

EFFECT OF MERCERIZATION FOLLOWED BY CROSS-LINKING ON COTTON FABRIC PROPERTIES

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ABSTRACT

This study aims to establish optimum conditions of mercerization and resin finishing treatments and obtain cross-linked cotton fabrics having good wrinkle resistance properties without severe strength loss. Plain weave cotton fabrics were mercerized using NaOH at concentrations ranges from 200 to 300 g/l. The optimum conditions of mercerization for every fabric structure depend on its weight per unit area. Results show that tension mercerization improved strength properties by changing the kidney shape of cotton fibres to elliptical cross-section and higher orientation. These fabrics were subjected to crease recovery treatments using Dimethylol Dihydroxy Ethylen Eurea (DMDHEU). Mercerization increased breaking load, fabric tear, and abrasion resistance by a percent ranges from 10 to 20 %, 8 to 21 %, and 7.1 to 17.3 % respectively. On the contrary, resin finish decreased the breaking load, fabric tear, and abrasion resistance by a percent 15 to 30 %, 8 to 38 %, and 22.4 to 43.9 % respectively. So, performing optimum mercerization followed by cross-linking retained enhancement in strength properties of cotton fabrics.

Keywords: Mercerization, Crease recovery, Fabric dimensional and mechanical properties.

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1. INTRODUCTION

Mercerization is widely used on cotton for many different reasons, it increases luster and softness, gives greater strength, soft, comfort, and improves affinity for dyes and wettability. Nowadays considerable quantities of cotton yarns and fabrics are mercerized to improve luster and/or dye uptake, and more recently without tension to produce stretch materials^[1, 4]. Resin or wrinkle resistance finishing is widely used in the textile industry to impart wrinkle resistance to cotton fabrics and garments. Considerable loss in strength and abrasion resistance of the finished fabrics has been a major concern for the industry. The strength of the fibre depends on how much the cross-linked chains can still be mutually displaced under tension in order to sufficiently resist the applied load. The rigid crosslinks that are formed with DMDHEU obviously prevent the redistribution of stress by preventing segmental movement within the fibre microstructure. The cross-linking of cellulose molecules with these relatively rigid crosslinks causes stiffening of the cellulosic macromolecular network and fibre embitterment, thus dropping the mechanical strength of the

treated cotton fabrics. These same mechanisms are responsible for reduced mechanical properties of the fibre surface, thus leading to strength loss^[1-5, 16].

2. REVIEW OF LITERATURE

Prashant^[6] treated cotton fabrics with various alkylated derivatives of DMDHEU then subjected it to hydrolysis by urea-phosphoric acid, hot 23% sodium hydroxide, and urea-phosphoric acid followed by sodium hydroxide. Stability of the cross-links generated by these resins depended on both the degree of alkylation of DMDHEU and the nature of the alkyl cap. This stability was reflected in greater amounts of residual nitrogen and formaldehyde after hydrolysis. Those residues, in turn, affected the residual breaking strength, flex abrasion resistance, and wrinkle recovery of the hydrolyzed fabrics. The stability of the cross-links also correlated well with formaldehyde release data for the treated fabrics. Lei and Lickfield^[7] subjected cotton fabrics to different concentrations of DMDHEU catalyzed with Mg Cl₂, their objective was to evaluate the effect of acid degradation and cross-linking of cellulose on the overall

