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# Characterization of Cotton Fabrics Treated with Dialdehydes

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## 1. INTRODUCTION

Many aldehydes have been suggested as crosslink reagents for cellulose to impart resilience of cellulosic fabrics.[1,2] Nevertheless, aside from formaldehyde, only dialdehydes such as glyoxal and glutaraldehyde provided improvement in wrinkle recovery angle and DP rating of the treated fabrics.[1] These dialdehydes are one of very few available non-nitrogenous cellulose crosslink reagents that exhibit high reaction rates required for DP finishing of cotton.[3]

DP finishing of cotton with glyoxal was examined by Welch in 1980s.[3-5] Unfortunately, no comparison study was made with glutaraldehyde. Furthermore, aside from physical performance, little information is available for cotton treated with such dialdehydes. Therefore, the present study compares two most common dialdehydes on the DP treatment of cotton fabrics by using thermal, FT-IR spectroscopy, X-ray diffraction analysis, and others.

## 2. EXPERIMENTAL

Scoured, bleached, and mercerized 100% cotton fabrics were treated with dialdehyde crosslink reagents, glyoxal(40% solution) and glutaraldehyde(25% solution) by pad-dry-cure process. Aluminum sulfate(AS) was employed as a catalyst. A polyethylene-type softener(Siligen VN) and wetting agent(Triton X-100) were also added in the treating bath.

Conditioned wrinkle recovery angles(CWRA), K/S values after staining, and water imbibition value were measured. FTIR and UV-Visible spectroscopies, thermal analysis, and X-ray diffraction method were also used to evaluate the treated fabrics

## 3. RESULTS AND DISCUSSION

Figure 1 shows effects of dialdehyde concentration on CWRA. In this experiment, mole ratios between dialdehyde and AS were kept constant. Surprisingly, CWRAs were consistently higher in glutaraldehyde-treated fabrics than in glyoxal-treated fabrics at the same concentration of dialdehyde. Since glutaraldehyde has larger molecular size than glyoxal, an actual mole of glutaraldehyde used in the treating bath is less than that of glyoxal. Two reasons could be attributed to higher CWRA of the glutaraldehyde-treated fabric even at smaller mole fraction. Firstly, the mole ratio of AS versus glutaraldehyde(0.02:1) was higher than that of AS versus glyoxal(0.013:1) due to large molecular size of glutaraldehyde. The higher mole ratio of AS versus glutaraldehyde could cause greater CWRA of the treated fabric. Secondly, in aqueous solution glyoxal is mainly remained as dimer, trimer, 5- or 6-membered ring, or polymeric forms. Contrarily, glutaraldehyde retains monomeric forms. In the case of glyoxal, therefore, such easy formation of oligomeric or polymeric structure could adversely affect on its sorption ability to cotton, resulting in low CWRA values.

Figure 2 represents % residue and DTG peak temperature of the cotton fabrics treated at different concentrations of dialdehyde. After an initial decrease % residue continually increased with increase in concentration of dialdehyde. It should be noted that at low concentrations of dialdehyde % residues were lower than that of untreated control sample(12%). Thermal stability of the DP treated fabrics could be related to two separate factors that act in opposite directions: acid degradation during the treatment, which could reduce thermal stability, and crosslink effect, which could enhance the stability. At low dialdehyde concentrations, therefore, acid degradation effect seems to surpass the crosslink effect, resulting in lower % residue.

Table I showed TGA results obtained from varying curing temperatures at constant dialdehyde(6%) and AS(0.475%) concentration. Even though there are some deviations, effects of curing temperatures on various thermal parameters are very similar to concentration study, *i.e.*, with increase in curing temperatures % residue increased, DTG peak temperature decreased, and maximum rate of weight loss decreased. Obviously, at higher curing temperatures increase in crosslinking of cotton cellulose resulted in such increase in % residue and decrease in DTG peak temperature and maximum rate of weight loss. Results showed that activation energies of the untreated control, glyoxal-treated sample(3.8% add-on), and glutaraldehyde-treated sample(2.6% add-on) were 170 kJ/mol, 262-270 kJ/mol, and 180-210 kJ/mol, respectively. The dialdehyde treatment, therefore, generally increased thermal stability of the cotton cellulose.

As shown in Table II, K/S values of the dialdehyde-treated samples after staining were considerably lower than that of the untreated control. Moreover, K/S values of the glutaraldehyde-treated fabrics were lower than the glyoxal-treated fabrics at the same dialdehyde concentration, supporting higher efficiency of glutaraldehyde treatment. We also carried out water imbibition study by using centrifuge. The trend of water imbibition value was the same as that of direct dye staining.

