〔보 문〕

Journal of the Korean Society
of Clothing and Textiles
Vol. 26, No. 12 (2002) p.1749~1755

# Development of Surface Modified Tencel Fabrics through the Control of Fibrillation(III)
## -Effect of DP Finishing Method and NaOH Pretreatment-
# 피브릴화 조절을 통한 다양한 감성의 텐셀소재 개발(제3보)
## -DP가공 방법 및 NaOH 전처리가 미치는 효과-

Dept. of Clothing and Textiles · Human Ecology Research Institute, Chonnam National University
Younsook Shin · Kyounghee Son

전남대학교 의류학과 · 생활과학연구소
신 윤 숙 · 손 경 희
(2002. 7. 24 접수)

**Abstract**

본연구의 목적은 DP가공에 의한 가교화를 통해 텐셀의 피브릴화를 조절할 때 DP가공 방법 및 NaOH 전처리 효과를 물성, 표면형태, 역학적 성질 및 태의 변화의 관점에서 고찰하는데 있다. SEM 분석결과 DP가공에 의해 피브릴 발생정도는 감소하였다. DP가공 방법에 있어서 WF법과 PDC법에 따른 물성의 차이는 나타나지 않았다. 역학적특성의 경우 DP가공은 DP가공 방법에 상관없이 효소처리한 직물의 인장선형성에는 큰 영향을 주지 않았으나 인장에너지, 굽힘강성, 압축선형성, 압축 레질리언스, 기하학적거칠기는 감소시켰고 인장 레질리언스, 굽힘이력, 압축에너지는 증가시켰다. 전단특성은 WF법에서는 증가한 반면, PDC법에서는 감소하여 DP가공 방법에 따른 차이를 나타내었다. WF법이 PDC법보다 더 높은 Koshi, Numeri, Fukurami 값을 보였으며, 종합태 값은 비슷하게 나타났다. NaOH 전처리에 의해 수지부착량은 감소하였으나 감량률은 증가하였으며, DP성/물성은 더 낮게 나타났다. NaOH 전처리에 의해 인장선형성, 인장에너지, 압축 레질리언스, 전단 및 굽힘특성은 증가하였으나 인장레질리언스와 압축선형성, 압축에너지, 표면특성은 감소하였다. NaOH 전처리한 경우 Koshi는 증가하였고, Numrei와 Fukurami는 감소하였으며, 종합태 값은 가장 낮았다. 처리한 시료들은 각각 다른 감성과 촉감을 나타냈다.

***Key words***: fibrillation control, DP finishing, PDC method, WF method, NaOH pretreatment;
피브릴화 조절, DP가공, PDC법, WF법, 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.[1-3] 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 appearance 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.[4-7]

In this study, 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.

## II. Experimental

### 1. Material

The fabric used was a desized and scoured 100% Tencel(3/1 twill, 110×74/inch$^2$, 237g/m$^2$, 0.39mm). Cellulase(Bio-Blue, Pacific Co., Ltd., Korea) from Tricoderma viride with an activity of 23.0 units/mg solid was used. Lubricant(Modarez ACA, Protex Korea Co., Ltd., Korea) was used during mechanical fibrillation. Dimethyloldihydroxyethylene urea (DMDHEU, Fixapret 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(2g/$l$) 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 $CH_3COOH$) 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 suiting 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.[8] 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.[9] 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.[10] PDC method gave slightly higher strength than WF method.

### 2. Effect on Surface Change

Figure 1 shows the effect of crosslinking through

**Table 1. Performance properties of the treated fabrics**

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

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 finshing. 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