strength loss, and investigate the relationship between the strength loss due to cross-linking and the molecular structure of the cross-linking agent. At the same temperature, the activated catalyst caused greater tensile strength loss for the cotton fabric cross-linked with DMDHEU than the standard catalyst system. The percent fabric tensile strength loss increased as the concentration of the catalyst is increased up to approximately 2% concentration, above which the fabric tensile strength loss levels became constant. However, for the activated catalyst system, the fabric tensile strength loss continued to increase with increasing catalyst concentration. This increased loss in fabric tensile strength was due to the higher acid concentration in the activated catalyst system. Weilin Xu et al.^[8, 14, 15] performed a treatment on cotton fabrics at different curing temperatures with different concentrations of 1,2,3,4-butanetetracarboxylic acid (BTCA) catalyzed with sodium hypophosphite (SHP). Strength retention and wrinkle recovery angle (WRA) were tested, and the results declared that high curing temperature and high BTCA concentration reduced fabric strength and increase WRA. Chen^[9] declared that the combination of inadequate abrasion resistance and relatively severe tensile strength loss has been the major disadvantage for durable press finished 100% cotton fabrics. The overall objectives of that work was to: characterize the surface nature of the wear of durable press (DP) finished cotton fabric; investigate the cause and mechanism of loss in abrasion resistance of cross-linked cotton fabrics; further develop relationships between the molecular structure of cross-linking agents and their effect on the mechanical properties of cross-linked textile structures; and develop technology for improving the abrasion resistance of DP finished cotton fabrics by preventing and/or removing the cross-links on the fabric surface. Olivera^[10] made a comparison of the cross-linking effect on mercerized and un-mercerized cotton cellulose. The results appeared that best cross-linking results were obtained on mercerized samples treated with the highest mass fraction of BTCA (7 %). Structural changes caused by mercerization decreased crystallinity of fibers to 20 %, which increased fiber absorption.

Weilin Xu^[11] made a treatment on cotton fibers and their fabric with BTCA with the catalyst SHP at different concentrations of treating agent and different curing temperatures. Results showed that low curing temperatures and low agent concentrations have a slight effect on the reduction of crystallinity and crystallite size, but when the curing temperature is higher than 180°C and the BTCA concentration is higher than 6%, crystallite size and crystallinity decreased significantly. Initial slight crystallinity loss induced high strength loss in the fabric. That was perhaps due to the initial slight reduction of crystallinity, which caused serious interfacial binding strength losses between the crystals and the amorphous regions. Avinash^[12] subjected fabric samples to NaOH and KOH pretreatments then resin finishing. The treated samples were tested to determine the influence of alkali pretreatments on the tenacity, abrasion resistance, and crease recovery of resin-finished lyocell. Alkali pretreatments resulted in a higher surface concentration of the cross-linking reagent in fabrics, leading to lowered crease recovery and abrasion resistance.

The pretreatments also exerted a deleterious influence on the tensile strength in resin-finished samples. However, the differential distribution of cross-linking reagent within the textile structure did not appear to be the only factor responsible for the changes observed in substrate properties; other factors also were responsible for the results observed. Spencer^[13] made a treatment on 100% plain weave cotton fabric which has been scoured, desized, bleached, and heat set. The optimal treatment evaluated with this work was to provide the largest increase in dry WRA, the largest increase in wet WRA, the best DP rating, no increase in stiffness, no discoloration, and moderate increases in strength and elongation as compared to the other treatments. That method involved carboxy methylation in two steps. The first step was NaOH treatment at concentration 20% using a pad-dry procedure. The second step was a pad-batch treatment of the fabric with 1.0 M chloroacetate. A third step was fabric cross-linking. It was performed through exhaustion of 3% cationic glycerin in a bath, followed by a pad-dry-cure sequence. Most of previous research studied either mercerization effect only or resin finishing. Some of them deal with the combined effect between mercerization and wrinkle resistant finish, from here the need of continued work was started to complete the research on the effect of mercerization then cross-linking treatments on cotton fabrics structure and performance.

3. EXPERIMENTAL WORK

3.1. Aim of the present work

The main objective of the present work is to establish optimum conditions of mercerization and resin finishing treatments with respect to fabrics having different structures and to obtain mercerized cotton fabrics having good wrinkle resistance properties without great strength loss.

3.2. Materials and Chemical Treatments

In order to study the effect of mercerization and cross-linking treatments on cotton fabrics structure and performance, the general plan of this research will be as follow:

- a) Bleached cotton fabrics were subjected in Misr / Mehalla Spinning and Weaving company to these treatments:
 - Mercerization with different NaOH concentrations viz., 200, 220, 240, 260, 280 and 300 g/l under the same conditions (mercerizing machine speed, fabric tension, rollers pressure and temperature), and;
 - Mercerization under different mercerizing times 10, 15, 20, 25, 30 sec using same NaOH concentration 260 g/l.
- b) Bleached and mercerized cotton fabrics were heated with a pad-bath containing DMDHEU as a cross-linking agent along with magnesium chloride catalyst and softeners based on fatty acids and wetting agent. DMDHEU concentrations were 40, 60, 80, 100 g/l and catalyst (Mg Cl₂ · 6 H₂O) was used at concentrations of 8, 12, 16, 20 g/l, respectively. After padding to a wet pick 80 %, the fabrics were dried at ambient conditions then cured at 160°C for different curing times (3, 4, 5 minutes).

