Development of Surface Modified Tencel Fabrics through the Control of Fibrillation(III)
-Effect of DP Finishing Method and NaOH Pretreatment-

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Abstract


Key words: fibrillation control, DP finishing, PDC method, WF method, NaOH pretreatment; 피브릴화 조절, DP가공, PDC방, WF방, NaOH 전처리

I. Introduction

Tencel has a high modulus, resulting low laundering shrinkage and shows higher strength in both wet and dry states compared to viscose rayon. However, it shows a distinct tendency to fibrillate during wet abrasion. This fibrillation property can be manipulated to obtain peach skin and other soft touch effects but it prevents Tencel from applying more widely in textile products due to adverse effect on the appearance of a garment after repeated washing during use. Therefore, it is necessary to control fibrillation for better
appeal and easier maintenance of a garment. Since fibrillation is caused by weak lateral cohesion of cellulose chain in crystalline region, inserting covalent bond between cellulose chains possibly leads to the reduction of fibrillation. Crosslinks for controlling fibrillation can be inserted by applying reactive compounds such as cationizing agents, durable press(DP) finishing agents and reactive dyes.²³

In this study, Tencel fabrics were crosslinked with a DP finishing agent, dimethyl dihydroxyethylene urea(DMDHEU), by using pad-dry-cure (PDC) and wet-fixation(WF) method before cellulase treatment for fibrillation control. Also, NaOH pretreatment was done to investigate its effect on strength retention during DP finishing. The effects of DP finishing method and NaOH pretreatment on performance properties, surface change, mechanical properties, and hand were investigated.

II. Experimental

1. Material

The fabric used was a desized and scoured 100% Tencel(3/1 twill, 110×74/inch², 237g/m², 0.39mm). Cellulase(Bio-Blue, Pacific Co., Ltd., Korea) from Tricoderma viride with an activity of 23.0 units/mg solid was used. Lubricant(Moderaz ACA, Protex Korea Co., Ltd., Korea) was used during mechanical fibrillation. Dimethyl dihydroxyethylene urea (DMDHEU, Floapret CL, BASF Inc.) was used for crosslinking with aluminum sulfide and citric acid as a mixed catalyst. Chemicals for preparing buffer and analysis were first grade. All other chemicals were reagent grade.

2. Fabric Treatment

1) NaOH Pretreatment

Fabric samples were treated with 19.8% aqueous NaOH solution for 1 min under tension, neutralized with 5% acetic acid solution, and rinsed with distilled water.

2) DP Finishing

For pad-dry-cure(PDC) method, samples were padded with solution containing DMDHEU(5% o.w.b) and catalyst(0.5% o.w.r. citric acid and 2.5% o.w.r. aluminum sulfide), dried at 100°C for 3 min, cured at 160°C for 3 min, and washed for further evaluation. For wet-fixation(WF) method, the padded fabrics were sealed in polyethylene bag and stored at 80°C for 20 min, dried, and cured at the same conditions as in PDC method.

3) Fibrillation

Fibrillation treatment was done in a bath containing lubricant(1g/ l ) at 80°C for 60 min at a liquor ratio of 30:1 with a rotary drum washer before cellulase treatment.

4) Enzyme Treatment

The fibrillated samples were treated with cellulase in a buffer[0.9M NaOH/1.4M CH₃COOH] solution of pH 5.0 at 60°C for 60 min in a rotary drum washer. To terminate the enzymatic reaction, the fabrics were treated with hot water (80°C) for 10 min, rinsed twice with warm water (40°C) for 10 min, and then dried.

3. Evaluation

1) Weight loss and Add-on

Fabric weight loss and resin add-on were determined on the basis of the conditioned weight of fabric sample after cellulase treatment and DP finishing, respectively.

2) Performance Properties

Performance properties were evaluated by
standard procedures including tensile strength, ASTM D-1682-64; tear strength by Elmendorf tester, ASTM D-1424-83; wrinkle recovery angle, AATCC 66-1990; DP rating, AATCC 124-1967. All the tests except wrinkle recovery angle were conducted in the warp direction.

3) Surface Change
For scanning electron microscopy (SEM, JEOL JSM 5400), samples were sputtered with gold/palladium and were examined to determine changes in surfaces.

4) Mechanical and Hand Properties
The low stress mechanical properties (tensile, shear, bending, compression and surface) of the treated fabrics were measured using the KES-FB. From the measured values of mechanical properties, primary hand values (HV) were determined by KN-101-WINTER for men’s winter suitimg materials. Total hand value (THV) was determined by KN-301-WINTER.

