Scanning Electron Microscopic Study of Wet Processing of Cotton Fabrics

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A poplin fabric was desized and subsequently given different treatments using (i) solvent, (ii) caustic in a kier under pressure, and (iii) sodium carbonate at atmospheric pressure in the laboratory. The desized sample and the three scoured samples were bleached by mild and severe processes using sodium hypochlorite. All the bleached samples were subsequently mercerized conventionally in a mill. A scanning electron microscopic study of the above fabrics reveals that increasing severity of chemical processing conditions increases fibre damage. The fibre damage appears to be maximum with the fabric processed under the following sequence: desized-kier boiled-severely bleached-mercerized. The damage was minimum in the case of a desized-mild bleached-mercerized sequence. Surprisingly, the fibre damage is always restricted to the warp crown fibres, while the rest of the warp and all the weft fibres appear undamaged under the severest conditions employed. The physical properties of the fabric were measured at different stages. Tear strength and flex abrasion cycles decrease considerably with the processing steps, but the tensile strength does not change significantly. A comparison of the deterioration in properties suggests that solvent scouring and kier boiling operations are more drastic than an open soda boil, which is obviously more severe than desizing alone.

Earlier studies had shown that severe conditions of caustic scouring affect the tear strength, flex abrasion resistance and toughness of a fabric. It can be safely assumed that these changes in physical properties are due to (i) degradation of fibre structure by chemical action, (ii) mechanical damage to the fibre structure before and during the processing treatments, and (iii) a combination of chemical and mechanical damage. The scanning electron microscope (SEM) offers an easy means of assessing fibre damage, when the damage occurs in the form of changes in the surface of individual fibres. With the SEM, fabric samples are inserted into the instrument and using the wide range of magnifications available one is able to view a large area of the fabric at low magnification and then examine selected areas at increasing magnification until surface detail of a single fibre can be seen.

A systematic study has been undertaken using the SEM to determine the extent of fibre damage resulting from the effect of various wet processing treatments. The results of this SEM investigation and the physical properties of the fabric are presented in this communication.

The three most common wet processing treatments are scouring, bleaching and mercerization. In this study, we have examined a fabric without any scouring treatment, as well as fabrics with all combinations of the above mentioned processes in varying degrees of severity. Three scouring treatments were used: (i) solvent, (ii) caustic pressure boil, and (iii) open soda boil. Two bleaching treatments, mild and severe, were also carried out. The conventional mercerization treatment was given.

Using the above treatments, we were able to investigate the extent of fibre damage to varying degree ranging from the mildest treatment, i.e. simply a desized fabric, to a very severe treatment, viz. a desized, pressure caustic boiled, severely bleached, mercerized fabric.

Experimental Procedure

A Cambridge Stereoscan S4-10 scanning electron microscope was used in conjunction with an AEI vacuum coating unit. The specimens to be examined in the SEM must be able to withstand the effect of vacuum and be able to conduct electricity. Cotton fabrics are nonconductors of electricity and, therefore, they must be made conductive by the deposition through evaporation of a very thin (20-40 μ) layer of silver or gold onto the surface of the specimen.

Before coating the fabric samples with metal, they are mounted on the SEM mounts using a double sided adhesive tape and conducting cement. The metal is deposited on the specimen surface in a vacuum coating unit by thermal evaporation.

Examination in the SEM is carried out while operating the instrument at 5kV accelerating potential.

Desizing—A loom-stage grey fabric was desized with a commercial enzyme 0.2% and salt 2 gpl at 1 : 50 ml ratio. Temperature was maintained at 50-60° for 2 hr. The fabric was washed, soaped at 85-90° for 45 min with 2 gpl soap and 5 gpl soda, rinsed with hot water, and then with cold water, dried, cut into four pieces and conditioned.

Scouring—The desized fabric was extracted with carbon tetrachloride for 6 hr and then with ethyl alcohol-benzene (40: 60) mixture for 5 hr. Subsequently, the fabric was rinsed with ethyl alcohol and

water and dried. No additives were used in solvent scouring.

Caustic pressure boil—The desized fabric (about 1.5 kg) was piled in a laboratory kier. The liquor containing 4.0% NaOH, 0.5% sodium silicate and 0.5% wetting agent (all percentages are based on the weight of fabric) was added. The lid was secured, water flushed in and the contents were boiled at 120-125°C under 40 lb pressure for 5 hr. Then the liquor was drained out, the fabric washed with hot water and then with cold water until free from alkali.

Open soda boil—The desired fabric was boiled in an open stainless steel vessel using 5% sodium carbonate and 0.5% wetting agent. The material to liquor ratio was 1 : 6 and the time of boiling, 8 hr. The fabric was then rinsed free from alkali.