Table 1. Specifications of treated fabrics

Sample no.	Fabrics Specifications				
	Fabric structure	Fabric width (cm)	Yarns count (Ne)	Density (ends*picks /inch)	Fabric weight (g/m ²)
S ₁	Plain 1/1	160	30 * 30	76 * 68	123
S ₂	Plain 1/1	120	14 * 14	68 * 56	209

Table 2. Components of Resin finish solution

Ingredients	Concentration (g/l)	Function	Remarks
Durix fix (DMDHEU)	40-60-80-100	cross-linking agent	the main material (variable)
Magnesium chloride	8-12-16-20	Catalyst agent	depends on the resin conc.
Durix soft 1000 or per soft Al	20	Fatty acid (non-ionic)	
Sidco soft PE	20	Poly ethylene dispersion	
Afcosil AKM	20	Amino poly siloxane	
Wettalol (oil)	3	Wetting agent	
Asmawight 2PL	3	Optical brightener for cotton	

3.3. Testing Procedure

All the samples (non-mercerized, mercerized only, cross-linked only and mercerized then cross-linked) were subjected to these tests:

3.3.1. Dimensional Properties

Fabric dimensional properties were measured for raw and bleached plain weave such as fabric width shrinkage, warp and weft density, and fabric weight per unit area according to AATCC Method 135, standard testing method of ISO 7211-2:1984, and ASTM D3776-07 standard test methods respectively.

3.3.2. Mechanical Properties

These studies were undertaken with a view to clarify the impact of mercerization on the mechanical properties of cotton fabrics and to what extent these properties are affected by the force applied on the fabric during mercerization. These measurements are: 1) Breaking load (gf) and breaking elongation, (%) using Tensolab cloth strength tester, according to Egyptian standard specification 1506-1/2007, 2) Crease recovery angle (CRA), (degree) according to AATCC test method 66-1990, 3) Fabric tear strength, (gf) using Elmendorf tear tester, according to standard specification (ASTM D1424), 4) Fabric abrasion resistance, (cycles) using ring wear tester according to ASTM D3884 - 09.

4. RESULTS AND DISCUSSION

4.1. Effect of mercerization process on the fabric dimensional properties

Results of figure (1-a) show that the fabric width shrinkage increases by increasing the mercerizing time from 10 to 30 sec, by a percent ranges from -6.7 to -15 %. This can be interpreted by the contraction of the cellulose molecules of the weft yarns due to the higher swelling at higher mercerizing times. According to the statistical analysis of the

obtained results as shown in table (3), fabric width shrinkage is affected significantly by changing the mercerizing time within the studied range. Because of the fabric width shrinkage, the percentage increase of warp density increases by increasing the mercerizing time, by a percent range from 2.9 to 5.9 %. Because mercerization process is carried out under tension in the warp direction, the weft density decreases. Moreover, the percentage decrease of weft density increases by increasing the mercerizing time, by percent ranges from -3.6 to -5.4 %, which is also a significant change according to table (3). The obtained fabric cover factor after mercerization improved due to higher degree of uniformity and orientation of the cellulose molecules, which reflects on the yarns regularity and hairiness. As a result the yarns become much closer and the fabric becomes more cover. Mercerization as a chemical wet processing dissolves some of the cellulose molecules, thus decreases the fabric weight per unit area, The percentage decrease of fabric weight per unit area increases by increasing the mercerizing time by a percent ranges from -0.5 to -1.4 %. The decrease here is not significant as shown in table (3). These values also rely within the accepted ranges for the consumers.

