Production of High Tenacity Cotton Yarns by Mercerization

K. P. R. PILLAY
South India Textile Research Association, Coimbatore 14

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The influence of mercerization under different conditions on the tensile properties of cotton has been investigated and the conditions for imparting maximum strength have been identified. In general, mercerization in two stages rather than one (30% followed by 10% caustic soda solution) has been found to give the most satisfactory results. The extent of improvement in bundle strength for different cottons ranged from 49 to 104% for fibres and from 15 to 43% for yarns. The breaking elongation was, however, reduced by 24-63% for fibres and by 46-63% for yarns. Fibres and yarns which were suitably mercerized and dried after crosslinking possessed a strength almost equal to that of the unresinated samples. The development of a machine for continuous mercerization of single yarn on the basis of these observations has been attempted.

Alkali swelling treatments cause changes in cotton fibre structure and this affects their structural coherence and internal tensions. Of special interest in this connection is the effect of swelling, stretching, deswelling and drying techniques on the tensile properties of mercerized fibres and yarns.

The aims of this investigation were: (i) to obtain as favourable a method of mercerizing as possible for the production of fibres and yarns of high tenacity, and (ii) to use the knowledge so gained for the development of a single end mercerizing process.

Experimental Procedure

Materials

For preliminary studies, Moroccan Supima cotton and 2/86s yarn spun from it were used. To study the response of different cottons to mercerization, eleven more cottons and 2/30s yarn spun from them were used. Table I gives the ranges of fibre properties of these samples.

Methods

The fibres and yarns were treated in alkali for 15 min in the form of well parallelized bundles, fixed between tabs. Swelling and stretching of samples to different degrees were done on special frames fabricated for the purpose. The frames could be immersed vertically in a trough containing alkali for swelling and stretching to the desired extent.

Preparation of sodium zinctate solution — It has been reported that if zinc oxide is added to sodium hydroxide, the swelling power towards cotton is enhanced considerably. Sodium zinctate was prepared by shaking anhydrous zinc oxide with sodium hydroxide solution, heating the suspension to boiling and allowing it to cool. A molar ratio of zinc oxide to sodium hydroxide of 0.1 was used.

Washing and drying of treated samples — The fibre and yarn samples mounted on the respective frames with appropriate stretch were washed first with hot water (80-90°C) and then with tap water thoroughly to remove the alkali. They were then kept in acidified (1% sulphuric acid) water for 5 min, then washed in tap water and finally in distilled water till the wash liquor was neutral to litmus.

Four different temperatures, viz. (i) standard laboratory conditions (27°C and 65% RH) for 3 days, (ii) 70°C for 3 hr, (iii) 100°C for 2 hr, and (iv) 140°C for 1 hr were used for drying the samples. The samples were then conditioned at standard laboratory conditions before testing.

Details of treatments — The following techniques were used for the mercerization of fibres and yarn samples from Moroccan Supima cotton.

P. 1 : Control sample—untreated
P. 2 : Swelling without tension in 25% caustic soda at 27°C (slack mercerization)
P. 3 : Swelling without tension in 25% caustic soda at 27°C followed by stretching to original dimensions in the swelling medium, maintaining the dimensions during rinsing.
P. 4 : Swelling without tension in 25% caustic soda at 27°C followed by stretching to original dimensions during rinsing.
P. 5 : Using constant dimensions during swelling in 25% caustic soda at 27°C and rinsing treatments.
P. 6 : Swelling in 25% caustic soda at 27°C, stretch-
chung to original length in the swelling medium and releasing the tension during rinsing and drying.

P. 7: Swelling without tension in 25% caustic soda at 27°C followed by stretching to 5% greater length than original in the swelling medium, maintaining the dimensions during rinsing treatments.

P. 8: Swelling without tension in 30% caustic soda solution, transferring the yarn to 10% solution and then stretching to 5% greater than original length, maintaining the dimensions during rinsing.

P. 9: Swelling without tension in 25% caustic soda solution at 60°C first and then transferring the yarn to a solution at 20°C and stretching to 5% greater length, maintaining the dimensions during rinsing and drying.

P. 10: Swelling without tension in a solution of sodium zincate (molar ratio of zinc oxide to sodium hydroxide, 0.1) solution at 27°C stretching to 5% greater than original length, maintaining these dimensions during rinsing and drying.

P. 11: Swelling in 12.5% caustic soda solution at 27°C, stretching to 5% greater than original length in the swelling medium, maintaining the dimensions during rinsing.

