

# Effect of Additives on Durable-Press Cotton Fabrics Treated with a Glyoxal/Glycol Mixture

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**ABSTRACT:** Cotton fabrics were treated with a glyoxal/glycol mixture for a nonformaldehyde durable-press finish by a pad-dry-cure method. Aluminum sulfate was used as a catalyst. The effects of additives such as sodium hydrogen sulfate, polyurethane, and a silicone softener were examined. Sodium hydrogen sulfate improved the whiteness and strength retention of the treated fabrics. The degree of whiteness of the treated fabrics was similar to that of fabrics treated with 1,2,3,4-butanetetracarboxylic acid. Polyure-

thane improved the wrinkle recovery angle and tearing strength retention of the treated fabrics significantly but impaired the whiteness. The softening agent increased the retention of the tearing strength and abrasion resistance significantly. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 96: 975–978, 2005

**Key words:** crosslinking; glyoxal; nonformaldehyde

## INTRODUCTION

A durable-press finish prevents cotton fabrics from wrinkling and stabilizes the shapes and dimensions of fabrics because of the improved resiliency and elasticity resulting from crosslinking in the amorphous regions of the cellulose molecules. The crosslinking agent and catalyst used in cellulose fabrics should not change the whiteness of treated fabrics even at high curing temperatures. It is also important to retain durable-press performance throughout the product life.<sup>1</sup>

Dimethyloldihydroxyethylene urea, which forms an ether linkage with hydroxyl groups of cellulose,<sup>2</sup> is most widely used as a crosslinking agent for the durable pressing of cellulose fabrics. However, because it releases formaldehyde either from treated fabrics or during finishing processes, its use in textile industries is limited. Active investigations into nonformaldehyde crosslinking agents, such as poly(carboxylic acid), which forms an ester linkage with hydroxyl groups of cellulose, have been carried out.<sup>1,3–6</sup> 1,2,3,4-Butanetetracarboxylic acid (BTCA) is the most effective ester-type nonformaldehyde crosslinking agent nowadays, but its price is high, and sodium hypophosphite,<sup>7</sup> the most commonly used catalyst for BTCA, can cause shade changes in fabrics dyed with certain sulfur or reactive dyes, and it brings about

eutrophication in rivers and lakes because of its phosphorus.

Glyoxal, the simplest dialdehyde, has structurally different characteristics than formaldehyde with respect to the formation of pentagonal chelating compounds with metallic ions.<sup>8</sup> Studies on the crosslinking of cellulose with glyoxal began in the 1960s. Gonzales and Guthrie<sup>9</sup> used magnesium chloride as a catalyst for glyoxal, and Matsui and Hosokawa<sup>10</sup> developed a process in which treated fabrics were bleached with hydrogen peroxide after crosslinking was carried out with zinc borofluoride as a catalyst. Worth and a coworker<sup>11,12</sup> treated cotton fabrics and cotton-polyester fabrics with glyoxal and reactive silicone with catalyst mixtures of aluminum sulfate and magnesium sulfate. Welch<sup>13,14</sup> found that the aluminum salt was best for glyoxal among the catalysts used for *N*-methylol compounds for durable-press finishes, and when the optimum amount of glycol was added to a padding bath, the durable-press performance was improved and the yellowing of treated fabrics could be prevented through changes in the crosslinking reactions and the subsequent structure of the crosslinkage.

In this study, the influence of additives on the wrinkle recovery angle (WRA), strength retention, and whiteness of treated fabrics was investigated: 100% cotton fabrics were treated with a glyoxal/glycol mixture with the pad-dry-cure process.

## EXPERIMENTAL

### Fabrics

Desized, scoured, bleached, and mercerized cotton cloth (84 × 64), weighing 118.9 g/m<sup>2</sup>, was used.