III. Results and Discussion

1. Effect on Performance Properties
The performance properties of cellulase-treated (control), DP finished (PDC or WF)/cellulase-treated, NaOH-pretreated /DP finished (PDC) /cellulase-treated are shown in Table 1. Compared with the cellulase-treated sample, DP finishing increased DP rating and decreased strength retention. Strength loss is caused by acid hydrolysis during curing and stress concentration on crosslinks. Add-on, weight loss, DP rating and WRA of the treated samples are similar irrespective of DP finishing method, PDC or WF. However, the NaOH-pretreated sample shows relatively low add-on compared with NaOH-nontreated samples. During NaOH pretreatment, short chain molecules in amorphous region are extracted out, resulting the reduction of amorphous region. This resulted in lower add-on of the NaOH-pretreated samples. On the other hand, weight loss of the NaOH-pretreated sample was higher than that of nontreated counterpart because the fiber structure is changed into a relatively open-up structure due to NaOH pretreatment. Weight loss is caused by the removal of surface fibrils produced during mechanical fibrillation done before cellulase treatment. It is known that swelling treatments such as mercerization have an accelerating effect on enzymatic degradation of cotton fiber. The NaOH-pretreated sample showed lower DP rating and wrinkle recovery angle due to lower add-on. Nevertheless, its strength was similar to nontreated counterpart. This result is not consistent with the case of cotton cellulose. PDC method gave slightly higher strength than WF method.

2. Effect on Surface Change
Figure 1 shows the effect of crosslinking through

<table>
<thead>
<tr>
<th>Sample</th>
<th>Properties</th>
<th>Add-on (%)</th>
<th>WL (%)</th>
<th>DP rating</th>
<th>WRA (w+f, °)</th>
<th>BS (%)</th>
<th>TS (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td></td>
<td>-</td>
<td>2.02</td>
<td>2.5</td>
<td>227</td>
<td>84</td>
<td>80</td>
</tr>
<tr>
<td>DP(WF)/C</td>
<td></td>
<td>2.16</td>
<td>0.72</td>
<td>3.3</td>
<td>226</td>
<td>80</td>
<td>73</td>
</tr>
<tr>
<td>DP(PDC)/C</td>
<td></td>
<td>2.19</td>
<td>0.75</td>
<td>3.5</td>
<td>225</td>
<td>82</td>
<td>77</td>
</tr>
<tr>
<td>NaOH/DP(PDC)/C</td>
<td></td>
<td>1.3</td>
<td>0.85</td>
<td>2.9</td>
<td>194</td>
<td>81</td>
<td>78</td>
</tr>
</tbody>
</table>

C: Cellulase-treated, WL: Weight loss, BS: Breaking strength retention, TS: Tear strength retention
DP finishing on fibrillation control. Compared with the cellulase-treated sample (A), the DP/cellulase-treated samples (B - D) show much less fibrils on the surface of the limited area. Less fibrils are observed in the DP finished sample by PDC method rather than WF method. And the NaOH-pretreated sample shows less fibrils than nontreated one. It is considered that fibrillation is controlled by forming crosslinks between DMDHEU and cellulose. This would affect enzymatic hydrolysis and further affect mechanical properties and hand of the treated sample.

3. Effect on Mechanical Properties

Table II shows mechanical properties of the treated samples. Tensile properties indicate extensibility and recovery of fabric to external stress. Crosslinking by DMDHEU did not affect tensile linearity (LT) of the cellulase-treated samples irrespective of DP finishing method. Tensile energy (WT) decreased and tensile resilience (RT) increased due to DP finishing. There is no difference in tensile properties depending on DP finishing method. NaOH pretreatment increased LT and WT, and decreased RT. Decrease in RT of the

Fig. 1. SEM pictures of the treated fabrics (x2.0k):
A; cellulase-treated, B; DP(WF)/cellulase-treated,
C; DP(PDC)/cellulase-treated, and D; NaOH/DP(PDC)/cellulase-treated.
NaOH-pretreated sample is consistent with lower DP rating and wrinkle recovery angle.

Bending properties are related with wear performance of a clothing such as wrinkle property and drapability. Bending rigidity(B) slightly decreased and bending hysteresis(2HB) slightly increased due to DP finishing. There is no difference in bending properties depending on DP finishing method. On the other hand, NaOH pretreatment increased B and increased 2HB greatly, resulting stiff hand and low bending recovery. The NaOH-pretreated sample seems to be appropriate for summer clothing material because it imparts silhouette maintaining some space between body and clothing.

Shear properties are accompanied with biaxial tensile and thus related with the shape of a clothing when it hangs down, ie, drapability. WF method increased shear properties and PDC method decreased shear properties. Compared with WF method, PDC method gave lower shear rigidity(G) and shear hysteresis(2HG, 2HG5), imparting better silhouette to a clothing. Shear properties was affected more greatly by NaOH pretreatment than DP finishing. Higher G, 2HG, and 2HG5 of the NaOH-pretreated sample indicate lower recovery from shear deformation.

Compressional properties are related with the fullness and buildliness of fabric. Irrespective of DP finishing method, compressional linearity(LC) and compressional resilience(RC) decreased while compressional energy(WC) increased. There is no difference in compressional properties depending on DP finishing method. LC and WC decreased, and RC increased in the NaOH-pretreated sample. Decrease in RC indicates decrease in recovery from compressional deformation. Consequently, decrease in bulkiness of the treated sample was resulted by DP finishing.