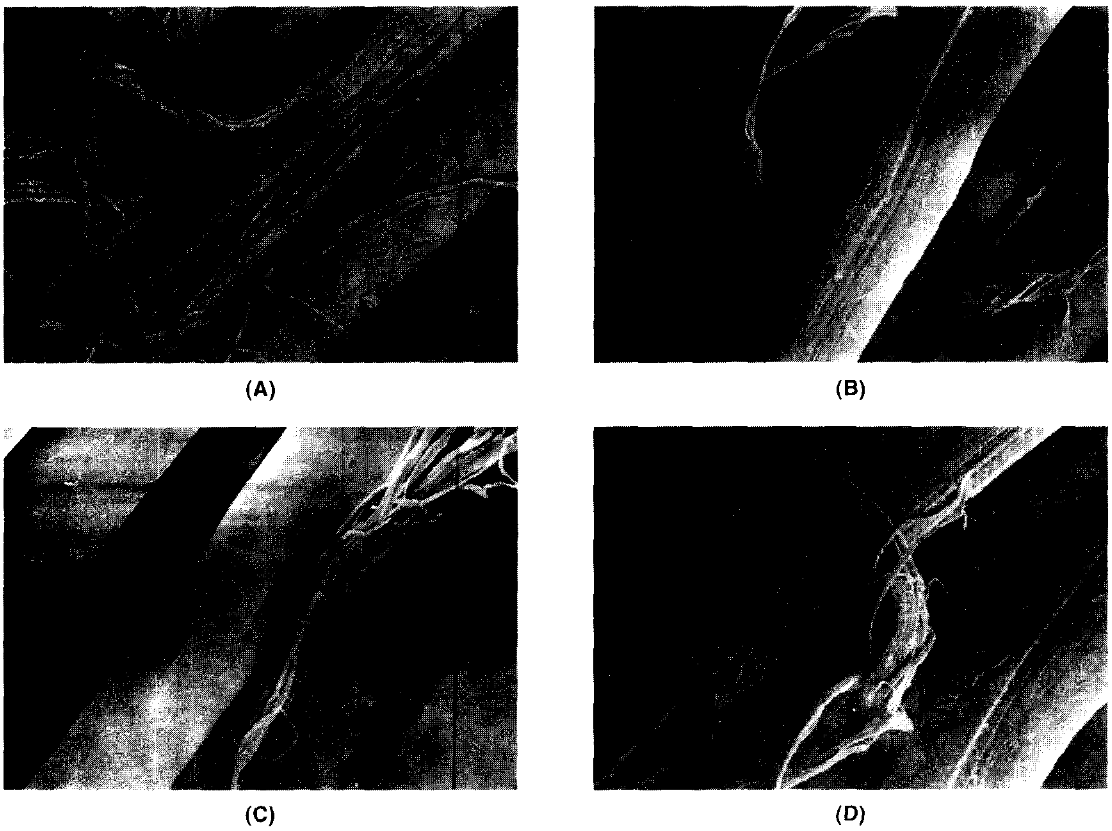


**Fig. 1. SEM pictures of the treated fabrics(x2.0k):**
**A; cellulase-treated, B; DP(WF)/cellulase-treated,**
**C; DP(PDC)/cellulase-treated, and D; NaOH/DP(PDC)/cellulase-treated.**

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.

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

| Treatment / Properties (unit) | C | DP (WF) /C | DP (PDC) /C | NaOH/ DP(PDC) /C |
|---|---|---|---|---|
| LT | 0.67 | 0.66 | 0.68 | 0.82 |
| WT(gf cm/cm$^2$) | 10.97 | 10.08 | 10.09 | 12.40 |
| RT | 50.21 | 52.34 | 52.02 | 45.8 |
| B(gf cm$^2$/cm) | 0.181 | 0.179 | 0.173 | 0.484 |
| 2HB(gf cm$^2$/cm) | 0.066 | 0.078 | 0.073 | 0.241 |
| G(gf/cm deg.) | 0.51 | 0.55 | 0.42 | 0.88 |
| 2HG(gf/cm) | 0.37 | 0.42 | 0.35 | 0.93 |
| 2HG5(gf/cm) | 2.16 | 2.23 | 1.93 | 4.24 |
| LC | 0.49 | 0.43 | 0.45 | 0.39 |
| WC(gf cm/cm$^2$) | 0.21 | 0.38 | 0.38 | 0.29 |
| RC | 34.21 | 29.15 | 27.55 | 36.87 |
| MIU | 0.204 | 0.205 | 0.214 | 0.185 |
| MMD | 0.016 | 0.015 | 0.015 | 0.015 |
| SMD(micron) | 3.85 | 3.46 | 3.74 | 3.17 |
| T(mm) | 0.600 | 0.785 | 0.767 | 0.680 |
| W(mg/cm$^2$) | 23.872 | 24.71 | 24.711 | 23.491 |

C: Cellulase-treated

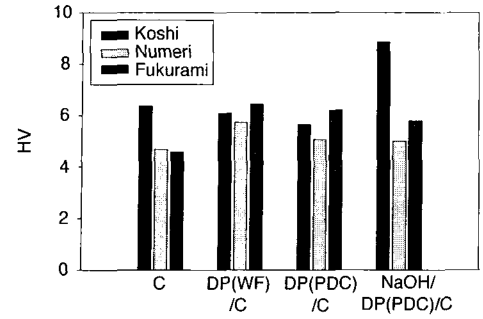
Compressional properties are related with the fullness and bulkiness of fabric. Irrespective of DP finishing method, compressional linearity(LC) and compressional resilience(RC) decreased while compressional energy(WC) increased. There is no difference in compressional properties depending on DP finishing 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 pretrearment, geometrical roughess(SMD) decreased.