Bleaching—Commercial sodium hypochlorite solution, ‘Cali Chlor’ was used after estimating the available chlorine. For mild bleaching, 1.0% chlorine on the weight of fabric was used and the reaction was continued until more than 90% of it was consumed. For severe bleaching, 2.0% chlorine on the weight of fabric was used. The liquor to material ratio was maintained at 50:1, and the bleaching solution strength was varied (0.2 gpl and 0.4 gpl for mild and severe bleaching respectively).

Mercerization—This was carried out conventionally on a chainless-padless machine in a local mill using 54°C Tw caustic boiling soda solution.

Results and Discussion

SEM Studies

Fig. 1 is a scanning electron micrograph of a desized fabric taken at low magnification. The fabric structure is clearly seen. The large depth of focus of the SEM enables the whole of the warp and weft yarns to be in focus. Fig. 2 illustrates the appearance of only the crown of a warp yarn. The fibres of the warp yarn show deposits of material over them. The material extends over the adjoining fibres as if to bond them together. It was observed that no such deposits are present on weft yarns, but that the deposits are present only on certain warp crowns. Warp crowns on both the surfaces of the fabric show such deposits. A simple iodine staining test was carried out on the fabric. Only the warp yarns stained the characteristic blue color which is given by iodine in the presence of starch. It can, therefore, be inferred that the deposits seen in Fig. 2 are most likely of starch size. However, other impurities, such as waxes, pectins, etc., could also be present.

The warp yarn also appears to be smudged or heavily pressed at the crown region (Figs. 1 and 2).

Scouring

Solvent—A warp crown of a solvent scoured fabric is shown in Fig. 3. Solvent scouring has surprisingly removed almost all the deposits seen in Fig. 3 from the warp crowns. Warp and weft appear mostly clean, although there is no great degree of damage to the surfaces of individual fibres at the warp crown. Some of the fibres at the warp crowns appear crumpled or pressed.

Open soda boil—As is observed in the case of solvent scouring, the fibres appear mostly clean and undamaged.

Pressure caustic boil—Fig. 4 shows a warp crown of a caustic boiled fabric. It is seen that any adhering material has been totally removed and some of the fibres show small fibrils raised up from their surfaces. These small fibrils can be considered to be the beginnings of fibre damage, even though total fibre breakdown has not begun to occur. All the weft yarn fibres appear clean with virtually no sign of damage on the surfaces.

Bleaching

Mild—In the case of desized mild bleached fabric, the adhering residual size or other material appears partly disintegrated, but not totally removed. Other portions of warp and all weft yarn fibres appear clean and undamaged. Mild bleaching of the scoured samples did not cause any further damage.

Severe—Fig. 5 shows that severe bleaching of a desized fabric removes practically the entire adhering material. Warp crown fibres show signs of damage and erosion to some extent. A trend of increasing severity of damage is observed in solvent scoured fabrics, soda boiled fabrics and pressure boiled fabrics which have subsequently been given a severe bleach treatment. In the solvent scoured fabric and soda boiled fabric which have had a severe bleach, damage occurs only on the primary wall (Fig. 6), but the primary wall is removed and the secondary wall is seen on fabrics which have had a pressure boil and severe bleach (Figs. 7 and 8).

Mercerization

Mercerization further increases the damage observed after bleaching. Fig. 9 shows a desized, severely bleached, mercerized fabric at a warp crown. In Fig. 10, the primary wall is seen to be partly removed and small secondary wall fibrils are protruding from the fibre surface. There is minor damage to the secondary wall.

Slight damage occurs to the primary wall of a solvent scoured severely bleached, mercerized fabric at the warp crowns (Fig. 11). Mercerization of a soda boiled, severely bleached fabric increases the damage at the warp crowns. Loose fibrils begin to appear, indicating breakdown of the primary wall.

Mercerization of a pressure boiled, severely bleached fabric causes extensive damage at the warp crowns (Figs. 12 and 13). Large amounts of primary wall are removed and breakdown of the secondary wall begins to occur. Even under these severe conditions, the weft shows no sign of damage (Fig. 14).

