

## EFFECT OF CREASE-RECOVERY REAGENTS ON PHYSICAL PROPERTIES OF YARN MADE FROM AC-134 COTTON

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**Abstract.** The effect of three crease-recovery reagents, dimethylolethyleneurea, dimethylolethyleneurea formaldehyde and carbamate reactant with two catalysts magnesium chloride and zinc nitrate, on yarn and cloth made from AC-134 cotton were studied.

Crease-recovery reagents without catalysts have very little effect on strength of cellulosic material. They, however, cause reduction in strength when used alongwith catalysts, but catalysts alone do not reduce strength. The reduction in strength is, thus, due to the interaction of crease-recovery reagents with cellulose and not by the acid degradation. There is almost a linear relationship between increase in crease-recovery angle and loss in strength of fabrics. Premercerization plays an important role in preservation of strength. Maximum increase in strength and elongation was observed when the yarn was treated at original length with caustic soda of 50–60° Tw. Softeners (22–39.3%) have no appreciable effect in preservation of tensile strength.

Many of the finishes for cotton originated from attempts to transform this substance into an imitation of the fabric made from expensive fibres. Methods were devised, for example, to improve its lustre, to simulate silk, to make it stiff to imitate linen or to impart crease-recovery which is a prominent characteristic of wool fabrics. These and many other finishes have considerably increased the utility and beauty of this 'Cinderella' of fibres which has continued to maintain its supreme position of being the most popular and most widely used textile material.

During the last forty years, the textile finishing industry has made very rapid and diversified progress. This was not only an era of development of chemical industry, but also the period in which many socioeconomic changes have taken place. The later developments raised the cost of labour and created need for greater leisure time in industrially developed countries, which resulted in demand for 'instant' products. In the case of textiles, laundry service became prohibitive in cost and, at the same time, the housewife was left with little time for ironing. This state of affairs was responsible for the introduction of 'easy-care', 'minimum iron' and ultimately 'wash-and-wear' and durable-press fabrics. Extensive research has been done to impart to cotton fabrics the property of shedding creases during drying after washing. The methods developed, for this purpose, in the industrialised countries were imported into Pakistan and applied to locally produced cotton textiles, but very little work has been done to study their effect on Pakistani cotton. The present study was, therefore, carried out to study the adverse side effects such as reduction in tensile

strength, etc. of the 'easy-care' or crease-recovery finishes on Pakistani cotton. For this purpose, AC-134 cotton, which is a major, long staple commercial cotton variety, was selected. Since the properties of strength and elongation can be more conveniently and accurately determined with yarn than with cloth, most of the tests have been carried out on single yarns after treating these with the three crease-recovery reagents, under standard conditions of application.

The resin finishing process involves the application of multicomponent solutions to cotton followed by drying at low temperature (110°C) and then curing at high temperature (140–160°C for 5–3 min). The principal components of the treating formulation are compounds capable of reacting with cellulose, with themselves, or with both. A compound, usually acidic in nature, is added to catalyse the reactions. These reactions give the cotton fibre a resilience, so that after laundering or wrinkling, it tends to return to the shape in which it was cured.

The property of resistance to creasing is accompanied by loss in tensile and tear strength of fabric. Cross-linking which has been accepted as one of the most effective means of improving the crease-recovery properties of cotton,<sup>1,4</sup> is considered responsible for the loss in strength of cellulosic fibres.<sup>1</sup> Some workers believe that degradation of the cellulose by the strong acid catalyst causes losses in tear and tensile strength,<sup>4</sup> but losses have also been observed even when the catalysts used are harmless.<sup>5</sup> Catalysis of dimethylolurea with base, for example, also produces the same properties as that by the acid.<sup>2</sup>

The loss in strength is claimed to be minimized by premercerization and by the use of softeners in the treating emulsion. Premercerization plays an important role in preservation of strength, because

