

# Performance Improvement of Nonformaldehyde Wrinkle Resistant Finished Cotton Fabrics Treated with Dialdehydes

Hyung-Min Choi\* and Yong Min Kim

*School of Textiles, Soongsil University, Seoul 156-743, Korea*

(Received September 11, 2001; Revised November 23, 2001; Accepted November 30, 2001)

**Abstract:** Additives, such as sodium perborate and borax, were examined in dialdehyde wrinkle resistant finishing of cotton. Results indicated that the whiteness index (WI) of cotton treated with dialdehyde and additive showed about 90% of WI of the untreated cotton but with decrease in wrinkle recovery angle (WRA) due to inhibition effect of these additives. Effect of additive on the WRA reduction was more prominent with glutaraldehyde than with glyoxal. Reduction in WRA of cotton treated with both dialdehydes and boron compound was minimized by simultaneous addition of formic acid in the bath. Addition of formic acid was also generally beneficial in maintaining WI retentions after 8 months storage. Furthermore, boron compounds were also effective in improving retentions of mechanical properties. By FTIR analysis the residual aldehyde group was detected on the dialdehyde-finished cotton, whereas no peak was shown by addition of boron compounds. This suggested that the residual aldehyde group was a main cause of fabric yellowing on the dialdehyde-finished cotton. Dialdehyde with boron compound, therefore, can be used to replace a conventional formaldehyde-containing wrinkle resistant finishing of cotton.

**Keywords:** Nonformaldehyde wrinkle resistant finish, Dialdehyde, Boron compound

## Introduction

In previous studies, efficacy of dialdehydes, such as glyoxal and glutaraldehyde, as wrinkle resistant finishing agent has been examined for cotton, ramie, and silk fabrics [1-14]. It was shown that these dialdehydes readily crosslinked cellulosic and protein fibers. Glutaraldehyde was more efficient crosslinker for cotton than glyoxal at the same dialdehyde concentration and curing temperature[2,4]. In addition, these dialdehydes are not only formaldehyde-free with low cost and commercially readily available, but also nitrogen-free, causing no chlorine retention[10-13].

Use of dialdehyde in wrinkle resistant finishing could also eliminate several problems associated with polycarboxylic acid such as butanetetracarboxylic acid. For instance, introduction of free carboxylic groups could cause dyeing problems for cotton with conventional anionic dyes, and use of the most efficient catalyst, sodium hypophosphite, produced shade changes in certain dyed fabrics[2]. Furthermore, the acetal bond is highly stable in alkaline condition used in successive dyeing and laundering processes[15]. When applied to cotton in the presence of acid catalyst, however, glyoxal tended to cause appreciable fabric yellowing and very high strength loss, for example, 71-81% loss for tearing strength and 66-74% loss for breaking strength at 145-155°C curing[11-14]. It was suggested that fabric yellowing during dialdehyde finishing was mainly due to the presence of unreacted aldehyde or hemiacetal groups in the treated fabric[8,11-14].

Various additives, such as ethylene glycol,  $\alpha$ -hydroxy acid, and polyethylene glycol[9-14], were examined to improve

performance properties of the cotton treated with dialdehydes. Addition of ethylene glycol suppressed yellowing of the treated fabrics along with increase in durable press appearance [12-14]. The presence of polyethylene glycol 600 or higher molecular weights to the glyoxal-finishing bath was also beneficial in enhancing strength retentions of the treated fabrics[9]. Oxidative after-wash treatment by using an oxidant (hydrogen peroxide) and stabilizer (sodium silicate) has also been suggested to minimize yellowing of glyoxal-finished cotton[8]. The use of an additional step, however, may hinder industrial application.

Alkali metal salts of weak acids have been used as buffering agents in glyoxal finishing[16]. Among the additives, sodium metaborate along with reactive silicone was highly effective for reducing the fabric acidity. However, the use of reactive silicone in finishing bath could induce undesirable water repellency[14]. Similarly, aluminum sulfate catalyst buffered with aluminum dihydroxyacetate stabilized with boric acid was examined in glyoxal-glycol process[13].

Nevertheless, the use of boron compounds in dialdehyde wrinkle resistant finishing process was never been fully investigated. Therefore, the objective of the present research is to study effects of two representative boron compounds, sodium perborate and borax, on performance properties of cotton fabrics treated with dialdehyde.

