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# 以預浸吸工程改進架橋劑在加工布上之分佈、沉積與物性之研究

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## 中文摘要

本研究以三種不同分子量大小的直接染料於40°C~60°C下對經過三種不同架橋劑(DMDHEU-AA(0), DMDHEU-AA(1)及DMDHEU)本研究以預浸處理研究此工程對棉防皺加工布之物理性及架橋劑於加工布上之架橋構造之影響。結果顯示,在特定之預浸吸條件下,加工布上之氮含量、乾皺紋回復角及濕皺紋回復角均比未經預吸工程者高。在同一加工布之架橋密度下,架橋長度以經過預浸吸工程者較未經預浸吸工程者長。同時在同一加工劑濃度、加工織物強力保持率、及架橋密度下,所有條件下其加工織物之乾皺紋回復角及濕皺紋回復角均以經過預浸吸工程者較未經預浸吸工程者高。經過預浸吸工程之加工布其上架橋劑之表面化較未經預浸吸工程者低,經過預浸吸工程之加工布其上纖維之膨潤性較未經預浸吸工程者稍大。經由SEM之纖維切片觀察、架橋構造分析及架橋劑表面化之檢測,結果證實經過預浸吸工程之後,架橋劑可以滲入纖維內部,產生自身縮合聚合,造成加工布之乾皺紋回復角及濕皺紋回復角均有相當程度之提升。

**關鍵詞:** 預浸處理、皺紋回復角、架橋密度、架橋長度

## 一、前言

Our previous report<sup>11</sup> showed that some physical properties were improved by the polymerization of crosslinking agents in the treated fabrics. Franklin *et al.*<sup>8</sup> revealed that the use of dimethyloldihydroxyethyleneurea and acrylic acid (DMDHEU/AA) under redox catalyst system could improve some physical properties of the finished cotton fabrics. They suggested that the polymerization occurred between N-Methylol compound and acrylic acid during curing process. Some other previous studies<sup>3,4,7</sup> pointed out that the changes of finishing process, swelling state of the crosslinked fibers, and crosslinking agents could affect the crease recovery property of the crosslinked fabrics obviously.

We have interested to find that the dry and wet crease recovery properties of the treated fabrics are increased with the use of steeped procedure before traditional pad-dry-cure process for a longer time period and higher temperature. The detailed information about agent distribution, crosslinking structures, and physical properties of the treated fabrics under steeped procedure are highly interested to us. We will examine the crosslinking of cotton fabrics by using steep-pad-dry-cure and pad-dry-cure processes with respect to nitrogen and formaldehyde content, distribution of crosslinks per anhydroglucose unit (CL/AGU), length of crosslinks, some physical properties, and the surface distribution of crosslinking agent of the treated fabrics.

## 二、實驗方法

In this study, we used desized, scoured, and bleached cotton fabric 20s x 20s end (60) and picks (60).

The crosslinking agents used were dimethyloldihydroxyethyleneurea (DMDHEU) and acrylic acid (AA). Acrylic acid, hydrogen peroxide, and other chemicals were all reagent grade. The fabric samples were steeped with freshly prepared mixtures of DMDHEU (2, 4, 6, and 8% w/w) and acrylic acid at mole ratio of 1 to 1 in the presence of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub> initiator used were 0.017, 0.033, 0.050, and 0.067% H<sub>2</sub>O<sub>2</sub> (35% v/v), separately) under a N<sub>2</sub> environment at 70°C for 60 minutes, then the ammonium sulfate catalyst (0.1 times the used amounts of DMDHEU) was added to the solutions and stirred for about 5 minutes. The fabric samples were padded twice to about 100% wet pickup. Padded fabrics were dried at 80°C for 5 minutes, cured at 160°C for 3 minutes, soaped, washed, and dried.

Formaldehyde and nitrogen determinations were made using chromotropic acid 10 and kjeldahl methods, respectively. Tensile strength of the warp yarns was measured on an Instron tensile tester. ASTM standard D 1295-67 was used to determine dry and wet crease recovery angles.

Treated fibers were brought to the boil in a 50% (by volume) aqueous solution of methanol containing 0.5% wetting agent (Triton X-100) for 1 hour and allowed to cool and soak overnight in this wetting solution. Then the wet fibers were laid on a glass slide and covered immediately with nitrocellulose, which had been dissolved in acetone. The cross sections of the treated fibers were observed by the scanning electron micrograph.