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it increases the strength of cellulosic material prior to treatment with crease-recovery agents. The increase in strength is due to improved orientation of crystallites caused during mercerising under tension and other factors.<sup>9</sup> The softening agents being lubricants counteract to some extent, the rigidity caused by cross-linking and, thus, improve tear strength although tensile strength may be adversely affected for the same reason, i.e. lubrication.<sup>5</sup>

### Material and Methods

**Yarn.** The yarn was spun from 100% AC-134 cotton on commercial scale at Kohinoor Textile Mills Lyallpur. An adequate supply was stored at the start of the work to ensure the uniformity of the material. The particulars of yarn are: count, 30; T.P.I., 24.15; uniformity, 13%; average strength, 260 g; neps/100 yards, 27; and moisture regain, 6.7% at 65% R.H. and 70°F.

**Fabric.** The samples of commercially bleached fabric (made from the same cotton) were taken from the finishing department of the same mills. The particulars are: ends/in, 130; picks/in, 80; weave, simple (one up, one down); warp, 30°; weft, 40°; and average strength, 300 lb.

**Crease-recovery Reagent.** R<sub>1</sub>, dimethylolethyleneurea (Calaroc EU, ICI product); R<sub>2</sub>, dimethylolethyleneurea formaldehyde (Unires EU, UCI product); and R<sub>3</sub>, carbamate reactant (Finish P Sandoz product).

**Catalysts.** C<sub>1</sub>, magnesium chloride (MgCl<sub>2</sub>·6H<sub>2</sub>O); and C<sub>2</sub>, zinc nitrate [Zn(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O].

**Softeners.** S<sub>1</sub>, cationic softener; Ceranine HCS (Sandoz product); and S<sub>2</sub>, anionic softener; Primenit VS (Hoechst product).

**Method of Treatment.** Yarn skeins were impregnated with the precondensate for about 2 min, squeezed to a wet pick up 120–125% dried at 110°C and, then, cured at 155±5°C for 4 min. Finally, the skeins were left hanging for 3–4 hr to gain moisture under standard conditions (65±2% R.H., 70±2°F). The fabric pieces were also treated with the same method, but wet pick up was 80–90%.

**Mercerization.** Cotton yarn skeins were mercerized with sodium hydroxide solutions at 30, 40, 50, 60, 70 and 80°Tw concentrations. These were first mounted on a reel and stretched by the adjustment of arms of the reel to keep the length of yarn constant. Skeins were kept in the liquor for 2–3 min at 23±2°C and then washed thoroughly while still under tension. The last traces of caustic soda were removed by neutralizing with oxalic acid.

**Strength and Elongation of Single Yarn.** Strength and elongation of single yarn were determined with the help of 'Uster Automatic Yarn Strength Tester'. One hundred breaks were tested for each sample.

**Tensile Strength of Fabric.** Tensile strength of fabric was tested with the help of 'Lea and Fabric Strength Tester' according to ASTM Committee (1968) (ASTM Designation : D 1682–64, part 24).

**Crease-recovery Angle of Fabric.** Crease-recovery angle of fabric was measured on 'Metefem Checking Instrument for Wrinkle-Undo (Hungary).

### Results and Discussion

The crease-recovery property of cellulosic fabrics is associated with the formation of cross-links or high molecular weight complexes of chemical reagents in between the molecular chains. The improvement in crease-recovery is normally accompanied by reduction in tear strength of fabric. To determine the relationship between crease-recovery angle and tensile strength, cotton fabric made from AC-134 cotton was treated with the three reagents R<sub>1</sub>, R<sub>2</sub> and R<sub>3</sub> at different concentrations varying from 0 to 20% (w/v) and a fixed concentration of catalyst C<sub>1</sub> at 1.4% (w/v). The material was padded, dried and cured and then the crease-recovery angles and tensile strength of the samples were determined. The results are given in Table 1 (Fig. 1). It is clear from Fig. 1 that there is essentially a linear inverse relationship between tensile strength and crease-recovery angle.