## Experimental

Desized, scoured, and bleached 100% cotton print cloth (Test fabrics #400) weighing approximately 107 g/m<sup>2</sup> was used throughout the study. Crosslinking agents used were glyoxal (40% aqueous solution) and glutaraldehyde (25% aqueous solution) from Junsei Chemicals Co., Ltd (Japan),

\*Corresponding author: hchoi@saint.soongsil.ac.kr

and acid catalyst was aluminum sulfate ( $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$ , Junsei). A mixture of magnesium chloride and citric acid supplied by Duksan Chemicals, Korea, was also examined as a catalyst in some cases. Triton X-100 (Aldrich) was used as a wetting agent and Siligen VN (polyethylene type from BASF) was used as a fabric softener. Sodium perborate ( $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$ , Junsei) and borax ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ , Shinyo Pure Chemicals, Co., Japan) were employed to improve whiteness of cotton treated with dialdehyde reagents. Formic acid (Duksan) was used to minimize adverse effects of boron compounds.

Cotton fabric was immersed for five minutes in a solution containing 6% o.w.b. (on the weight of bath) dialdehyde, 0.475% o.w.b. aluminum sulfate, 1% o.w.b. Siligen VN, 0.1% o.w.b. Triton X-100, and appropriate amounts of additives, unless otherwise noted. The liquor-to-goods ratio was 15:1. Two dips and nips using a laboratory padder gave a wet pickup of approximately 75%. The fabric was then dried at 85°C for 5 minutes and cured at 150°C for 3 minutes in a laboratory curing oven (Werner Mathis AG, Switzerland), unless otherwise specified. The sample was finally washed with running tap water at 40°C followed by drying at 85°C for 5 minutes.

After the finishing process, the sample was placed in a standard condition for at least 24 hours and %add-on was calculated. A Macbeth Color Eye 215 (D65 illuminant and 10° observer angle) with the Berger 76 method (ASTM D1925) was utilized to measure whiteness index of cotton fabrics. Other performance properties of the treated fabrics were determined by standard test methods such as Monsanto conditioned wrinkle recovery angle (ASTM 1295-67) and raveled-strip tensile strength (ASTM 1682-64) with H10K-S UTM (Houns Field Co., U.S.A.) in warp direction. The cotton samples were also analyzed by a Magma-FTIR

Spectrometer 55 (Nicolet, U.S.A.) with KBr pellet method at 32 scans and 4  $\text{cm}^{-1}$  resolutions.

## Results and Discussion

### Effect of Sodium Perborate (SP)

Table 1 shows effects of sodium perborate (SP) on whiteness index (WI) and wrinkle recovery angle (WRA) of the cotton fabrics treated with 6% glyoxal. The WI of cotton treated with glyoxal was 73.3% of that of the untreated control fabric. By addition of 0.2% SP, the WI of the glyoxal-treated cotton was considerably improved to 86%. Further addition of SP only slightly improved the WIs of cotton. At the same time, however, the WRAs of the treated cotton fabrics gradually decreased with increase in SP concentration. Addition of 0.2% SP resulted in most significant decrease in the WRA of the treated cotton, *i.e.*, 60°, compared with that of the cotton treated with glyoxal only. With addition of 1% SP the WRA of cotton was reduced by more than 100° along with substantial increase in mechanical properties of the treated cotton fabrics due to minimal crosslinking. Since the pHs of the baths without formic acid were relatively constant at around 3.1 regardless of the presence of SP, SP may interfere the reaction between glyoxal and cotton, presumably due to complex formation of SP with glyoxal[17].

To minimize such reductions in the WRA of cotton treated with glyoxal and SP while maintaining its WI, 2% formic acid (FA) was added in the treating bath. The WI of glyoxal-finished cotton fabric was initially decreased to 57.5% of that of the untreated control by adding 2% FA. However, simultaneous addition of SP and FA substantially improved the WIs and WRAs of cotton, suggesting synergistic effect of both additives. By addition of SP and FA in glyoxal