- The yarns and fabric cover factor for plain weave 1/1 can be calculated as follow:

Figure (1-b) shows the relationship between the mercerizing time and the fabric dimensional properties of plain weave bleached fabric, Results reveal that the fabric width shrinkage increases by increasing the mercerizing time from 10 to 30 sec, by percent ranges from -6.9 to -19.4 %. It is shown that the shrinkage percent of fabric width of the bleached fabric is higher than the raw fabric; this can be interpreted in terms of the morphological changes in the physical structure of the cotton fibres after bleaching process, which increases the ability of cotton fibres to absorb solutions. Statistical analysis of these results ensures that the fabric width shrinkage is increased significantly by increasing the mercerizing time, see table

(3). The percentage increase of warp density increases significantly by increasing the mercerizing time, by a percent range from 3.9 to 10.5 %. The percentage decrease of weft density increases by increasing the mercerizing time, by a percent range from -1.5 to -4.4 %, but this decrease is not significant. The percentage decrease of fabric weight per unit area increases significantly by increasing the mercerizing time by a percent range from -1.6 to -4.5%.

4.2. Effect of mercerization and cross-linking agents on the fabric breaking load

Figure (2) shows the effect of mercerization and cross-linking treatments on the fabric breaking load of plain weave in the warp direction. When these fabrics were subjected to mercerization with NaOH concentration 260 g/l, the tensile strength increases by a percent ranges from 5.4 to 13.62 %. It is seen that the breaking load increases significantly after mercerization regardless of the mercerizing time. This could be associated with the higher degree of molecular orientation of cotton during mercerization under tension. These fabrics were then subjected to cross-linking with DMDHEU concentration 80 g/l and the curing time was 4

minutes. Obviously, the breaking load decreases after resin finishing by a percent of 22.65 and 24.6 %, at mercerizing time 10 and 30 sec, respectively; as a result the mercerized then cross-linked samples lost a percent of its strength (compared to the non-mercerized, non-cross-linked samples) ranges from 7.59 to 18.68 % at mercerizing time 10 and 30 sec, respectively. The same action was found in the weft direction, i.e. mercerization improved the breaking load by a percent ranges from 7.9 to 12.9 % as shown in figure (2). Nevertheless, the breaking load in the weft direction increases by increasing the mercerizing time, a point which is opposite to the warp direction. It seems very likely that increasing the speed of the machine during mercerization causes a sort of contraction and shrinkage of the weft yarns in cotton fabric thereby detracting from their full benefit for the orientation of the cotton molecules. When these fabrics were subjected to cross-linking, the breaking load decreases by a percent ranges from 23.33 to 21.73 % at mercerizing times 10 and 20 sec, respectively. As a result the mercerized then cross-linked samples lost a percent of its strength ranges from 11.51 to 17.27 % compared to the non-mercerized samples.

Before Mercerization	After Mercerization
$K_1 = 76 / \sqrt{30} = 13.88$	$K_1 = 83 / \sqrt{30} = 15.15$
$K_2 = 68 / \sqrt{30} = 12.415$	$K_2 = 66 / \sqrt{30} = 12.05$
$K_c = K_1 + K_2 - \frac{K_1 * K_2}{28} = 20.14$	$K_c = K_1 + K_2 - \frac{K_1 * K_2}{28} = 20.68$

Table 3. ANOVA for effect of mercerization parameters on fabric dimensional properties

Dependent variable	Sig.* for raw fabric	Sig.* for bleached fabric
Fabric width	.000	.869
Warp density	.001	.001
Weft density	.000	.170
Fabric weight / m ²	.006	.002