P. 12: Drying the samples at 90°C for 1 hr and then treating in the same way as in P. 7.

Tensile tests on fibres and yarns — The Instron tensile tester was used to study the tensile properties of fibres and yarns. Gauge lengths of 1 cm for fibre bundles and 20 cm for yarn bundles were used uniformly. The specimens were extended to 3% elongation at the rate of 2 cm/min for fibres and 20 cm/min for yarn samples. They were allowed to relax for 2 min. The jaw motion was then reversed and the jaw returned to the starting point at the same speed. They were again allowed to relax for 2 min and then extended until the specimens broke. The mean breaking load, breaking elongation and initial modulus of the samples were measured from the load-elongation curves obtained on the chart of the Instron tester. Elastic parameters like immediate elastic recovery (IER), delayed recovery (DR) and permanent set (PS) were calculated from cyclic extension curve (Fig. 1). Chart speeds of 200 cm/min for yarns and 10 cm/min for fibres were used. The methods of calculation of these parameters have already been reported earlier1.

Resin treatment — The fibres and yarns in the untreated and mercerized (P. 7) conditions were treated with a solution containing 6% DMEU and 1.5% catalyst for 30 min. The solution was applied to a wet pick up of 70%. The samples were dried in an oven at 85°C for 1 hr and cured at 150°C for 4 min. They were afterwards washed with sodium carbonate solution, dried and conditioned.

Response of cottons to mercerization — The response of different cottons varying in fibre properties and 2/30s yarn spun from them to a selected mercerization treatment (P. 8) was assessed to study the influence of fibre structure on the changes in tensile properties after mercerization.

Results and Discussion

Breaking tenacity of fibres and yarns mercerized under different conditions — The breaking tenacities of fibres and yarns mercerized under different conditions are plotted in Fig. 2. There is a small reduction (5%) in the tenacity when fibre bundles are slack mercerized; the extent of reduction is, however, higher (23%) for yarns.

Mercerization is known to strengthen the weak links in fibres and improve the orientation slightly due to deconvolution. Consequently, a small improvement in fibre strength may be expected in mercerized samples at longer gauge lengths. The opposite trend noticed in this study is probably due to the reduction in the lateral cohesion of the structural elements and the greater degree of amorphous disorientation that is likely to take place as a result of alkali treatment. When cotton fibres and yarns are mercerized and stretched to their normal dimensions (P. 4), the breaking strength improves significantly (34 and 16% respectively) for fibres and yarns.

Stretching of the fibre bundles during rinsing (P. 4)
does not appear to be as effective as stretching them in the swelling medium. Perhaps the increase in the degree of orientation attained by the fibres is higher in the latter case. Treatment of the samples at constant dimensions during mercerizing, rinsing and drying (P. 5) gives lower breaking strength than mercerizing and stretching to normal dimensions in the swelling medium (P. 3) does not appear to be as effective as stretching them in the swelling medium. Perhaps the increase in the degree of orientation attained by the fibres is higher in the latter case. Treatment of the samples at constant dimensions during mercerizing, rinsing and drying (P. 5) gives lower breaking strength than mercerizing and stretching to normal dimensions in the swelling medium (P. 3).

Releasing the tension on fibres and yarns during rinsing and drying (P. 6) also gives lower strength than (P. 3) when the fibres are kept under tension during rinsing and drying. Stretching of the fibres and yarns to 5% greater than the original length in the swelling medium (P. 7) improves their strength further (about 48 and 17%, respectively). The increase in tenacity on mercerization and stretching is an indication of creation of less disorder or more cohesion with the treatment.

The maximum increases in strength on mercerization (32 and 67%, respectively) are obtained when fibres and yarns are given a two-stage mercerization treatment (P. 8). In the first stage, cotton is treated with a high concentration (30%) alkali solution and in the second stage with a solution of lower than usual (10%) concentration. Almost similar increase in strength is obtained when sodium zincate solution (molar ratio 0.1) is used as the swelling agent (P. 10). The use of two temperatures (60°C and 20°C) for mercerizing (P. 9) is also equally effective in giving good penetration of alkali and higher breaking strength after mercerizing. Swelling in alkali solution of lower concentration (12.5%) (P. 11) or drying the sample before swelling (P. 12) is not very effective in improving cotton fibre strength.

The importance of the evidence deduced so far is that the complete internal structure of cotton hair is affected by sodium hydroxide. The hair in its swollen state is deformable and the fibrils are now set into a new position. The tension applied in the swollen state is likely to bring about morphological and structural changes. But unless this tension is maintained till the alkali is removed, the new state is likely to be transitory. The speed and method of removal of alkali are thus critical for the process. Thus, increase in orientation parameter should mean increase in strength; this can be seen from a consideration of the morphology of cotton. Reducing the spiral angle and thus indirectly the X-ray angle results in better alignment of fibrils along the fibre axis with resultant decrease in breaks.

