep fall was noted at 4% resin (58.9%), and at further higher concentrations of DMDHEU, the tenacity retention remained around 50–54%. A comparison between treatments T_2 and T_3 at 12% NaOH showed that the strength retention in T_3 was always higher than that of T_2. A similar trend was observed at 15%

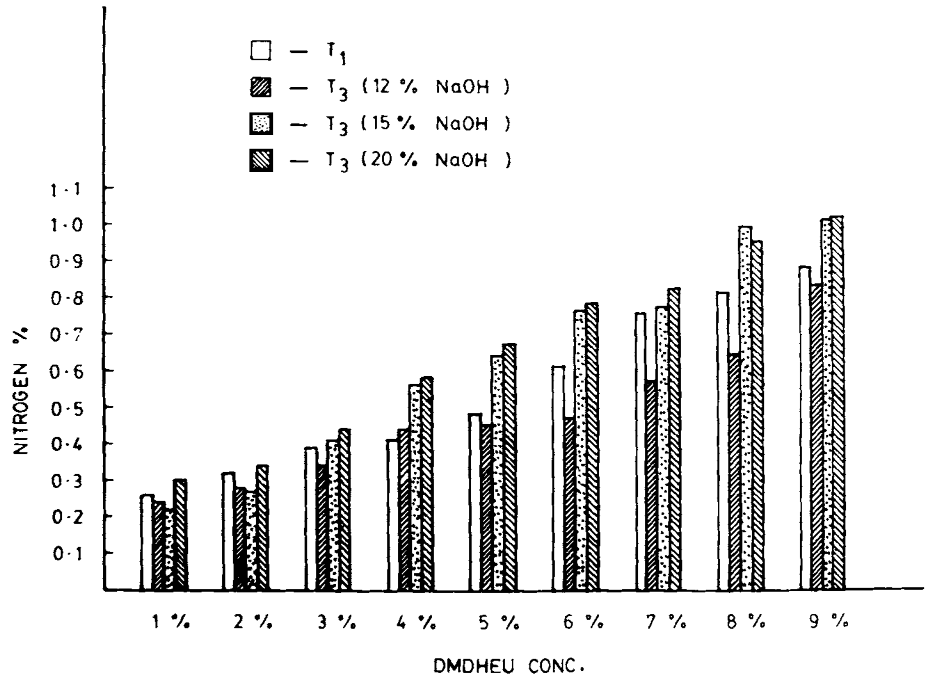

Figure 1 Nitrogen percentage values of treatment T_3 at different resin concentrations.

NaOH also, but strength retention in the latter was much higher. At 20% NaOH, treatment T_3 showed slightly lower tenacity retention up to a 4% resin concentration as compared to T_2, but, thereafter, up to a 9% resin level, it showed an increase in tenacity retention.

Elongation Retention

Table III presents the changes brought about in the yarn elongation in various treatments. It is seen that the yarn elongation of the uncross-linked samples falls on mercerization with NaOH under stretch conditions as compared to the control, whereas the same in treatment T_3 showed considerable improvement in elongation, as evident from the high elongation retention values. The yarn elongation of the resin-finished samples decreased with the increase in concentration of resin, probably due to the rigidification of the structure. It is observed that the cross-linked samples of treatment T_3 show much higher elongation retention as compared to treatment T_2 at all resin concentrations.

Table I Moisture Regain % of Treatment T_1, T_2, and T_3 at 20% NaOH Concentration at Different Resin Concentrations

DMDHEU (%)	T_1 (%)	T_2 (%)	T_3 (%)
0	6.3	9.0	9.3
1	6.5	8.4	9.2
2	6.5	8.2	9.4
3	6.7	8.4	9.4
4	7.0	8.3	—
5	6.7	8.4	9.3
6	6.5	8.4	9.2
7	—	8.7	9.4
8	7.3	8.4	9.0
9	6.3	8.2	9.4

Toughness Retention

Toughness is an indicator of the wear-life of fabric. It may be observed from Figure 2 that treatment T_3 as compared to T_2 gave uniformly higher toughness retention at all concentrations of NaOH and at all levels of resin. In the case of 20% NaOH, treatment T_3 recorded a very high toughness retention (45.3% at 9% level of DMDHEU) as compared to T_2 (36.7%).