A scanning electron microscopic study of various fabrics reveals that as the severity of chemical processing conditions increases, fibre damage increases at localized areas of the fabric, namely the warp crowns. Fibre damage is almost always located only at the surface fibres of warp crowns. The rest of the warp fibres and all the weft fibres show little or no sign of damage. At the desired stage, some residual
Fig. 1 — Desized fabric at low magnification showing the warp and weft yarns (x100)

Fig. 2 — Desized fabric showing deposits on the warp crown (x500)

Fig. 3 — Warp crown of a solvent scoured fabric (x500)

Fig. 4 — Warp crown of a severely scoured fabric showing fibrillation (x500)
SCANNING ELECTRON MICROSCOPIC STUDY OF WET PROCESSING OF COTTON FABRICS

Fig. 5 — Warp crown of a desized severely bleached fabric showing erosion of the primary wall (x 550)

Fig. 6 — Primary wall removal at the warp crown of a solvent scoured severely bleached fabric (x 2400)

Fig. 7 — Severely scoured severely bleached fabric showing extensive fibrillation at warp crown (x 900)

Fig. 8 — The fabric in Fig. 7 at the higher magnification showing the secondary wall of a single note (x 4500)
Fig. 9 - Warp crown of a desized, severely bleached and mercerized fabric (x 550)

Fig. 10 - The fabric in Fig. 9 illustrating the removal of primary wall (x 2100)

Fig. 11 - A solvent scoured, severely bleached and mercerized fabric [only minor damage is seen] (x 440)

Fig. 12 - A severely scoured, severely bleached and mercerized fabric showing extensive damage at a warp crown (x 500)
sizing material appears to be adhering to the warp yarns, which is subsequently removed during scouring or bleaching. Solvent scouring appears least harmful in comparison with open soda boiling and pressure boiling. Pressure boiling with caustic is the severest operation which distinctly causes damage to fibre surfaces. Severity of bleaching also affects fibres, but to a smaller extent, while mercerization increases damage to the maximum extent. A pressure boiled, severely bleached and mercerized fabric shows distinctly the secondary walls of warp crown fibres at several places.

It is interesting to note that weft yarns are almost always clean and undamaged even after treatment under the severest of conditions. This is illustrated in Fig. 14 which shows the weft yarn of a desized, pressure boiled, severely bleached and mercerized fabric. The fibres of the yarn are clean and relatively undamaged. Wherever changes in the fibre surface are observed, they are always at the warp crowns.

It is difficult to prove or conclude at this stage how only fibres at the warp crowns form the site of damage. However, it can be seen from the various photomicrographs presented here that increasing severity of conditions of chemical processing increases the chances of observable damages to the warp crown fibres. Whether the observed damages find their origin inherently in the chemical processing or in the prehistory of the fabric during its mechanical processing is yet to be established. If it were only due to chemicals and severity of conditions of chemical treatments, damage would have been expected on warp and weft to the same extent with no choice of position of damage like warp crowns. Apparently, therefore, warp crowns seem to have suffered stresses or damages during the pre-wet processing stages and chemical treatments accentuate the damages, depending on the degree of damage and severity of conditions of processing. This is the probable explanation for damage at the warp crowns. Since they are protruding more above the fabric surface, they are more prone to any kind of abrasion or mechanical action. A further study will be undertaken to determine the reasons for damage to occur only at the warp crowns.

Physical Properties at Various Stages of Chemical Processing

After each stage of chemical processing, the physical properties of the fabrics were determined. The results are given in Table 1. The fabrics are grouped according to the initial scouring treatment given, i.e. no scour, solvent scour, kier boiled and soda boiled. Within each group, the fabrics are listed according to what is believed to be increasing severity of chemical treatment. By examining the physical properties of the fabrics in each group, one can see that there is no significant change in the properties of the fabric within a group after the initial scouring treatment. It can be inferred that after the initial scouring treatment, mild or severe bleaching and mild or severe
### Table 1 — Effect of Processing Conditions on Physical Properties of Cotton Fabric (39s/39s/120/72)