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TABLE I  
Effect of SHS on the Physical Properties of the Treated Fabrics

Sample	Glycol	SHS/glyoxal molar ratio	Weight gain (%)	WRA ( $w + f$ )	Breaking strength retention (%)	Tearing strength retention (%)	CIE whiteness index
1		0	7.17	294	27.5	19.9	56.73
2		0.5	7.07	284	31.22	23.7	66.27
3		0.75	6.98	282	31.45	24.6	67.41
4		1	7.04	282	31.66	24.2	71.34
5		1.25	6.88	278	31.18	24.8	71.86
6	EG	1.5	6.85	274	31.25	25.3	73.09
7		0	10.3	311	33.4	25.3	58.98
8		0.5	8.28	307	33.46	26.3	72.23
9		0.75	8.01	306	34.68	27	72.81
10		1	8.25	304	34.82	27.5	72.93
11		1.25	8.38	299	35.26	27.6	73.14
12	DEG	1.5	8.5	294	37.37	28	73.16

CIE = Commission Internationale de l'Eclairage;  $w$  = warp;  $f$  = filling.

Treating solution: glyoxal 5% (on weight of bath [owb]) and catalyst in water; wet pickup =  $93 \pm 2\%$ ; dry:  $85^\circ\text{C}$ , 3 min; curing:  $150^\circ\text{C}$ , 3 min; washing:  $50^\circ\text{C}$ , 30 min.; glycol/glyoxal molar ratio = 1.0.

## Chemicals

Glyoxal (Aldrich Chemical Co., Milwaukee, WI), ethylene glycol (EG; Aldrich Chemical), and diethylene glycol (DEG; first-grade; Duksan Pharmaceutical Co., Ansan, Korea) were used as crosslinking agents. Aluminum sulfate (Shinyo Pure Chemical Co., Osaka, Japan) was used as a catalyst. Triton X-100 (Shinyo Pure Chemical Co.) was used as a wetting agent. The additives were sodium hydrogen sulfate (SHS; Shinyo Pure Chemical Co.), polyurethane (30% solution; Pararesin UT-10N, Ohara Paragium Chemical Co., Kyoto,

Japan), and a silicone softener (Avivan 7066, Ciba Specialty Chemicals, Basel, Switzerland).

## Treatment of the fabrics

Aqueous padding solutions with or without additives consisted of crosslinking agents, a catalyst, and a wetting agent. The fabrics were padded with the two-dip/two-nip method, and the wet pickup was  $93 \pm 2\%$ . The dry temperature and time were  $85^\circ\text{C}$  and 3 min, respectively. The cure temperature and time were  $150^\circ\text{C}$  and 3 min, respectively. The treated fabrics were thoroughly washed with frequent stirring in

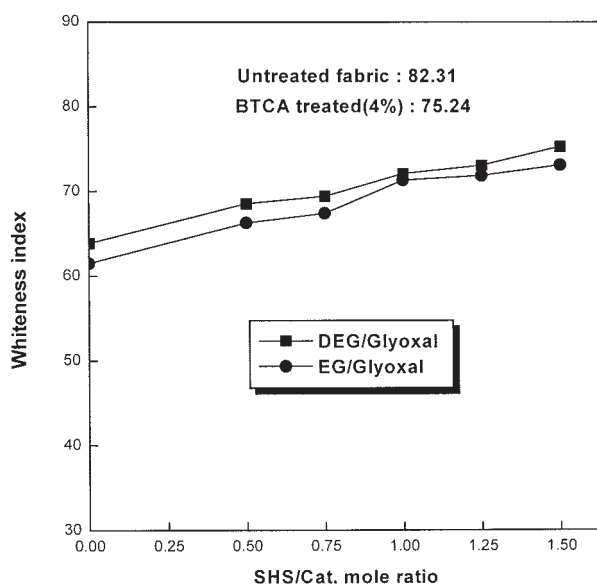


Figure 1 Effect of the SHS concentration on the Commission Internationale de l'Eclairage whiteness index of treated fabrics [glyoxal concentration = 5% (owb), glycol/glyoxal molar ratio = 1.0,  $\text{Al}_2(\text{SO}_4)_3$ /glyoxal molar ratio = 0.04, curing temperature =  $150^\circ\text{C}$ , time = 3 min].