Crease Recovery Angle

Table IV gives the crease recovery angle (CRA) of the modified as well as unmodified samples. The modified samples with their increased reactivity, as

Table II Tenacity Retention % Values after Treatments T_1, T_2, and T_3 at Different Resin Concentrations

		Treatment T_2			Treatment T_3		
DMDHEU (%)	Treatment T_1	12% NaOH	15% NaOH	20% NaOH	12% NaOH	15% NaOH	20% NaOH
0	100	102.2	106.4	107.7	99.2	103.0	97.2
1	80.2	78.4	89.4	90.6	79.1	94.2	86.1
2	63.4	72.7	83.5	89.6	74.5	86.2	85.6
3	61.0	70.3	81.9	85.6	71.0	85.6	84.1
4	48.9	59.9	73.3	83.6	58.9	81.6	82.3
5	35.2	56.5	72.4	78.6	57.4	78.1	80.6
6	34.8	50.6	71.1	76.5	55.9	76.8	78.5
7	34.5	49.9	63.9	76.0	54.4	71.1	77.5
8	32.7	48.9	58.6	75.0	50.4	70.6	74.0
9	31.2	47.7	58.3	70.5	49.9	67.6	73.0

evident from higher nitrogen content (Fig. 1), should have imparted a better CRA, but it is surprising to note the results to the contrary. Nevertheless, the CRA of the modified samples are higher than the corresponding CRA of the unmodified samples at all resin concentrations. It may also be observed from Table II that the CRA of the samples of treatment T_3 have invariably recorded a higher CRA or one on par with samples of treatment T_1. But at 20% NaOH, samples of treatment T_3 have registered a higher CRA accompanied by good tenacity retention.

Easy-Care Efficiency Index

In any attempt to impart wash-wear properties to fabric, utmost attention is paid to the wear-life. Wear-life is a parameter that is determined by a combination of various factors such as tensile strength, elongation, toughness, tear strength, abrasion resistance, etc. But the present work is on the yarn, and, hence, within the limited scope of the parameters studied, toughness is considered as an indicator of wear-life. To identify the treatment that provides a better balance of CRA and the wear-life, a new index was worked out. This is defined as follows:

$$\text{Easy-care efficiency index} = TR \times (\theta_1 - \theta_2)$$

where TR = toughness retention (over dewaxed and kiered sample), θ_1 = CRA of the treated sample, and θ_2 = CRA of the control (dewaxed and kiered) sample. The higher the index, the better is the balance of CRA and wear-life.

Table V gives the efficiency index of the samples. In general, it can be observed that with the increase in concentration of the resin the index comes down.

Table III Elongation Retention % Values after Treatments T_1, T_2, and T_3 at Different Resin Concentrations

		Treatment T_2			Treatment T_3		
DMDHEU (%)	Treatment T_1	12% NaOH	15% NaOH	20% NaOH	12% NaOH	15% NaOH	20% NaOH
0	100	64.0	62.0	62.0	84.0	66.0	70.0
1	95.0	60.0	68.0	70.0	84.0	64.0	70.0
2	76.0	56.0	64.0	62.0	82.0	64.0	68.0
3	74.0	50.0	64.0	62.0	68.0	62.0	68.0
4	61.0	46.0	60.0	60.0	64.0	60.0	68.0
5	59.0	42.0	58.0	58.0	64.0	54.0	66.0
6	57.0	38.0	52.0	58.0	58.0	52.0	66.0
7	53.0	34.0	50.0	56.0	52.0	52.0	64.0
8	52.0	34.0	44.0	56.0	52.0	48.0	62.0
9	51.0	32.0	42.0	52.0	48.0	48.0	62.0