## 三、結果與討論

The dry crease recovery angle (DCRA), wet crease recovery angle (WCRA), and tensile strength retention (TSR) of the finished fabrics under different resin concentrations with pad-dry-cure and steep-pad-dry-cure processes are listed in Table I. Table I shows that the values of DCRA and WCRA of the treated fabrics with specific steeped procedure are higher than those of normal treated fabrics and that the DCRA and WCRA values of the treated fabrics are gradually increase with the increasing of resin concentration used, but TSR values decrease in all cases. Table I also shows the values of DCRA, WCRA, and TSR of the treated fabrics under different resin concentration in the bath. At a given resin concentration all the DCRA, WCRA, and TSR values for the steep-pad-dry-cure process are higher than that for pad-dry-cure process.

The relationships between DCRA, WCRA, and TSR of the finished fabrics with the two processes are shown in Figures 1(a), 1(b), and 1(c), separately. From the relationships between DCRA and WCRA of the finished fabrics (Figures 1(a)), we find that the WCRA values of the treated fabrics for steep-pad-dry-cure process are higher than those for pad-dry-cure process at a given DCRA. Figures 1(b) and 1(c) show the plots of DCRA and WCRA versus TSR of the finished fabrics separately. For a given value of TSR all the DCRA and WCRA values of the treated fabrics for steep-pad-dry-cure process are higher than those for pad-dry-cure process. The higher DCRA and WCRA values for steep-pad-dry-cure process may be caused by the different amounts of resin bonded, crosslinking agent distribution, and crosslinking structure on/in the treated fabrics.

The nitrogen and formaldehyde contents of the cotton fabric crosslinked with varying resin concentrations for steep-pad-dry-cure process and pad-dry-cure process are presented in Table II. As expected, the values of nitrogen and formaldehyde contents show a gradual increase with increasing resin content in the bath in all cases. The nitrogen contents of the finished samples for steep-pad-dry-cure process are slightly higher than those for pad-dry-cure process at a given resin concentration in pad bath, but formaldehyde contents are inversely (shown in (a) and (b) of Figure 2 separately). Those results of nitrogen and formaldehyde contents at a same resin concentration support that the higher DCRA and WCRA of the steep-pad-dry-cure treated fabrics are not mainly caused by the amounts of resin bonded.

The number of crosslinks per anhydroglucose unit (CL/AGU), length of crosslinks (CL length), and the mole/AGU of nitrogen and formaldehyde of the finished fabrics listed in Table II, obtained using the methods of Frick *et al.*<sup>5,6</sup>, indicate that both increase as the concentration of the resin in the bath increases for all the two processes. The curvilinear relationship between the length of crosslinks and CL/AGU of the treated fabric samples for all the crosslinking agents (Figure 3) is similar to that reported in our previous study<sup>2</sup>. For a given number of CL/AGU the CL length values for steep-pad-dry-cure process are higher than those for pad-dry-cure process. We account that the longer CL length for steep-pad-dry-cure process is caused by the higher self-condensation of the crosslinking agents in steeped bath.

Figures 4(a), 4(b), and 4(c) respectively reveal the relationships between the values of DCRA, WCRA, and TSR and the values of CL/AGU of the various treated fabrics, which show that the values of DCRA and WCRA for steep-pad-dry-cure process are significantly higher than those for pad-dry-cure process; however, the values of TSR for steep-pad-dry-cure process are somewhat lower than those for pad-dry-cure process at a same value of CL/AGU. The significantly higher DCRA and WCRA values for steep-pad-dry-cure process may be attributed to the beneficial distribution of crosslinking agents under steeped procedure. The slightly lower TSR may be caused by the degradation of cellulose molecules in the presence of hydrogen peroxide at relative higher temperature and longer time period during steeped procedure.

## 四、結論

In this study, we used the steeped procedure to research the physical properties and crosslinking structure and it is found that the values of N content, DCRA, and WCRA of the treated fabrics with any specific steeped procedure are higher than those of normal treated fabrics. At a given resin concentration all the DCRA, WCRA, and TSR values for the steep-pad-dry-cure process are higher than that for pad-dry-cure process. For a given value of TSR all the DCRA and WCRA values of the treated fabrics for steep-pad-dry-cure process are higher than those for pad-dry-cure process. The CL length values for steep-pad-dry-cure process are higher than those for pad-dry-cure process for a given number of CL/AGU. The values of DCRA and WCRA for

steep-pad-dry-cure process are significantly higher than those for pad-dry-cure process, however the values of TSR for steep-pad-dry-cure process are somewhat lower than those for pad-dry-cure process at a same value of CL/AGU. The surface distribution of crosslinking agent on the finished fabrics for pad-dry-cure process is

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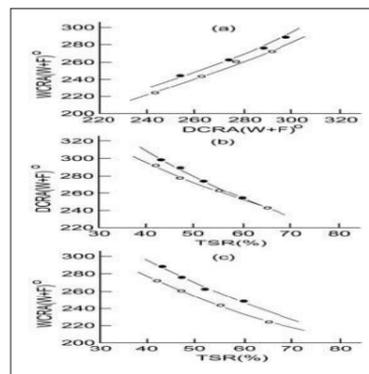


Figure 1. Relationship between (a) DCRA and WCRA, (b) DCRA and TSR, and (c) WCRA and TSR of the pad-dry-cure (○) and steep-pad-dry-cure (●) treated fabrics respectively.