* Significance at confidence interval of 95 %

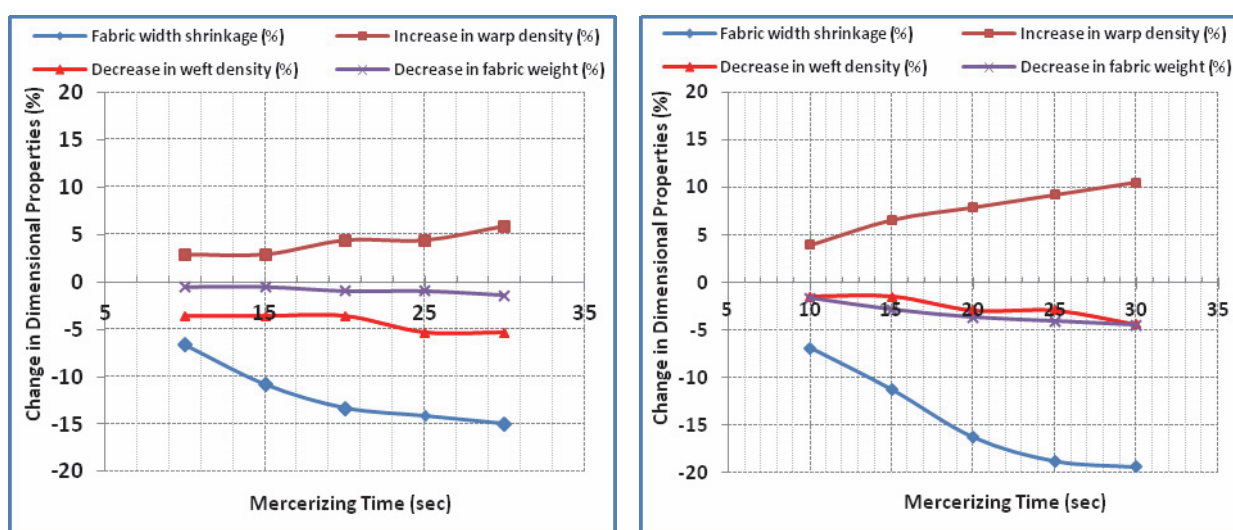


Fig 1. Effect of mercerizing time on the fabric dimensional properties a) Plain weave raw fabric, b) Plain weave bleached fabric

4.3. Effect of mercerization and cross-linking agents on the fabric breaking elongation

Figure (3) shows the effect of the mercerizing time on fabric breaking elongation in the warp direction. Obviously, the percentage loss of breaking elongation in the warp direction decreases by increasing the mercerizing time from 10 to 30 sec, by about -14.9 % at mercerizing time 10 sec and -9.6 % at 30 sec. This may be interpreted in terms of changes in the micro structural features of the cotton cellulose and the influence of the caustic soda (NaOH) under different tensions as pointed out in case of the breaking load. When these fabrics then subjected to cross-linking, the percentage loss of breaking elongation decreases to about -13.3 % at mercerizing time 10 sec and -8.51 % at 30 sec, compared to the non-mercerized, non-cross-linked samples. At the outset of this paragraph it is emphasized that fabric width before mercerization acquires a value of 160 cm; this value decreases to 145 cm after mercerization. Figure (3) shows the relation between the breaking elongation of mercerized cotton fabric in the weft direction and the mercerizing time. As is evident the percentage increase of breaking elongation in the weft direction increases by increasing the mercerizing time within the range studied, i.e. from 10 to 30 sec, reasons cited above in case of the breaking load may be taken into consideration to account for this. On the other hand the breaking elongation values after mercerization are higher than the values before mercerization. The pre-mentioned shrinkage in the fabric width after mercerization together with changes in fine physical structure of cotton cellulose under the effect of NaOH which acquires mercerization strength would account for the outstanding increase in breaking elongation after mercerization regardless of the speed of the mercerizing machine. The breaking elongation increased after mercerization by a percent ranges from 28.5 to 41.9 %. After cross-linking, the breaking elongation decreased by a percent ranges from 15.7 to 19.6% at mercerizing times 30 and 10 sec, respectively.

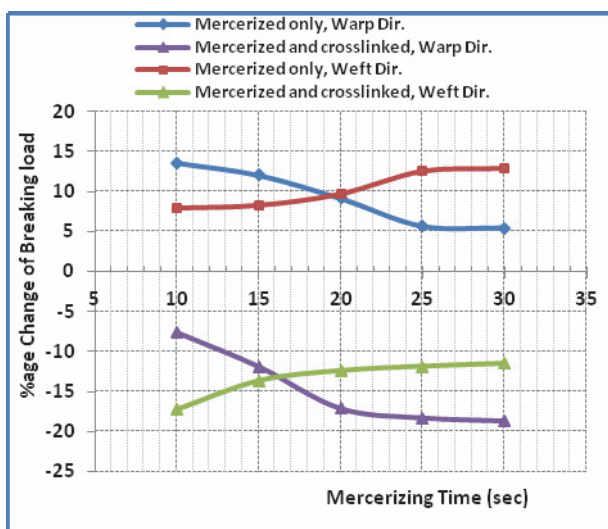


Fig 3. Effect of mercerization and cross-linking treatments on breaking elongation.