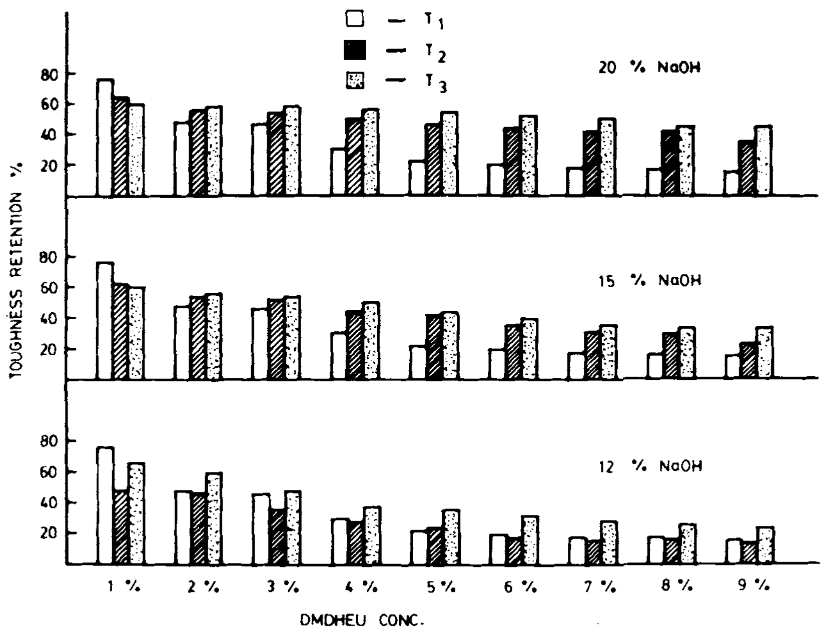

Figure 2 Toughness retention values of treatments T_1, T_2, and T_3 at different resin concentrations.

In the cases of T_2 as well as T_3 treatments, values of the index show an upward trend with the increase in the concentration of alkali used for preswelling. It is clear from Table V that treatment T_3 is superior to T_2 at all concentrations of alkali and also at all the resin levels. It is interesting to note that treatment T_3 at 20% NaOH gives the best balance of toughness and CRA.

The above discussion has brought out clearly the superiority of the modified substrate over the unmodified in cross-linking reactions. The treatment involving swelling with alkali followed by partial acetylation and subsequent cross-linking has shown encouraging results in terms of higher tenacity retention, better improvement in crease recovery angle, higher toughness retention, and better wear-life.

CONCLUSION

The properties of the resin-finished cotton are determined by the resin content and the nature of distribution of cross-links in the cellulose matrix. Because resin finishing is a heterogeneous reaction, the diffusion of the resin molecules and the distribution of the cross-links in the cellulosic structure are con-

Table IV Crease Recovery Angle (CRA) (in Degrees) after Treatments T_1, T_2, and T_3 at Different Resin Concentrations

		Treatment T_2			Treatment T_3		
DMDHEU (%)	Treatment T_1	12% NaOH	15% NaOH	20% NaOH	12% NaOH	15% NaOH	20% NaOH
0	168	212	210	204	208	216	196
1	202	228	232	230	228	240	228
2	226	240	240	234	244	246	240
3	238	250	246	238	254	256	248
4	246	262	252	242	262	262	262
5	260	270	258	250	274	268	264
6	268	280	264	256	282	272	268
7	274	286	270	260	290	274	272
8	276	294	278	264	294	284	280
9	280	296	282	268	298	290	284

Table V Easy-Care Efficiency Index Values after Treatments T_1, T_2, and T_3 at Different Resin Concentrations

DMDHEU (%)	Treatment T_1	Treatment T_2 12% NaOH	Treatment T_2 15% NaOH	Treatment T_2 20% NaOH	Treatment T_3 12% NaOH	Treatment T_3 15% NaOH	Treatment T_3 20% NaOH
1	12.95×10^2	14.10×10^2	19.46×10^2	19.65×10^2	19.92×10^2	21.71×10^2	18.09×10^2
2	13.98	14.65	19.22	18.91	23.22	21.53	20.95
3	15.78	14.43	20.83	18.58	20.77	23.36	22.88
4	11.62	12.97	18.48	18.57	17.72	23.03	26.32
5	9.57	12.09	18.90	18.70	19.45	21.10	25.53
6	9.90	10.75	17.76	19.54	18.47	20.75	25.90
7	9.70	9.97	16.32	19.60	17.26	19.61	25.79
8	9.18	10.46	16.61	20.16	16.51	19.66	25.70
9	8.90	9.79	13.96	18.35	15.60	19.82	26.27

trolled to a large extent by the physical state of the substrate. The modification treatment T_3, involving preswelling with sodium hydroxide at normal stretch and retaining the swollen structure through partial acetylation without washing off the alkali, seems to have an overall beneficial effect on the properties of the cross-linked yarn. Treatment T_3 has not only made the yarn more reactive but has also enhanced the accessibility that is evident from the higher moisture regain values. The higher reactivity of the yarn toward the resin DMDHEU is evident from the higher nitrogen percentage of the cross-linked samples at all resin concentrations in the above treatment.

However, in spite of the increased reactivity and higher uptake of resin, the modified samples have shown only marginal improvement in the CRA. The cross-linked samples in treatment T_3, especially at 20% NaOH preswelling concentration, display higher tenacity, elongation, toughness, and, in general, better wear-life as compared to the control. This may be due to the uniform distribution of cross-links in the modified cellulose matrix. The above modification treatment show promise from a commercial standpoint for the production of high-strength, wash-wear all-cotton textiles.

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