Two-stage mercerization appears to be more effective, presumably because 30% alkali being a relatively low swelling solution can achieve much better penetration and wetting out of the fibre and yarn bundles. Subsequent treatment in 10% caustic brings the overall concentration close to that of maximum swelling. The result would be improved uniformity of treatment throughout the yarn.

It is also interesting to note that although the response of fibres and yarns is similar for different treatments, the extent of change in the two cases is different. Probably in the case of fibres, the swelling action is more rapid and complete than in the case of yarns. The improvement in orientation is much better in fibre bundles than in yarn bundles. Moreover, the geometry of yarn has a significant influence on yarn strength. It is well known that only a part of the aggregate fibre strength is realized in yarn strength.

Breaking elongation — The percentage elongation of fibres and yarns (Fig. 2) increases on swelling without tension, but the increase is much higher (600%) for yarns than for fibres (20%). The obvious reason for this is the great cross-sectional swelling and longitudinal shrinkage undergone by the fibres on mercerization and the consequent loss in cohesion between molecules. When fibres and yarns are mercerized, stretched, washed and dried, the additional points of cohesion formed under these conditions stabilize the structure of the material and consequently increase in tenacity and reduction in breaking elongation are noticed. Thus, in mercerized fibres, breaking elongation varies between 13.3 (for slack mercerized sample) and 5.7% (for mercerized and stretched samples), the corresponding values for yarn bundles being 31.9 and 2.7% respectively. Fibre bundle elongations are higher than the corresponding yarn elongations, except in the case of slack mercerized yarns. Probably because of the small gauge lengths used for testing fibres, small slippages between jaws influence the final elongation values significantly.

Initial modulus — The initial modulus (IM) varies between 2.6 and 6.0 g/tex% elongation for yarns, and 1.07 and 5.7 g/tex% elongation for untreated yarns and fibres respectively (Fig. 3). Slack mercerization reduces IM by about 75% of the untreated state. The high degree of intra-crystalline swelling undergone by the fibres on mercerization breaks down the stabilizing inter-chain secondary bonds which are most important for the production of stiffness and rigidity in fibres, resulting in decrease in elastic modulus. Swelling and stretching of the samples to original dimensions or releasing the tension on stretching bring about partial increase in IM. The highest values of IM are generally shown by samples mercerized and stretched to 5% greater than original length.

Effect of mercerization on recovery properties:
Immediate elastic recovery (IER) — The immediate elastic recovery (Fig. 3) is maximum for untreated
fibres and yarns (23.0 and 28.8% of the conditioning elongation 3.0%). The IER produced on mercerizing fibres and yarns is obviously due to the intra-crystalline nature of the swelling treatment and the consequential high degree of swelling. Stretching after mercerization increases the IER and the improvement is maximum for samples stretched to 5% greater than their original length. The higher elastic recovery of mercerized and stretched samples over the slack mercerized samples can be attributed to the greater orientation in fibres. There are some differences in the response of fibres and yarns to mercerization and stretching. Probably in yarn, the geometry of the yarn structure also influences the extent of fibre modification on swelling and stretching.

Delayed recovery — In untreated fibres and yarns, delayed recovery (DR) forms 57.8 and 45.5% respectively of the conditioning elongation (3%). The change in DR with mercerization techniques shows similar trends in fibres and yarns. In general, mercerized and stretched fibres and yarns show greater DR than mercerized samples, while mercerized samples show lower DR than untreated samples.

Permanent set — The permanent set (PS) of untreated fibres and yarns is 19.2 and 25.7% respectively of the conditioning elongation. The PS increases slightly on mercerization and shows a tendency to fall with higher degrees of stretch. No definite tendency is, however, noticed in the changes of this property under different conditions of mercerization and stretch.

Effect of drying temperature on tensile properties — There is a small increase in breaking strength when fibres are dried at 70°C. The small increase in tensile strength as a result of drying at 70°C may be due to the increase in the amount of ordered fraction in the fibres. There is evidence that the accessibility and amount of disordered fraction of cotton as measured by the sorption of iodine and barium hydroxide are decreased by drying at high temperatures. Further increase in temperature does not show any significant change in tensile strength. The other tensile properties do not show any consistent trend with variation in drying temperature. The changes are small and probably non-significant.

Response of different cottons to mercerization — Having observed that the breaking strength of cotton fibres can be improved substantially by alkali swelling, stretching and drying under appropriate conditions, it was thought desirable to study the response of different cottons varying in fibre properties to mercerization. The two-stage mercerization process (P. 8) was used for this purpose.