To further study the effect of the crease-recovery reagents on strength and elongation on AC-134 cotton yarn, skeins were impregnated in the solutions of the three reagents R<sub>1</sub>, R<sub>2</sub> and R<sub>3</sub> at varying concentrations with a fixed concentration of the catalyst C<sub>1</sub>. The effect of concentration of reagents on strength and elongation of yarn is illustrated in Fig. 2. There is a large reduction in strength and elongation of yarn up to about 4% (w/v) concentration of all the three reagents, but at higher concentrations, there is only a slight additional reduction in their values.

**Effect of Catalysts.** It is well known that the improvement in crease-recovery of fabrics or reduction in its strength is possible only when a catalyst is present alongwith crease-recovery reagents. This

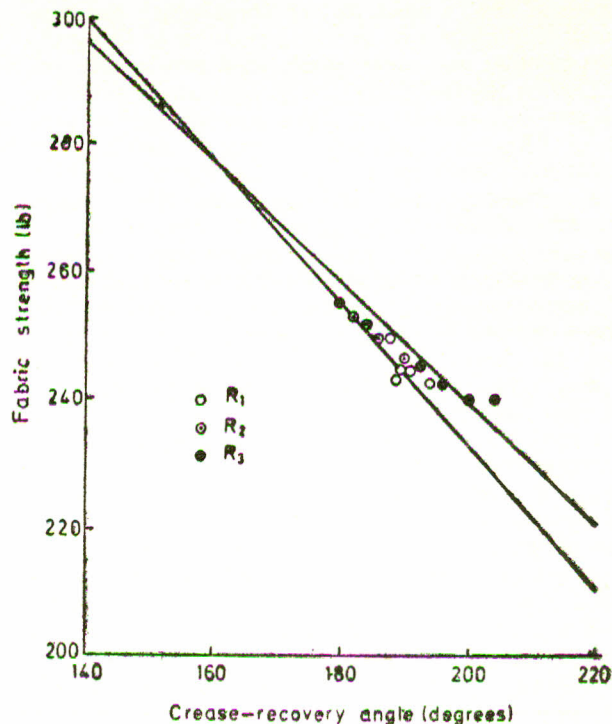


Fig. 1. Relationship between tensile strength and crease-recovery angle of fabric.



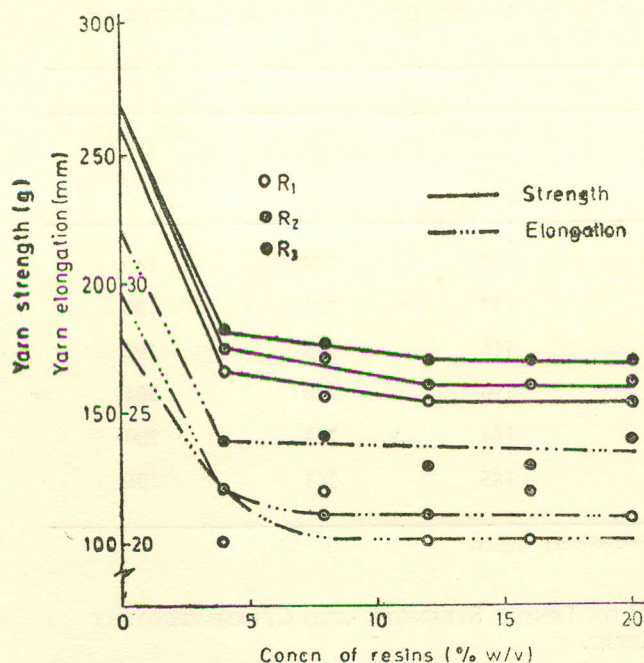


Fig. 2. Effect of resin concentration on yarn strength and elongation when catalyst  $C_1$  is kept constant (1.4% w/v).