**Table 1.** Effect of SP on performance properties of cotton treated with 6% glyoxal<sup>a)</sup>

SP Conc. (%)	2% FA <sup>b)</sup>	Bath pH	Add-on (%)	WRA <sup>b)</sup> (°, w + f)	WI <sup>b)</sup>	RTS <sup>b)</sup> (%)	RE <sup>b)</sup> (%)
Control	--	--	--	190	22.1	100.0	100.0
0.0	No	3.1	2.8	314	16.2	22.1	64.9
0.2		3.1	2.7	254	19.0	41.0	86.3
0.4		3.1	2.8	234	19.5	55.3	93.3
0.6		3.1	3.1	233	19.6	76.2	107.0
0.8		3.2	3.1	249	19.8	71.1	103.4
1.0		3.1	2.8	199	20.0	74.3	107.6
0.0		Yes	1.9	2.7	320	12.7	18.2
0.2	2.0		4.0	291	18.0	39.4	79.5
0.4	2.2		3.9	290	18.8	64.2	95.7
0.6	2.3		4.0	276	19.1	70.2	96.4
0.8	2.3		4.0	283	19.2	69.5	96.3
1.0	2.4		3.9	281	18.9	73.1	100.3

<sup>a)</sup>The treating bath also contained 0.44% aluminum sulfate (AS), 1% Siligen VN, and 0.1% Triton X-100. The samples were dried at 85°C for 5 min and cured at 150°C for 3 min, <sup>b)</sup>FA=formic acid, WRA=wrinkle recovery angle, WI=whiteness index, RTS=retention of tensile strength, RE=retention of elongation.

**Table 2.** Effect of SP on performance properties of cotton treated with 6% glutaraldehyde<sup>a)</sup>

SP Conc. (%)	2% FA	Bath pH	Add-on (%)	WRA ( <sup>o</sup> , w + f)	WI	RTS (%)	RE (%)
Control	--	--	--	190	22.1	100.0	100.0
0.0	No	2.9	2.2	292	3.1	19.8	65.3
0.2		4.6	3.0	279	15.1	43.2	87.8
0.4		4.4	2.5	223	19.0	77.0	109.5
0.6		4.5	2.5	219	20.7	85.8	112.8
0.8		4.6	2.3	191	20.6	87.1	134.8
1.0		4.9	1.9	206	19.4	93.6	110.4
0.0	Yes	2.0	2.0	276	-0.34	21.6	62.7
0.2		2.0	1.8	301	11.8	36.7	76.8
0.4		2.1	1.8	291	18.1	52.8	89.0
0.6		2.2	1.6	273	19.3	58.6	92.1
0.8		2.3	1.4	270	19.3	70.6	97.6
1.0		2.4	1.2	272	20.0	72.5	97.9

<sup>a)</sup>For key, see Table 1.

finishing, mechanical properties of the treated cotton fabrics also increased considerably. This was probably due to the fact that FA acted as an acid catalyst to accelerate crosslinking reaction between dialdehyde and cotton fabric resulting in increase of WRA, whereas bleaching and buffering effects of SP increased WI and mechanical properties of the treated fabrics. Bleaching effect, due to formation of hydrogen peroxide, and buffering effect of SP are previously stated in the reference[16]. Results indicated that at 0.4% SP and 2% FA the treated cotton showed an optimal balance among WRA, WI, and retention of mechanical property.

Glutaraldehyde is also a highly effective crosslinking agent for cotton cellulose, but with yellowing[2]. As shown in Table 2, 6% glutaraldehyde treatment caused severe yellowing on cotton fabric indicated by mere 14% of the WI of the untreated control. Addition of SP drastically enhanced the WI of cotton, but the reduction in WRA was also great.

At 0.4% or higher concentration of SP the WRAs of the treated cotton were similar to that of the untreated control.

It should be noted that in the absence of FA, addition of SP increased pH of the bath containing glutaraldehyde to 4.6 from 2.9. This tended to suggest that a role of SP in glyoxal- and glutaraldehyde-containing baths might be different. Two contributing factors could be considered here on reduction of WRA by addition of SP: direct inhibition of SP on the crosslinking reaction between dialdehyde and cotton cellulose, and pH effect. With glyoxal the former factor was a main contributing factor whereas with glutaraldehyde both factors could influence on the reduction of WRAs, resulting in greater reduction (Tables 1 and 2). Nevertheless, both contributing factors might be associated with easier complex formation of SP with glutaraldehyde than with glyoxal.