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Table I. Effect of curing temperatures on thermal properties of the finished fabrics.

| Curing temperature (°C) | % add-on | | % residue | | Maximum rate of weight loss (%/min) | | DTG peak temperature (°C) | |
|---|---|---|---|---|---|---|---|---|
| | A[a] | B[b] | A | B | A | B | A | B |
| 100 | 2.0 | 2.5 | 13.3 | 17.8 | 25.1 | 24.8 | 345.3 | 330.8 |
| 110 | 2.6 | 2.6 | 14.6 | 17.2 | 18.9 | 22.7 | 329.0 | 330.2 |
| 125 | 3.3 | 2.6 | 22.4 | 18.2 | 17.8 | 27.2 | 327.2 | 355.1 |
| 140 | 3.6 | 3.1 | 17.7 | 20.8 | 17.8 | 16.7 | 324.6 | 334.4 |
| Untreated | -- | | 12 | | 28 | | 368 | |

[a]6% glyoxal and 0.475% AS. [b]6% glutaraldehyde and 0.475% AS.

Table II. K/S and water imbibition values of dialdehyde-treated fabrics cured at 125°C

| Dialdehyde conc.(%owb) | K/S values after staining | | Water imbibition values (%) | |
|---|---|---|---|---|
| | Glyoxal | Glutaraldehyde | Glyoxal | Glutaraldehyde |
| 3.6 | 1.42 | 0.80 | 17.6 | 16.3 |
| 4.8 | 1.17 | 0.68 | 19.9 | 15.6 |
| 6.0 | 1.12 | 0.66 | 22.9 | 14.4 |
| 7.2 | 1.04 | 0.60 | 17.0 | 13.8 |
| Untreated control | 3.887 | | 24.8 | |

## 4. REFERENCES

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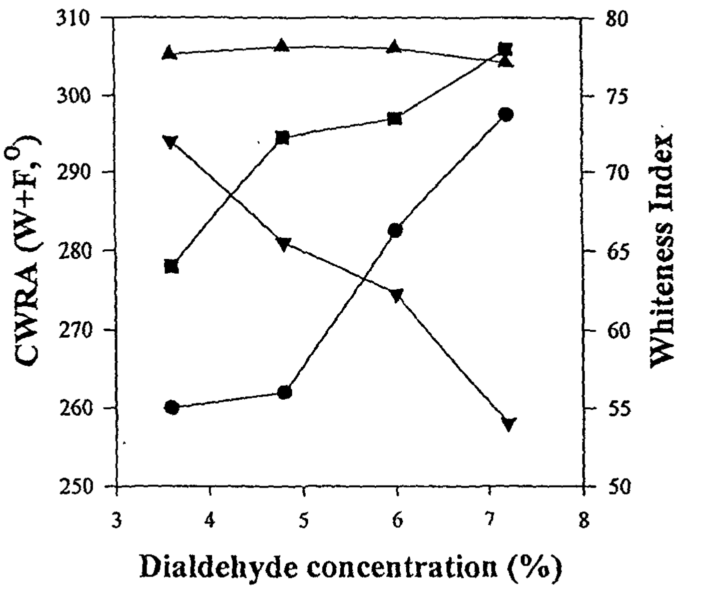


Fig.1 Effect of dialdehyde concentration on CWRA of the treated cotton cured at 125°C for 2 min.

● CWRA of the glyoxal-treated, ■ CWRA of the glutaraldehyde-treated,

▲ WI of the glyoxal-teated, ▼ WI of the glutaraldehyde-treated.

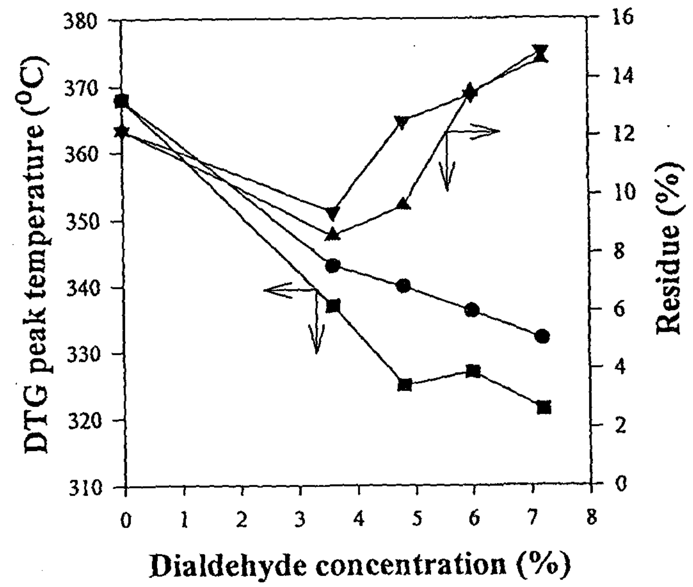


Fig.2 Effect of dialdehyde concentration on DTG peak temperature and %residue of the treated cotton.

● DTG peak temperature of the glyoxal-treated, ■ DTG peak temperature of the glutaraldehyde-treated

▲ % residue of the glyoxal-teated, ▼ % residue of the glutaraldehyde-treated.