is confirmed by comparing the results given in Tables 1 and 2 which show the effect of crease-recovery reagents with and without catalyst, respectively on fabric strength and crease-recovery angles. It may be noted that, with increase in concentration of crease-recovery reagent on the fabric, the crease-recovery angle improves and tensile strength falls progressively. In the absence of catalyst, there is only a very slight reduction in strength and increase in crease-recovery for the same concentrations of the reagents. Further experiments were done on yarn which was treated with different concentrations of the reagents without catalysts. The results are illustrated in Fig. 3, which indicate that there is only a slight reduction in strength and no appreciable change in elongation of yarn thus treated.

The effect of catalysts during crease-recovery treatments on tensile strength of the treated fabrics is controversial. Many investigators<sup>4,8</sup> consider that the reduction in strength is due to hydrolysis of cellulose chains by the acid generated from catalysts during heat treatments. On the other hand, some workers point out that cotton loses strength even when the catalysts applied are harmless<sup>3</sup> or alkaline.<sup>2</sup> Furthermore, the tenacity of viscous rayon, a cellulosic fibre, is improved by resin treatment instead of falling. Marsh and Wood provided direct evidence of the lack of chemical degradation by acid hydrolysis when they showed that the removal of amino-aldehyde resin from a treated fabric restored its original physical properties. To further investigate the point especially with reference to Pakistani cottons, experiments were carried out on 30<sup>s</sup> yarn, which was treated with different concentrations of catalysts without the crease-recovery reagents under conventional conditions of crease-recovery treatments. There is practically no loss in tensile strength and elongation of yarn on treat-

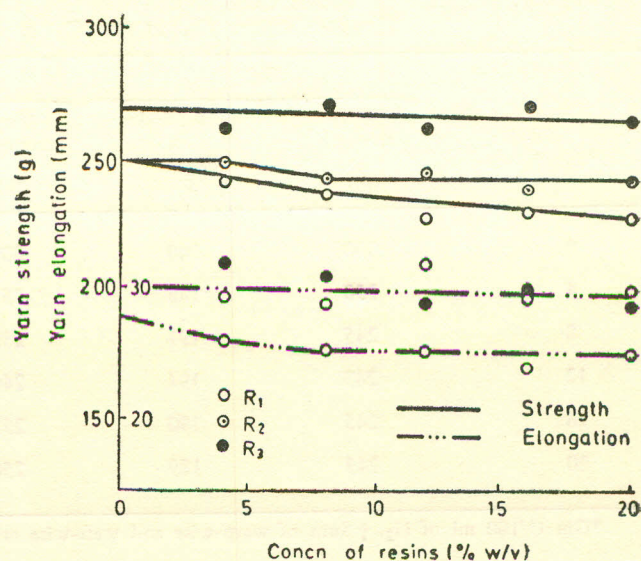


Fig. 3. Effect of resins (used without catalysts) on strength and elongation of yarn.

ment with catalysts alone (Table 3). These observations support the view that losses in strength and elongation are caused by crease-recovery reagents and not by degradation due to acid generated from catalysts. The catalysts, thus, appear to help the formation, cross-links or resin agglomeration in between the cellulosic chains, which reduce the strength of material by blocking the slippage of molecular chains past one another.