Addition of FA in the treating bath further decreased the WI of the cotton treated with glutaraldehyde, resulting in negative WI value (Table 2). Simultaneous addition of SP

**Table 3.** Effect of BO on performance properties of cotton treated with 6% glyoxal<sup>a)</sup>

BO Conc. (%)	2% FA	Bath pH	Add-on (%)	WRA ( <sup>o</sup> , w + f)	WI	RTS (%)	RE (%)
Control	--	--	--	190	22.1	100.0	100.0
0.0	No	3.1	2.8	314	16.2	22.1	64.9
0.2		3.3	3.2	285	19.8	35.3	77.5
0.4		3.4	3.2	268	19.3	56.3	90.2
0.6		3.5	3.3	261	19.6	66.9	95.7
0.8		3.6	3.4	252	19.4	67.7	97.0
1.0		3.6	3.2	245	19.8	70.0	99.1
0.0	Yes	1.9	2.7	320	12.7	18.2	59.3
0.2		2.0	4.1	313	17.8	30.5	71.6
0.4		2.1	4.1	288	18.9	50.9	85.4
0.6		2.2	4.2	287	19.2	63.3	94.8
0.8		2.3	4.0	276	19.0	72.7	99.7
1.0		2.4	3.9	254	18.9	60.7	100.6

<sup>a)</sup>For key, see Table 1.

and FA again substantially improved the WI of the treated cotton while maintaining its WRA, but at least 0.4% SP was needed to obtain an appropriate level of WI with high WRA value. Retentions of mechanical properties were also enhanced with increase in SP concentrations for both with or without FA, as similar to the glyoxal-treated fabrics. Retentions were again lower with FA than those without FA.

### Effect of Borax (BO)

Table 3 shows effect of borax (BO) on performance properties of cotton treated with glyoxal with or without FA. Effects of BO on glyoxal-treated cotton were similar to those of SP in many aspects. In the absence of FA, the optimal balance between WI and WRA of the treated cotton was obtained at 0.2% BO which showed about 90% WI of the untreated control. Further addition of BO did not improve WI of the treated cotton, but the decrease in WRA was considerable. Nevertheless, the rate of the WRA reduction in BO-treated cotton fabric was much slower than that of the SP-treated cotton fabric (Table 1). Simultaneous addition of 2% FA and 0.2% BO in the bath increased WI of the treated cotton at high WRA. This result again substantiated that

addition of FA was needed in glyoxal finishing to enhance WRA and mechanical properties of cotton while maintaining high degree of whiteness.

Glutaraldehyde treatment of cotton resulted in substantial reduction in WI of the treated fabric as described previously. As shown in Table 4, however, addition of 0.4% BO increased the WI of cotton to about 90% of the WI of the untreated control from 14% without the additive. At the same time, reduction in WRA of the treated cotton was drastic with addition of BO and was minimized by simultaneous addition of BO and FA. Like the bath containing glutaraldehyde and SP, the addition of BO in glutaraldehyde bath increased the bath pH to the range of 3.4-4.2 from 2.9. Further addition of BO progressively reduced WRAs of the treated cotton fabrics regardless of the presence of FA, but rate of WRA reduction was much slower in the presence of FA.

In general, effects of two additives, SP and BO, on performance properties of cotton fabrics treated by glyoxal and glutaraldehyde were quite similar. The produced oxygen from both SP and BO could oxidize unreacted aldehyde groups and, consequently, increased WI of the treated cotton

**Table 4.** Effect of BO on performance properties of cotton fabrics treated with 6% glutaraldehyde<sup>a)</sup>

BO Conc. (%)	2% FA	Bath pH	Add-on (%)	WRA (°, w + f)	WI	RTS (%)	RE (%)
Control	--		--	190	22.1	100.0	100.0
0.0	No	2.9	2.2	292	3.1	19.8	65.3
0.2		3.4	1.9	280	14.2	36.3	78.4
0.4		3.8	2.0	232	19.8	65.9	100.6
0.6		3.9	2.0	210	20.9	75.6	111.3
0.8		4.0	1.9	194	20.7	90.5	113.4
1.0		4.1	2.3	169	19.2	89.5	122.0
0.0	Yes	2.0	2.0	276	-0.34	21.6	62.7
0.2		2.0	2.3	297	11.4	35.2	75.3
0.4		2.2	2.2	277	18.3	46.7	82.3
0.6		2.2	1.9	242	20.0	69.1	96.4
0.8		2.5	2.1	201	20.0	75.5	104.6

<sup>a)</sup>For key, see Table 1.