4.4. Effect of mercerization and cross-linking agents on the fabric breaking elongation

Figure (3) shows the effect of the mercerizing time on fabric breaking elongation in the warp direction. Obviously, the percentage loss of breaking elongation in the warp direction decreases by increasing the mercerizing time from 10 to 30 sec, by about -14.9 % at mercerizing time 10 sec and -9.6 % at 30 sec. This may be interpreted in terms of changes in the micro structural features of the cotton cellulose and the influence of the caustic soda (NaOH) under different tensions as pointed out in case of the breaking load. When these fabrics then subjected to cross-linking, the percentage loss of breaking elongation decreases to about -13.3 % at mercerizing time 10 sec and -8.51 % at 30 sec, compared to the non-mercerized, non-cross-linked samples. At the outset of this paragraph it is emphasized that fabric width before mercerization acquires a value of 160 cm; this value decreases to 145 cm after mercerization. Figure (3) shows the relation between the breaking elongation of mercerized cotton fabric in the weft direction and the mercerizing time. As is evident the percentage increase of breaking elongation in the weft direction increases by increasing the mercerizing time within the range studied, i.e. from 10 to 30 sec, reasons cited above in case of the breaking load may be taken into consideration to account for this. On the other hand the breaking elongation values after mercerization are higher than the values before mercerization. The pre-mentioned shrinkage in the fabric width after mercerization together with changes in fine physical structure of cotton cellulose under the effect of NaOH which acquires mercerization strength would account for the outstanding increase in breaking elongation after mercerization regardless of the speed of the mercerizing machine. The breaking elongation increased after mercerization by a percent ranges from 28.5 to 41.9 %. After cross-linking, the breaking elongation decreased by a percent ranges from 15.7 to 19.6% at mercerizing times 30 and 10 sec, respectively.

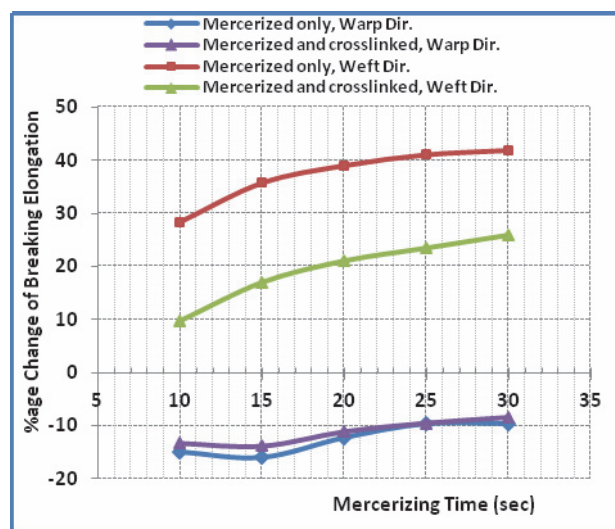


Fig 2. Effect of mercerization and cross-linking treatments on breaking elongation.

4.5. Effect of resin finish on fabric tear of mercerized and non-mercerized cotton fabrics

Figure (4) illustrates the effect of resin concentration on the fabric tear of mercerized and non-mercerized plain weave in the warp direction, the percentage loss in fabric tear increases by increasing the resin concentration. However, the tear values for mercerized samples are higher than those of the non-mercerized samples. The best resin

concentration is 40 g/l. The best mercerizing time is 10 sec. Similarly figure (5) illustrates the effect of resin concentration on the fabric tear of mercerized and non-mercerized plain weave in the weft direction. The fabric tear decreases by increasing the resin concentration, but the tear values for mercerized samples are higher than those of the non-mercerized samples. The best resin concentration is 40 g/l. The best mercerizing time is 20 sec.