Surface properties are related with the smoothness of fabric. Irrespective of DP finishing method and NaOH pretreatment, geometrical roughness(SMD) decreased.

Thickness(T) and weight(W) of the treated samples are increased by DP finishing, as expected.

### Table II. Mechanical properties of the treated fabrics

<table>
<thead>
<tr>
<th>Treatment Properties (unit)</th>
<th>C</th>
<th>DP (WF) / C</th>
<th>DP (PDC) / C</th>
<th>NaOH/ DP (PDC) / C</th>
</tr>
</thead>
<tbody>
<tr>
<td>LT</td>
<td>0.67</td>
<td>0.66</td>
<td>0.68</td>
<td>0.82</td>
</tr>
<tr>
<td>Wt(g/cm²)</td>
<td>10.87</td>
<td>10.08</td>
<td>10.09</td>
<td>12.40</td>
</tr>
<tr>
<td>RT</td>
<td>50.23</td>
<td>52.34</td>
<td>52.02</td>
<td>45.8</td>
</tr>
<tr>
<td>BG(gf/cm²)</td>
<td>0.181</td>
<td>0.179</td>
<td>0.173</td>
<td>0.484</td>
</tr>
<tr>
<td>2HG(gf/cm²)</td>
<td>0.066</td>
<td>0.072</td>
<td>0.073</td>
<td>0.241</td>
</tr>
<tr>
<td>G(gf/cm deg)</td>
<td>0.51</td>
<td>0.55</td>
<td>0.42</td>
<td>0.88</td>
</tr>
<tr>
<td>2HG(gf/cm)</td>
<td>0.37</td>
<td>0.42</td>
<td>0.35</td>
<td>0.93</td>
</tr>
<tr>
<td>2HG5(gf/cm)</td>
<td>2.10</td>
<td>2.23</td>
<td>1.95</td>
<td>4.24</td>
</tr>
<tr>
<td>LC</td>
<td>0.49</td>
<td>0.43</td>
<td>0.45</td>
<td>0.39</td>
</tr>
<tr>
<td>WC(gf/cm²)</td>
<td>0.21</td>
<td>0.38</td>
<td>0.38</td>
<td>0.29</td>
</tr>
<tr>
<td>RC</td>
<td>34.21</td>
<td>29.15</td>
<td>27.56</td>
<td>36.87</td>
</tr>
<tr>
<td>MIU</td>
<td>0.204</td>
<td>0.235</td>
<td>0.214</td>
<td>0.185</td>
</tr>
<tr>
<td>MMD</td>
<td>0.016</td>
<td>0.015</td>
<td>0.015</td>
<td>0.015</td>
</tr>
<tr>
<td>SMD(micron)</td>
<td>3.85</td>
<td>3.46</td>
<td>3.74</td>
<td>3.17</td>
</tr>
<tr>
<td>T(mm)</td>
<td>0.600</td>
<td>0.785</td>
<td>0.767</td>
<td>0.680</td>
</tr>
<tr>
<td>W ingin/cm²</td>
<td>23.872</td>
<td>24.71</td>
<td>24.711</td>
<td>23.491</td>
</tr>
</tbody>
</table>

C: Cellulose-treated
Irrespective of DP finishing method, THV of the DP finished sample was similar. The NaOH-pretreated sample showed relatively lower THV among the treated samples. However, it is considered that each sample gave differentiated sensibility and hand.

IV. Conclusions

Tencel fabrics were crosslinked with a DP finishing agent, dimethylol dihydroxyethylene urea(DMDHEU), by using pad-dry-cure(PDC) and wet-fixation(WF) method before cellulase treatment for fibrillation control. Also, NaOH pretreatment was done to investigate its effect on strength retention during DP finishing. The effects of DP finishing method and NaOH pretreatment on performance properties, surface change, mechanical properties and hand were investigated.

Less fibrils are observed in the DP finished sample by PDC method rather than WF method. And the NaOH-pretreated sample shows less fibrils than nontreated one.

DP finishing increased DP rating and decreased strength retention. Add-on, weight loss, DP rating and WRA of the treated samples are similar irrespective of DP finishing method, PDC or WF. Crosslinking by DMDHEU did not affect LT, decreased WT, B, LC, RC, SMD, and increased RT, 2HB, and WC of the cellulase-treated samples irrespective of DP finishing method. Shear properties increased due to WF method, decreased due to PDC method. Only shear properties was affected by DP finishing method. WF method gave higher Koshi, Numeri and Fukurami value than PDC method. Irrespective of DP finishing method. THV of the DP finished sample was similar.

NaOH pretreatment resulted in lower add-on and consequently lower DP rating and WRA.
NaOH pretreatment increased LT, WT, RC, bending and shear properties and decreased RT, LC, WC, and surface properties. The NaOH-pretreated sample showed higher Koshi value and lower Numeri and Fukurami value. The NaOH-pretreated sample showed lowest total hand value. However, it is considered that each sample gave differentiated sensibility and hand, therefore more choices for uses.

Acknowledgement

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References