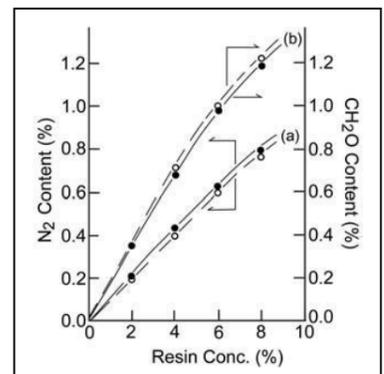


Figure 2. The (a) nitrogen contents and (b) formaldehyde contents of the pad-dry-cure (○) and steep-pad-dry-cure (●) treated fabrics for different resin concentrations respectively.

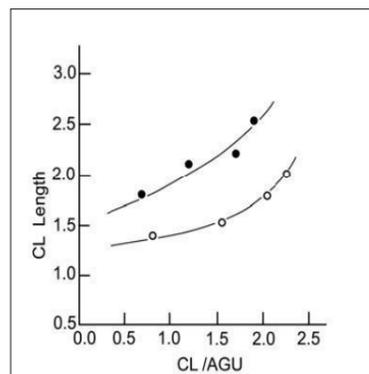


Figure 3. Relationship between CL length and CL/AGU of the pad-dry-cure (○) and steep-pad-dry-cure (●) treated fabrics for different resin concentrations.

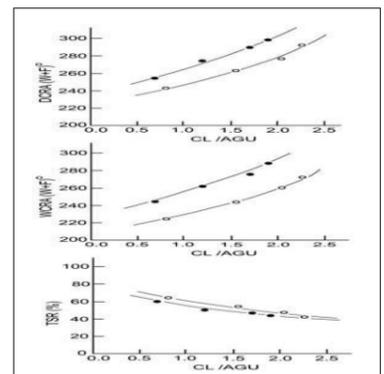


Figure 4. Relationships between (a) DCRA and CL/AGU, (b) WCRA and CL/AGU, and (c) TSR and CL/AGU of the pad-dry-cure (○) and steep-pad-dry-cure (●) treated fabrics for different resin concentrations.

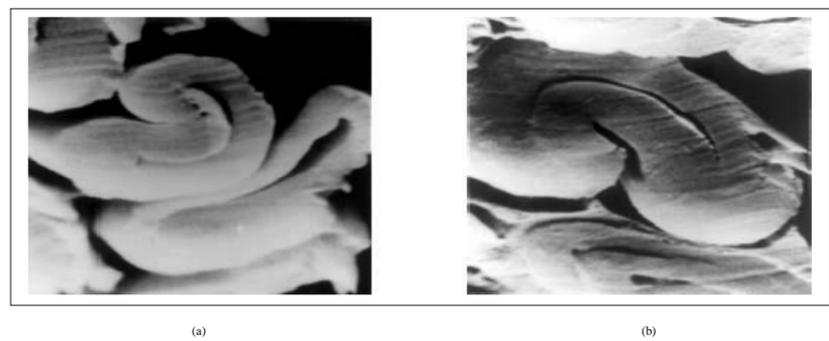


Figure 5. The expansion patterns (SEM) of cross section of the 4% mixture of DMDHEU and AA (mole ratio 1 to 1 under 70°C for 60 minutes) treated fibers with (a) pad-dry-cure process and (b) steep-pad-dry-cure process respectively.

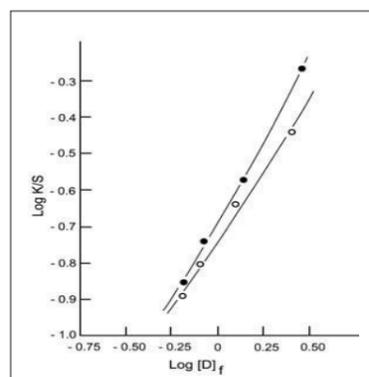


Figure 6. Plots of  $\log K/S$  versus  $\log [D]_f$  of the fabric samples treated with pad-dry-cure process (○) and steep-pad-dry-cure process (●) respectively.