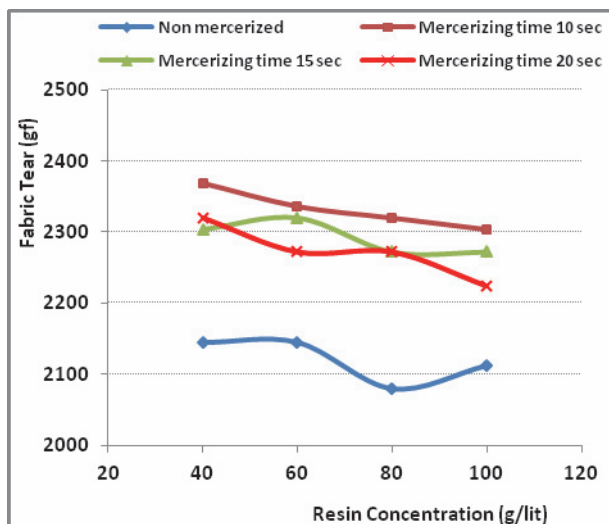


Fig 5. Effect of resin concentration of mercerized and non-mercerized fabrics on fabric tear in weft direction.

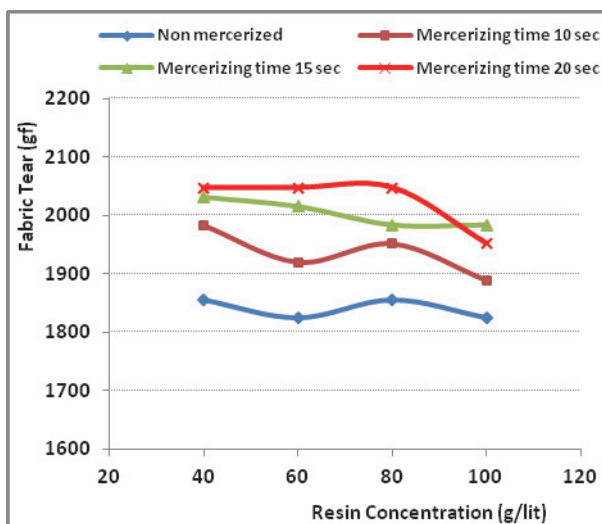


Fig 4. Effect of resin concentration of mercerized and non-mercerized cotton fabrics on fabric tear in the warp direction.

4.6. Effect of resin concentration on fabric abrasion resistance

Loss in fabric abrasion resistance observed upon using higher DMDHEU concentrations is a manifestation of excessive cross-linking occurring at such concentrations. It is understandable that the hydroxyl groups of the cellulose chain in the cotton fabric are immobile. Reactions of these groups with DMDHEU via cross-linking would, therefore, relies on availability of DMDHEU molecules in the vicinity of the cellulose hydroxyls. Logically, greater number of DMDHEU molecules would be available in the proximity of the hydroxyl groups of cellulose at higher DMDHEU concentrations and, as a result, excessive cross-linking takes place. Excessive cross-linking and factors associated with such as restriction of segmental movement would certainly cause fiber-fabric rigidity. Once this is the case fabric abrasion resistance decreases; in agreement with the results of other strength properties previously reported within the framework of current endeavor. On conclusion of the above, figure (6) shows that the fabric abrasion resistance increased as increasing the resin concentration from 40 to 80 g/l then decreased at 100 g/l. The best resin concentration is 80 g/l, and the best mercerizing time is 20 sec.

4.7. Effect of resin concentration on crease recovery angles (CRA)

Figure (7) shows the effect mercerization and resin finish processes on the total percentage improvement of CRA in the warp and weft directions. Results of the pre-mentioned figure conclude that: a) The percentage increase in CRA increases by increasing the resin concentration from (40 to 80 g/l) then decreases thereafter; b) The best resin concentration is 80 g/l and; c) The best mercerizing time is 20 sec. The enhancement in CRA by increasing the resin concentration up to 80 g/l is due to abundance of cross-linking. On the other hand, the decrease in CRA at resin concentrations higher than 80 g/l suggests relatively lower rate of resin diffusion in the interior of the cotton structure because of greater viscosity of the treating solution at higher resin concentrations. It is also likely that the interaction between the resin and the cotton at higher resin concentrations proceeds initially very fast and results in chemically modified cotton (cross-linked cotton) with relatively lower susceptibility towards reaction with the resin during the later stages of the finishing treatment thereby breaking about lower CRA.