<table>
<thead>
<tr>
<th>No.</th>
<th>Sequence of processing</th>
<th>DCRA (W + F)</th>
<th>WCRA (W + F)</th>
<th>Flex abrasion cycles</th>
<th>Tear strength, g</th>
<th>Tensile strength, kg</th>
<th>Wax, %</th>
<th>Fluidity Rhes</th>
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<tr>
<td>A</td>
<td>Desized</td>
<td>190</td>
<td>117</td>
<td>5400 3300</td>
<td>1200 1100</td>
<td>30.4 27.1</td>
<td>0.56</td>
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<tr>
<td>A-1</td>
<td>Mild Bleached</td>
<td>157</td>
<td>109</td>
<td>4800 3400</td>
<td>1200 1100</td>
<td>29.5 26.8</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td></td>
<td>Mercerized</td>
<td>172</td>
<td>158</td>
<td>4300 2550</td>
<td>1200 1100</td>
<td>32.1 27.7</td>
<td>—</td>
<td>4.1</td>
</tr>
<tr>
<td>A-2</td>
<td>Severe Bleached</td>
<td>176</td>
<td>111</td>
<td>3300 3450</td>
<td>1150 1200</td>
<td>26.5 27.4</td>
<td>—</td>
<td>—</td>
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<tr>
<td></td>
<td>Mercerized</td>
<td>183</td>
<td>160</td>
<td>4550 2850</td>
<td>1200 1050</td>
<td>31.2 28.8</td>
<td>—</td>
<td>4.4</td>
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<tr>
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<td>Solvent scoured</td>
<td>153</td>
<td>141</td>
<td>500 550</td>
<td>700 700</td>
<td>31.5 30.2</td>
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<tr>
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<td>156</td>
<td>125</td>
<td>400 500</td>
<td>750 750</td>
<td>28.5 30.6</td>
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<tr>
<td></td>
<td>Mercerized</td>
<td>172</td>
<td>161</td>
<td>500 500</td>
<td>850 650</td>
<td>30.7 28.8</td>
<td>—</td>
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<tr>
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<td>162</td>
<td>130</td>
<td>450 550</td>
<td>800 750</td>
<td>29.8 29.8</td>
<td>—</td>
<td>—</td>
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<tr>
<td></td>
<td>Mercerized</td>
<td>171</td>
<td>162</td>
<td>450 650</td>
<td>700 650</td>
<td>31.3 26.1</td>
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<td>AC</td>
<td>Kier boiled</td>
<td>144</td>
<td>129</td>
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<td>1050 1150</td>
<td>26.3 26.7</td>
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<td>161</td>
<td>125</td>
<td>650 1000</td>
<td>850 800</td>
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<td>163</td>
<td>1450 1100</td>
<td>900 850</td>
<td>30.4 26.3</td>
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<td>—</td>
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<tr>
<td>AC-2</td>
<td>Severe bleached</td>
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<td>119</td>
<td>900 1150</td>
<td>950 850</td>
<td>28.2 26.7</td>
<td>—</td>
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<tr>
<td></td>
<td>Mercerized</td>
<td>183</td>
<td>162</td>
<td>1450 1600</td>
<td>1050 1050</td>
<td>30.5 24.0</td>
<td>—</td>
<td>4.5</td>
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<tr>
<td>AD</td>
<td>Soda boiled</td>
<td>172</td>
<td>123</td>
<td>2000 2000</td>
<td>1100 950</td>
<td>27.3 26.3</td>
<td>0.47</td>
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<tr>
<td>AD-1</td>
<td>Mild bleached</td>
<td>164</td>
<td>123</td>
<td>1350 750</td>
<td>950 850</td>
<td>28.2 26.0</td>
<td>—</td>
<td>—</td>
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<tr>
<td></td>
<td>Mercerized</td>
<td>183</td>
<td>160</td>
<td>1100 750</td>
<td>1100 1050</td>
<td>31.5 25.6</td>
<td>—</td>
<td>3.7</td>
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<tr>
<td>AD-2</td>
<td>Severe bleached</td>
<td>165</td>
<td>133</td>
<td>1400 1500</td>
<td>1100 1000</td>
<td>28.3 27.9</td>
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<tr>
<td></td>
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<td>158</td>
<td>1150 1100</td>
<td>1050 1050</td>
<td>32.8 21.1</td>
<td>—</td>
<td>4.5</td>
</tr>
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</table>

bleaching followed by mercerization does not change the properties to a great extent. There are of course minor changes in the properties, but these will not be discussed here.

Examination of the fabrics in the SEM has shown that the fabric which has been damaged the most is that which has had a kier boil, severe bleach and mercerization treatment, while the least damaged fabrics are those which have had no scour or an initial solvent scour. From Table 1 it is evident that a solvent process has the most detrimental effect on the properties. Kier boiling and soda boiling treatments have an intermediate effect.

Almost complete extraction of lubricants and cementing materials from the surface layers of cotton fibres by solvent scouring can affect properties like tear strength and abrasion resistance. This is confirmed by the data given in Table 1. The flex abrasion resistance and tear strength for the solvent scavenged fabrics are seen to have dropped drastically.

The effects of severe conditions of kier boiling are to reduce the initial crease recovery, to extract waxes and impurities, to reduce tear strength and abrasion resistance of the fabric and to dissolve or to create crevices in the cuticle and primary wall of cotton fibres. Under milder conditions of scouring, the fabric is seen to retain the initial crease recovery, tear strength and abrasion resistance, which subsequently have favourable effects on resin finishing.

Kier boiling and soda boiling etch the surface fibres, as seen in the scanning electron microscope, but these treatments do not affect the properties of the fabric as much as solvent extraction. Scouring, bleaching and mercerization, particularly the last operation, are considered more drastic for fibres which have had a prehistory of surface damage caused by mechanical means. The above three treatments accentuate the damage by exposing or removing primary or secondary wall layers at the warp crown in the fabric. It should be noted that only a very small percentage of the fibres in the warp crown show damage. It is assumed that the change in wax content observed for the soda boiled and kier boiled fabrics is partly due to the removal of wax at the damaged warp crown sites.

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### References