**Table 5.** Effect of curing temperature on performance properties of cotton treated with 9% glyoxal and 0.2% SP or BO<sup>a)</sup>

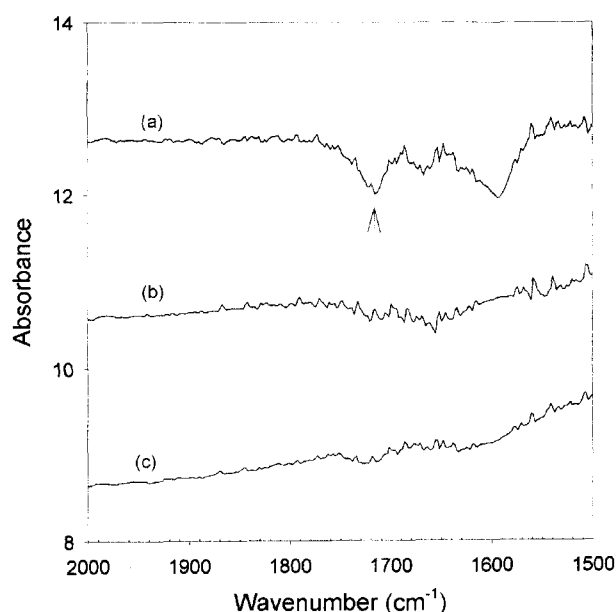
Curing temp. (°C)	Additive	Add-on (%)	WRA (°, w + f)	WI	RTS (%)	RE (%)
Control	--	--	190	22.1	100.0	100.0
100	No	4.5	285	22.1	51.6	83.2
	SP	3.7	265	22.1	63.9	92.7
	BO	3.9	279	22.1	60.4	89.3
130	No	4.9	302	20.9	29.6	66.8
	SP	4.6	291	21.4	53.2	83.2
	BO	5.1	294	20.9	45.1	76.5
150	No	4.1	323	15.6	17.1	59.7
	SP	4.7	319	18.4	27.8	66.8
	BO	4.6	307	17.7	27.6	66.5

<sup>a)</sup>The treating bath also contained 0.67% AS, 1% Siligen VN, and 0.1% Triton X-100. No FA was added. The samples were cured for 3 min.

[8]. Furthermore, these results consistently indicated that SP and BO tended to inhibit the reaction between dialdehyde and cellulose, presumably due to complex formation. This inhibition effect was greater with glutaraldehyde than with glyoxal. To obtain an optimal balance between WI and WRA, the use of FA or higher concentration of dialdehyde was needed.

### Effect of Curing Temperature

Table 5 indicated that at low temperature curing, fabric yellowing was not a problem with glyoxal finishing. Up to 130°C curing, WIs of the treated cotton fabrics were more than 90% of that of the untreated control regardless of the presence of SP or BO. The data showed that at least 130°C curing temperature was needed to obtain acceptable WRAs. At curing temperatures of 110°C and 130°C, SP and BO affected more on retentions of mechanical properties than on WI. This result suggested that SP and BO could also be added to improve retentions of mechanical properties of the treated cotton fabrics. Effects of boron compounds on mechanical properties of dialdehyde-treated have never been reported previously. SP tended to be somewhat better in enhancing retention of mechanical property of the treated cotton than BO. However, effects of SP and BO on WI of cotton were considerable at high curing temperature, *i.e.*, 150°C. It should be noted that WIs listed in this study were measured within two days after finishing process.



**Figure 1.** Subtracted FTIR spectra of the glutaraldehyde-treated cotton fabrics: (a) spectrum of cotton treated with 6% glutaraldehyde, (b) spectrum of cotton treated with 6% glutaraldehyde and 1% BO, and (c) spectrum of cotton treated with 6% glutaraldehyde and 1% SP. The spectrum of untreated cotton was subtracted from each spectrum.