Table 2 shows that there are wide differences in the response of cottons and yarns spun from them to mercerization. In the case of fibres, the increase in strength ranges from 14.0 for Supima cotton to 104.6% for MCU 1. The corresponding reductions in breaking elongation of these cottons are 32.8 and 66.7% respectively. Similarly, the yarn strength improves from 14.5 to 43.5% and breaking elongation reduces by 46.2 to 62% as a result of mercerizing, stretching and drying. Supima and MCU 1 cottons give the lowest and the highest increase in breaking strength (14.0 and 104.6%). The reduction in breaking elongation is minimum for Sujata (22.1%) and maximum for MCU 1 (66.7%).

Effect of fibre properties on the changes in strength and elongation on mercerization — It has been shown that, in general, cottons having higher orientation show smaller increases in breaking strength and higher increases in breaking elongation on mercerization. But the results of this study show no such clear trend. Probably the high stretch (5% over the normal length) used in this study might have been too high for some cottons to upset the normal relationship. However, Fig. 4 shows that the change in breaking strength of cottons is inversely related to their fibre quality index. Similarly, the changes in breaking strength and elongation of cottons and yarns are correlated with the strength and elongation in the untreated condition (Figs. 5 and 6). Stronger

<table>
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<tr>
<th>Cotton</th>
<th>Untreated fibres</th>
<th>Mercerized fibres</th>
<th>Untreated 2/30s yarn</th>
<th>Mercerized 2/30s yarn</th>
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<td>Breaking strength</td>
<td>Elongation %</td>
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Table 2 — RESPONSE OF DIFFERENT COTTONS TO MERCERIZATION
cottons and yarns show lower improvement in strength and larger increase in breaking elongation on mercerization. Figs. 5 and 6 further show that the different types of cottons segregate themselves into different groups, showing that the type of cotton also influences the extent of change on mercerization.

**Effect of resin treatment on mercerized fibres and yarns** — The application of resin for easy-care properties reduces the breaking strength and abrasion resistance of fibres and yarns to an undesirable extent. It has been further shown that this strength loss can be reduced significantly or even brought to zero by suitable swelling, stretching and drying treatments to make the fibre structure more uniform along the length and cross-section of the fibres. This has been verified by treating fibres and yarns in the untreated and mercerized conditions with DMEU resin.

It is clear from Fig. 7 that there is considerable reduction (about 45%) in the breaking strength of fibres and yarns on resin treatment. The breaking elongation is also lowered appreciably (12%). However, when the fibres and yarns are mercerized and then resin treated, the fall in breaking strength is practically eliminated and the reduction in breaking elongation is lowered.

**Preliminary Trials on Single End Mercerization**

Commercial mercerization of cotton yarns is generally carried out in hank form. Despite the higher production achieved (50–60 kg/hr), hank mercerization has many limitations like non-uniformity of alkali penetration, incidence of knots due to breakages in hank winding, etc. These shortcomings could be overcome by single end mercerization (SEM). Using the two-bath process (P. 8), an attempt was made to fabricate an experimental set-up (Fig. 8) for SEM.

The yarn to be mercerized is unwound from a cone and guided round a driver roller rotating in a small trough (1) of alkali containing a wetting out agent. The yarn wet with alkali is passed round a wheel immersed in a tank (2) of caustic soda solution of 30% concentration where good penetration of alkali and an intra-crystalline swelling take place.
It is important that the thread be dried under tension to stabilize the dimensional change that has been brought about by the process. Table 3 gives the results obtained on a few samples of yarn mercerized by this process by giving 2–3% stretch. It is seen that SEM yarns give 19–24% increase in strength, whereas commercially mercerized yarns give 12–17% increase. In the SEM process, the strength improvement can be controlled, so that loss in elongation is maintained at an acceptable level.

A rough calculation of the economics of this process compared with that of hank mercerizing shows that SEM yarn will be costlier than hank mercerized yarn by about Re 1 per kg. But the former is likely to yield better quality of yarn, which will have many industrial applications.

Summary and Conclusions

1. Cotton fibre strength can be improved considerably (49–104%) by a two-stage mercerization process, accompanied by stretching, washing and drying under tension. The corresponding improvement in yarn strength is much lower (15–43%).

2. The breaking elongation of fibres and yarns is significantly reduced (24–63%) by the above treatment.

3. Generally, cottons having higher fibre quality index give lower increases in strength and higher increases in breaking elongation on mercerization.

4. The possibility of using the knowledge gained by this research for developing a SEM process is indicated.

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