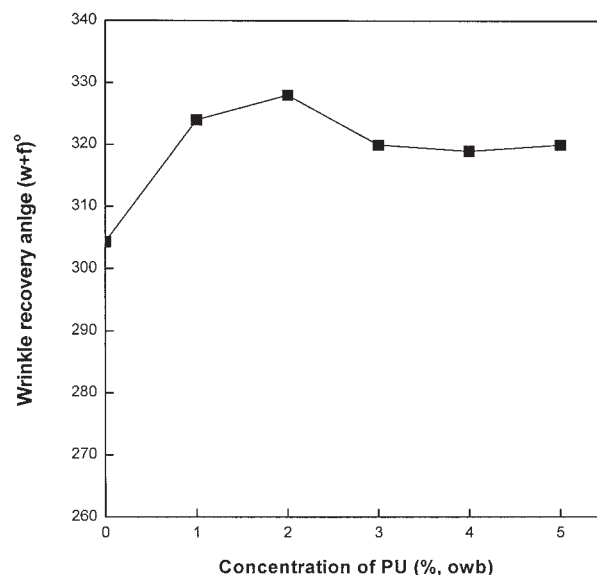


Figure 2 Effect of the polyurethane concentration on the WRA of treated fabrics [glyoxal concentration = 5% (owb), glycol/glyoxal molar ratio = 1.0,  $\text{Al}_2(\text{SO}_4)_3$ /glyoxal molar ratio = 0.04, curing temperature =  $150^\circ\text{C}$ , time = 3 min].

**TABLE II**  
Effect of the Polyurethane Concentration on the Physical Properties of the Treated Fabrics

Sample	Polyurethane concentration (% owb)	Weight gain (%)	WRA ( $w + f^o$ )	Breaking strength retention (%)	Tearing strength retention (%)	CIE whiteness index
1	0	8.25	304	34.8	27.5	72.93
2	1	16.4	324	31.9	34.7	63.65
3	2	16.3	328	31.8	42.7	63.8
4	3	16.7	320	32.5	40.5	63.68
5	4	16.8	319	31.4	38.2	67.1
6	5	16.8	320	31	37	65.74

CIE = Commission Internationale de l'Eclairage;  $w$  = warp;  $f$  = filling.

Treating solution: glyoxal 5% (on weight of bath [owb]); aluminum sulfate/glyoxal molar ratio = 0.04; DEG/glyoxal molar ratio = 1.0; SHS/catalyst molar ratio = 1.0; wet pickup =  $93 \pm 2\%$ ; dry: 85 °C, 3 min; curing: 150 °C, 3 min; washing: 50 °C, 30 min.

50°C water for 30 min and then dried. All the samples were conditioned at 20°C and 65% relative humidity.

### Testing and analysis

WRA was tested according to AATCC 66-1978. The tensile strength, tearing strength, and abrasion resistance were tested according to ASTM D 1682-64 (1-in. raveled strip), ASTM D 1424-83 (Elmendorf), and ASTM D 3883-80 (flex abrasion), respectively. The whiteness index was measured with an X-Rite spectrophotometer with illuminant D<sub>65</sub> and a 10° field.

## RESULTS AND DISCUSSION

### Effect of SHS

Cotton fabrics were treated with a padding solution containing 5% glyoxal, glycol (glycol/glyoxal molar ratio = 1.00), a catalyst (aluminum sulfate; catalyst/glyoxal molar ratio = 0.04), SHS, and a wetting agent (0.2% owb). Table I shows the effect of SHS on WRA, the retention of the breaking strength, and the whiteness of the treated fabrics. As the SHS concentration increased in the padding solutions, WRA decreased

slightly, whereas both the strength retention and the whiteness of the treated fabrics increased. In particular, the whiteness was significantly improved and was comparable to that treated with BTCA (4% owb) when the molar ratio of SHS to the catalyst was not less than 1.00 in glyoxal/EG padding solutions and 0.25 in glyoxal/DEG padding solutions (Fig. 1). Both the strength loss and yellowing of the durable-press finish fabrics were closely related to the activity of the catalyst. SHS, added to a padding bath, was competitive with aluminum sulfate and lowered the activity; consequently, it brought about a decrease in WRA and an increase in the whiteness of the treated fabrics. Therefore, SHS is a good additive for improving the whiteness of fabrics treated with either glyoxal/EG or glyoxal/DEG.