For a more detailed study of the effect of catalysts in the presence of crease-recovery reagents on the strength and elongation of yarn, yarn skeins were treated with a fixed concentration (8.0% w/v) of  $R_1$ ,  $R_2$  and  $R_3$ , but varying concentration of  $C_1$  and  $C_2$ . The results are shown in Tables 4 and 5. It may be seen that, with all the three reagents, there is a great reduction in strength and elongation up to a concentration of 1.6% (w/v) of magnesium chloride ( $C_1$ ) and with further increase in concentration of  $C_1$ , there is only a slight reduction in both strength and elongation of yarn. Zinc nitrate ( $C_2$ ) also shows similar trend, but large reductions in strength and elongation are indicated at a comparatively lower concentrations of 0.6–0.8% (w/v). The maximum loss in strength of yarn by the addition of catalyst  $C_1$  are 37.2, 40.8 and 37.0% with  $R_1$ ,  $R_2$  and  $R_3$ , respectively. While similar figures for  $C_2$  are 37.6, 36.0 and 37.0%. The maximum reduction in elongation of yarn for  $C_1$  are 25.0, 28.0 and 24.2% and for  $C_2$  are 28.6, 28.6 and 26.6% with  $R_1$ ,  $R_2$  and  $R_3$  in respective order.

*Effect of Mercerization.* As has already been discussed, the strength and elongation of yarns are adversely affected by the use of crease-recovery reagents. Attempts have been made to reduce the losses in strength and elongation by mercerization. In the present study, yarn was mercerized at constant length, at various concentrations of caustic soda and, then, treated with crease-recovery reagents (resin 8% +  $C_1$  1.4% w/v). The strength of treated yarn increased with increase in concentration



TABLE 1. EFFECT OF RESIN CONCENTRATION ON TENSILE STRENGTH AND CREASE-RECOVERY ANGLE OF FABRIC WHEN CATALYST C<sub>1</sub> IS KEPT CONSTANT (1.4% w/v).

Concn of resins (*w/v)%	R <sub>1</sub>		R <sub>2</sub>		R <sub>3</sub>	
	Warp strength	C. R. angle in degrees † (W+F)	Warp strength	C. R. angle in degrees (W+F)	Warp strength	C. R. angle in degrees (W+F)
	(lb)		(lb)		(lb)	
0	300	140	300	140	300	140
4	250	188	255	182	255	180
8	245	191	250	186	245	192
12	243	194	246	190	240	204
16	245	190	252	184	240	200
20	243	189	250	185	243	196

\*Gm O/100 ml of H<sub>2</sub>. † Sum of warp-wise and weft-wise crease-recovery angles.

TABLE 2. EFFECT OF RESINS (USED WITHOUT CATALYSTS) ON TENSILE STRENGTH AND CREASE-RECOVERY ANGLE OF FABRIC.

Concn of resins (w/v %)	R <sub>1</sub>		R <sub>2</sub>		R <sub>3</sub>	
	Warp strength	C. R. angle in degrees (W+F)	Warp strength	C. R. angle in degrees (W+F)	Warp strength	C. R. angle in degrees (W+F)
	(lb)		(lb)		(lb)	
0	300	140	300	140	300	140
4	285	147	290	145	295	142
6	283	147	292	144	290	147
8	280	150	290	146	285	146
10	270	154	287	147	286	150
12	275	151	285	146	285	150
14	280	152	285	144	283	145
16	275	153	290	143	280	152
18	273	152	286	143	281	151
20	270	153	285	146	280	148

of caustic soda up to a maximum at 50–60°Tw strength of caustic soda. Beyond this concentration, strength is somewhat diminished (Table 6). The maximum increases in strength of yarn due to pre-mercerization followed by treatment with R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> (8% + C<sub>1</sub>) (1.4% w/v), are up to 50, 66 and 55% and increases in elongation are 45.5, 50.0 and 50.0% respectively. The strength of treated material under optimum conditions of mercerization ranges from 250 to 274 and elongation 25.5 to 32 mm while strength and elongation of unmercerized and untreated yarn ranges from 250 to 270 and 28 to 31mm respectively.

On comparing the results of mercerized and treated yarn with unmercerized and untreated yarn, it is concluded that losses in strength and elongation

TABLE 3. EFFECT OF MAGNESIUM CHLORIDE AND ZINC NITRATE ON STRENGTH AND ELONGATION OF UNTREATED YARN.