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

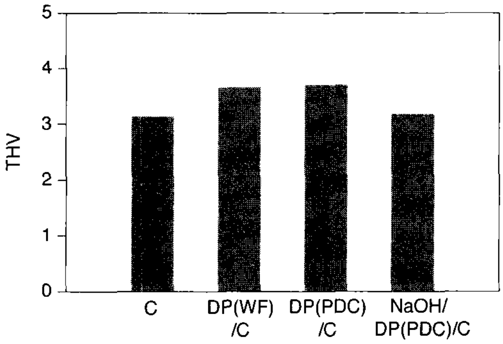
### 4. Effect on Primary and Total Hand

Figure 2 shows primary hand value(HV) of the treated samples. Koshi indicates stiffness and is highly related with bending and shear properties. The NaOH-pretreated sample having higher bending and shear properties showed the most stiff hand among the treated samples. The DP/cellulase-treated samples produced more flexible hand than the cellulase-treated sample. Numeri indicates smoothness and softness, and is




C: Cellulase-treated

**Fig. 2. Primary hand values of the treated fabrics.**




C: Cellulase-treated

**Fig. 3. Total hand values of the treated fabrics.**

influenced by surface properties. Numeri value increased in all samples, indicating that NaOH pretreatment and DP finishing increased smoothness and softness of the treated sample. Fukurami indicates bulkiness and resilience, and is related with compressional properties. Fukurami value increased in the DP/cellulase-treated sample compared with the cellulase-treated sample. Compared with PDC method, WF method gave higher Koshi, Numeri, and Fukurami value. The NaOH-pretreated sample showed stiffer, less bulky and less smooth hand than other samples.

Figure 3 shows total hand value(THV) of the treated samples. THV is the combination of HV. The DP/cellulase-treated samples show higher THV compared with the cellulase-treated sample. 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

This study was financially supported by the Chonnam National University in 1998. The authors deeply appreciate for the support.

## References

1. Woodings, C. R., "The development of advanced cellulosic fibres", *Int. J. Biol. Macromol.* **17**, 305-309(1995).
2. Albrecht, W., Reintjees, M., and Wulfhorst, B., "Lyocell fibers-alternative regenerated cellulose fibers", *Chem. Fibers Int.*, **47**, 298-304(1997).
3. Eichinger, D., and Eibl, M., "Lenzing lyocell-an interesting cellulose fiber for the textile industry", *Chem. Fibers Int.*, **46**, 26-30(1996).
4. Kumar, A., and Harnden, A., "Cellulase enzymes in wet processing of Lyocell and its blend", *Text. Chem. Color. & Am. Dyest. Reptr*, **1**(1), 37-41(1999).
5. Collier, B. J., "Effect of finishing and dyeing on Lyocell and Lyocell-blend fabrics", *Text. Chem. Color. & Am. Dyest. Reptr*, **1**(2), 40-45(1999).
6. Nechwatal, A., Nicoli, M., and Mieck, K. P., "Crosslinking reactions of spun-wet NMMO fibers and their influence on fibrillability", *Text. Chem. Color.*, 28(5), 24-27(1996).
7. Nicoli, M., Nechwatal, A., and Mieck, K. P., "Textile crosslinking reaction to reduce the fibrillation tendency of Lyocell fibers", *Text. Res. J.*, **66**, 575-580(1996).
8. Lewin, M, and Sello, S. B. Functional finishes, Marcel Dekker, Inc. New York & Basel, 13, 1983.
9. Mori, R., Haga, T., and Takagishi, T., "Changes in dyeability and morphology of cotton fiber subjected to cellulase treatment", *J. Appl. Polym. Sci.*, **65**, 155-164(1997).
10. Buschle-Diller, G., and Zeronian, S. H., "Enzymatic and acid hydrolysis of cotton cellulose after slack and tension mercerization", *Text. Chem. Color.*, **26**(4), 17-24(1994).