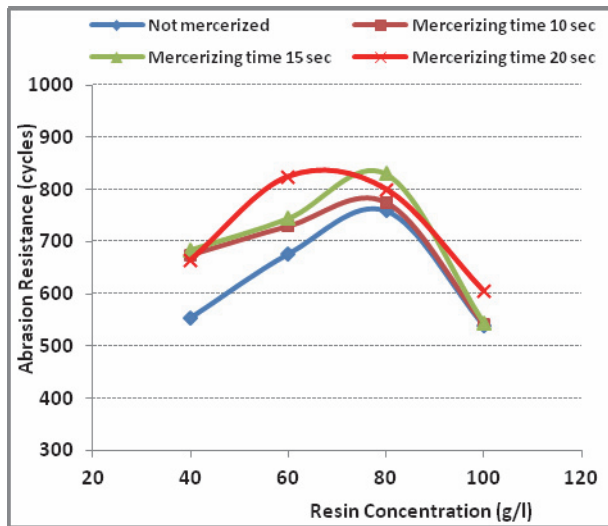


Fig 7. Effect of resin concentration of mercerized and non-mercerized cotton fabrics on total CRA (warp + weft)

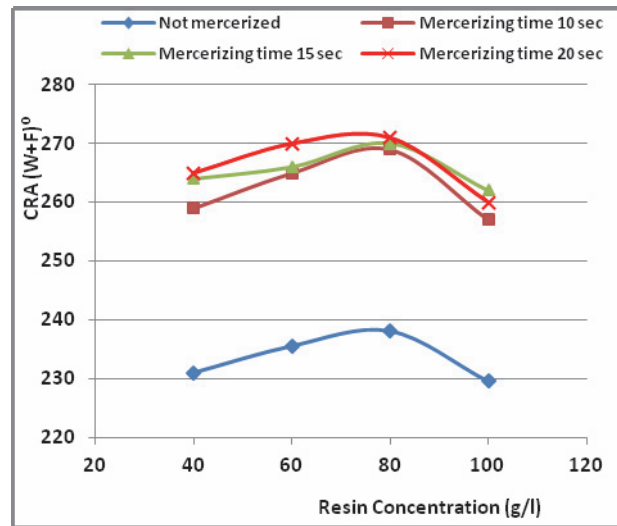


Fig 6. Effect of resin concentration on fabric abrasion resistance of mercerized and non-mercerized cotton fabrics

Table 4. Effect of mercerization and cross-linking on fabric mechanical properties

Significance	Independent variables	Dependent property
.000	Mercerizing time, NaOH conc.	Breaking load
.000	Mercerizing time, resin conc., cure time	Fabric tear
.001	Mercerizing time, resin conc., cure time	Abrasion resistance
.000	Resin conc., cure time, mercerizing time	Crease recovery

5. CONCLUSION

The philosophy behind the candidate own work to enhance the strength properties of 100 % cotton fabrics in order to compensate for strength losses when these fabrics were subjected to easy care finishing treatment. Emphasis was placed on mercerization as a pretreatment process to achieve improvement in fabric strength against strength losses caused by the after-treatment process, namely cross-linking using DMDHEU. Both of the pretreatment and the after-treatment were pursued under a variety of conditions using cotton fabrics having different structures. Fabrics before and after mercerization as well as those before and after cross-linking were evaluated for breaking load, breaking elongation, crease recovery angle, tear strength and abrasion resistance. These properties were monitored in both warp and weft directions of the fabrics. There are optimum conditions of mercerization for every fabric structure and weight per unit area; i.e.: a) for light fabrics NaOH concentration was 260 g/l and the mercerizing time was 20 sec, while for heavy fabrics NaOH concentration ranges from 280 to 300 g/l, depending on the fabric structure; b) the best DMDHEU concentration was 80 g/l in the presence of magnesium chloride ($MgCl_2 \cdot 6H_2O$) as a

catalyst at 16 g/l, the curing temperature was 160°C and the curing time was 4 minutes. Mercerization increased breaking load, fabric tear, and abrasion resistance by a percent ranges from 10 to 20 %, 8 to 21 %, and 7.1 to 17.3 % respectively. On the contrary, resin finish decreased the breaking load, fabric tear, and abrasion resistance by a percent 15 to 30 %, 8 to 38 %, and 22.4 to 43.9 % respectively. Wrinkle resistance finish increased the CRA by a percent ranges from 7 to 27 %. Thus, severe strength loss can be limited by effective mercerization before cross-linking.

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