Concn (w/v %)	Magnesium chloride		Zinc nitrate	
	Strength (g)	Elongation (mm)	Strength (g)	Elongation (mm)
	0	210	26.00	210
0.4	216	27.50	214	26.00
0.8	210	27.50	210	26.00
1.2	214	26.00	210	25.00
1.6	210	27.00	206	27.50
2.0	206	26.00	210	26.50

caused by cross-linking can be eliminated by premercerization. The results detailed in Table 6 also support the view of other investigators. They reported that yarn-mercerized grey fabric after wash-and-wear treatment equalled or exceeded unmercerized uncross-linked fabric in both break and tear strength.<sup>7</sup> Premercerization is effective in decreasing strength losses in cotton during cross-linking, because it increases the strength prior to cross-linking. The gain in strength of mercerized yarn, under tension, arises from increase in compactness of structure and fibre cling.<sup>6</sup> The high

orientation of crystallites is also responsible for retaining high strength after cross-linking.<sup>9</sup>

*Effect of Softeners.* It is well known that the use of softeners alongwith crease-recovery reagents improves tear strength of the fabrics considerably. To study their effect on tensile strength and elongation of treated yarn, two softeners Ceranine HCS(S<sub>1</sub>) and Primenit VS(S<sub>2</sub>) were used at different concentrations. The results are given in Table 7. These indicate that there is virtually no change in tensile strength or elongation of the treated material. To further investigate the effect, yarn was treated with

TABLE 4. EFFECT OF ADDITION OF MAGNESIUM CHLORIDE (C<sub>1</sub>) IN RESINS (8% w/v) ON STRENGTH AND ELONGATION OF YARN.

Concn of C <sub>1</sub> (w/v %)	R <sub>1</sub>		R <sub>2</sub>		R <sub>3</sub>	
	Strength	Elongation	Strength	Elongation	Strength	Elongation
	(g)	(mm)	(g)	(mm)	(g)	(mm)
0	250	28.00	250	25.00	270	31.00
0.4	175	22.00	164	18.00	186	24.00
0.6	173	21.25	157	18.50	176	24.00
0.8	170	22.00	154	18.00	179	24.00
1.0	167	21.00	160	21.50	175	24.00
1.2	165	21.50	152	20.00	173	23.50
1.4	157	22.00	148	18.00	174	25.00
1.6	165	21.00	151	22.00	170	24.00
1.8	160	21.50	150	19.00	169	24.50
2.0	158	22.50	149	20.00	170	24.25

TABLE 5. EFFECT OF ADDITION OF ZINC NITRATE (C<sub>2</sub>) IN RESINS (8% w/v) ON STRENGTH AND ELONGATION OF YARN.

Concn of C <sub>2</sub> (w/v %)	R <sub>1</sub>		R <sub>2</sub>		R <sub>3</sub>	
	Strength	Elongation	Strength	Elongation	Strength	Elongation
	(g)	(mm)	(g)	(mm)	(g)	(mm)
0	250	28.00	250	28.00	270	30.00
0.4	171	24.25	176	21.50	186	24.00
0.6	156	20.00	175	22.50	176	24.00
0.8	158	21.00	160	20.00	170	22.00
1.0	156	21.00	160	20.00	171	24.00
1.2	156	20.00	161	20.00	173	25.50
1.4	158	21.50	164	20.00	170	24.00
1.6	157	21.50	160	21.50	170	24.00
1.8	156	21.00	160	20.50	172	24.00
2.00	156	22.50	162	20.00	170	24.00



two softeners without crease-recovery reagents. The results are given in Table 7, which indicate that again there is no change in tensile strength or in elongation of yarn on treatment with these two softeners which are commonly used in crease-recovery process. These experiments show that increase in tear strength of crease-recovery treated fabrics is not due to increase in tensile strength of yarn, but is caused by some other factors.