### Effect of polyurethane

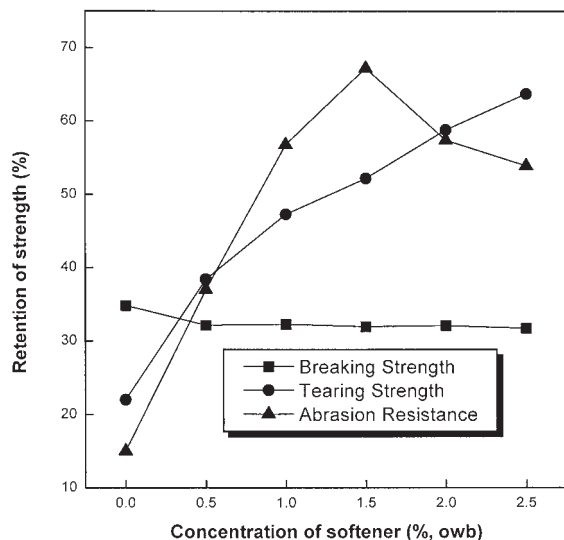
The cotton fabrics were treated with a padding solution containing 5% glyoxal, DEG (DEG/glyoxal molar ratio = 1.00), a catalyst (aluminum sulfate; catalyst/glyoxal molar ratio = 0.04), SHS (SHS/catalyst molar ratio = 1.00), polyurethane, and a wetting agent (0.2% owb). Table II shows the effect of polyurethane on the

**TABLE III**  
Effect of the Softener Concentration on the Physical Properties of the Treated Fabrics

Sample	Softener concentration (% owb)	Weight gain (%)	WRA ( $w + f^o$ )	Breaking strength retention (%)	Tearing strength retention (%)	Abrasion resistance retention (%)	CIE whiteness index
1	0	8.25	304	34.82	27.5	15.1	72.93
2	1	8.31	302	32.14	38.4	37.1	68.49
3	2	8.24	302	32.27	47.3	56.8	67.57
4	3	8.41	304	31.96	52.2	67.2	67.46
5	4	8.18	305	32.11	58.8	57.4	67.28
6	5	8.09	303	31.74	63.7	53.9	67.26

CIE = Commission Internationale de l'Eclairage;  $w$  = warp;  $f$  = filling.

Treating solution: glyoxal 5% (on weight of bath [owb]); aluminum sulfate/glyoxal molar ratio = 0.04; DEG/glyoxal molar ratio = 1.0; SHS/catalyst molar ratio = 1.0; wet pickup =  $93 \pm 2\%$ ; dry: 85 °C, 3 min; curing: 150 °C, 3 min; washing: 50 °C, 30 min.



**Figure 3** Effect of the softener on the strength retention of treated fabrics [glyoxal concentration = 5% (owb), glycol/glyoxal molar ratio = 1.0, SHS/ $\text{Al}_2(\text{SO}_4)_3$  molar ratio = 1.0, curing temperature = 150°C, time = 3 min].

WRA, strength retention, and whiteness of the treated fabrics. Figure 2 shows the WRA of the treated fabrics as a function of the polyurethane concentration. When polyurethane was added to a padding bath, the WRA and tearing strength retention increased, whereas the whiteness decreased. The WRA and tearing strength retention of the treated fabrics were thought to be enhanced by the film formation of polyurethane.<sup>15</sup>

### Effect of a softening agent

The cotton fabrics were treated with a padding solution containing 5% glyoxal, DEG (DEG/glyoxal molar ratio = 1.00), a catalyst (aluminum sulfate; catalyst/glyoxal molar ratio = 0.04), SHS (SHS/aluminum sulfate molar ratio = 1.00), a softening agent, and a wetting agent (0.2% owb). Table III shows the effect of a silicone softening agent on the physical properties of the treated fabrics. Figure 3 shows the strength retention as a function of the concentration of the softening

agent. As the concentration of the softening agent increased in a padding bath, the WRA and whiteness of the treated fabrics almost did not change, whereas the retention of both the tearing strength and abrasion resistance improved very much. It is thought that the softening agent acted as a lubricant in the treated fabrics so that slippage could occur between the fibers or at points of the intersection of the warp and filling when they were stressed.

### CONCLUSIONS

SHS, added to a glyoxal/glycol padding bath, improved the whiteness and strength retention of treated fabrics but reduced the WRA. The presence of polyurethane in a glyoxal/glycol padding bath increased both the WRA and tearing strength retention but reduced the whiteness of treated fabrics. The retention of both the tearing strength and abrasion resistance of treated fabrics improved very much, almost without the deterioration of the WRA and the whiteness when the softening agent was added to a glyoxal/glycol padding bath.

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