### Summary

When the crease-recovery reagents, namely, dimethylolethyleneurea  $R_1$ , dimethylolethyleneurea formaldehyde  $R_2$  and carbamate reactant  $R_3$  are applied alongwith catalyst magnesium chloride  $C_1$  or zinc nitrate  $C_2$ , there is a great reduction in strength and in elongation of cotton yarn and fabric. A linear relationship was found in crease-recovery angle and strength of fabric on treatment with all the three reagents.

The maximum losses in strength and elongation are caused when about 12% (w/v) resins were used with fixed concentration of  $C_1$  (1.4%, w/v). To study the effect of catalyst, fabric samples were treated with  $R_1$ ,  $R_2$  and  $R_3$  without catalysts under normal conditions of application. It was learnt that there is only a very slight improvement in crease-recovery angles and very little reduction in tensile strengths of the fabrics thus treated. To elucidate the controversial role of acid generating catalyst in lowering tensile strength of cellulosic material, samples of yarn were treated with both magnesium chloride and zinc nitrate at different concentrations under normal application conditions and, then, strength and elongation of yarn were determined. It was found that no significant difference exists between the untreated and treated yarns. This supports the view that reduction in strength of cellulosic material on giving crease-recovery finishes, is mainly due to crease-recovery mechanism and not by acid degradation.

TABLE 6. EFFECT OF PREMERGERIZATION AT VARIOUS CONCENTRATIONS OF NaOH ON STRENGTH AND ELONGATION OF YARN (TREATMENT AFTER MERCERIZATION RESINS (8% +  $C_1$  1.4% w/v).

Concn of NaOH (°Tw)	$R_1$		$R_2$		$R_3$	
	Strength (g)	Elongation (mm)	Strength (g)	Elongation (mm)	Strength (g)	Elongation (mm)
0	167	22.00	158	20.00	177	20.00
30	195	22.50	210	20.00	201	21.00
40	200	26.00	210	24.00	250	26.00
50	239	25.50	226	30.00	274	30.00
60	250	32.00	262	27.00	269	28.00
70	235	30.00	262	29.00	265	29.00
80	230	30.00	260	30.00	265	29.00

TABLE 7. EFFECT OF ADDITION OF SOFTENERS IN PRECONDENSATE ON STRENGTH AND ELONGATION OF YARN ( $R_1$  8% +  $C_1$  1.4% w/v).

Concn of softeners (w/v %)	Untreated				Treated			
	$S_1$		$S_2$		$S_1$		$S_2$	
	Strength (g)	Elongation (mm)	Strength (g)	Elongation (mm)	Strength (g)	Elongation (mm)	Strength (g)	Elongation (mm)
0	250	30.00	250	32.00	172	23.00	170	22.50
1	250	30.00	247	30.00	176	22.00	170	23.00
2	250	30.00	248	31.00	170	22.00	172	24.00
3	254	29.00	244	29.00	170	23.00	168	21.00
4	250	29.00	249	31.00	174	21.00	170	21.50
5	250	30.00	250	32.00	169	22.00	168	22.00



With fixed concentration of crease-recovery reagents (8% w/v), the reduction in strength and elongation of cotton yarn is comparatively high up to concentrations of 1.4–1.6% w/v of C<sub>1</sub> and 0.6–0.8% w/v of C<sub>2</sub>; but, above these concentrations, the changes in strength and elongation are low.

Effect of premercerization on the tensile strength of cellulosic material which have undergone crease-recovery treatment was also studied. It was learnt that, as compared with the treated yarn, the strength and elongation increased with all the three reagents at different concentrations of caustic soda solution. The maximum increase in strength and elongation was observed when the yarn was treated at constant length with caustic soda solution of 50–60°Tw concentrations.

Softeners are normally added in commercial recipes for improving tear strength of cellulosic fabrics. Yarn was treated with two softeners (Cera-nine HCS and Primenit VS) at different concentrations with and without crease-recovery reagents.

No appreciable difference in strength and elongation was noticed in either case.

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