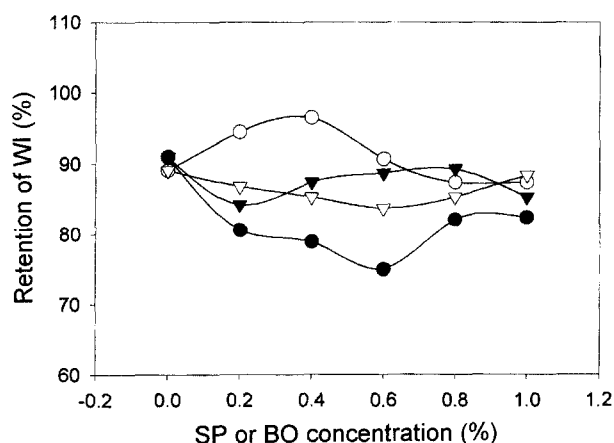
### FTIR Analysis

To investigate effects of SP and BO on dialdehyde-finished cotton, FTIR analysis was carried out for the fabrics with FA. The spectra shown in Figure 1 were obtained by subtracting the spectrum of untreated cotton from the spectrum of cotton treated with 6% glutaraldehyde without additives (Figure 1(a)), and from the spectra of cotton fabrics treated with glutaraldehyde and BO (Figure 1(b)) and glutaraldehyde and SP (Figure 1(c)). A small, but definite aldehyde carbonyl stretching peak at 1714  $\text{cm}^{-1}$  appeared in Figure 1(a) while no peak was shown at around 1720  $\text{cm}^{-1}$  in Figures 1(b) and 1(c), indicating absence of residual aldehyde groups with additives. These results revealed that the presence of residual aldehyde group was a main cause of fabric yellowing in dialdehyde treatment of cotton.

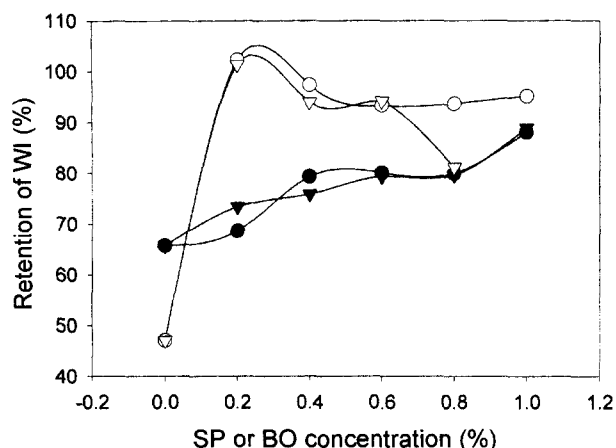
Hydrogen peroxide and oxygen liberated from SP and BO could behave as oxidizing agents for the unreacted aldehyde groups, resulting in increase in WI of the cotton fabrics treated with dialdehydes. It was also reported that oxidative after-washing treatment with  $\text{H}_2\text{O}_2$  significantly enhanced WI of the glyoxal-treated cotton by oxidation of the residual aldehydes to carboxylic acids and cleavage of the hemiacetals [8]. Nevertheless, no evidence of carboxyl carbonyl in the presence of additives was found in Figures 1(b) and 1(c). This was probably due to oxidation of aldehyde to hydroperoxide rather than carboxylic acid.

### Retention of WI during Storage

Effects of additives, such as BO and SP, on WI of cotton fabrics upon prolonged storage for 8 months in atmospheric environment were also measured and the results are shown in Figures 2 and 3. The WI retention of the untreated cotton fabrics was 95.3% after 8 months. In the absence of FA, addition of SP in glyoxal finishing substantially reduced WI



**Figure 2.** Retentions of WI of cotton fabrics measured after 8 months storage. The fabrics were treated with glyoxal in the presence of SP or BO: ●: SP and no FA, ○: SP and FA, ▼: BO and no FA, ▽: BO and FA.



**Figure 3.** Retentions of WI of cotton fabrics measured after 8 months storage. The fabrics were treated with glutaraldehyde in the presence of SP or BO: ●; SP and no FA, ○; SP and FA, ▼; BO and no FA, ▽; BO and FA.

of cotton fabrics after 8 months' storage whereas WI of cotton fabrics treated with BO and glyoxal was relatively consistent (Figure 2). Simultaneous addition of FA and SP substantially improved the WI retentions after 8 months. Effect of FA on WI of cotton treated with glyoxal and BO was not significant and at some concentrations the WI retentions were even slightly decreased.

As shown in Figure 3, however, effects of FA on maintaining WI retentions after 8 months' storage were more prominent on cotton treated with glutaraldehyde in the presence of SP or BO than those of cotton fabrics treated with glyoxal. In general, the presence of 0.2-0.4% SP or BO with FA was most beneficial in enhancing WI retentions of cotton treated with dialdehyde. Therefore, these results justify simultaneous addition of FA and SP or BO in dialdehyde treatment of cotton for nonformaldehyde wrinkle resistant finishing.

### Conclusions

SP and BO were equally effective in improving WI of cotton treated with dialdehydes. Simultaneous addition of FA and SP or BO, however, was generally needed to maintain WRA of the treated cotton due to inhibition effect of such additives on crosslinking reaction of dialdehyde and cotton. In addition, FA addition with SP or BO was generally beneficial in enhancing WI retentions of the treated fabrics after 8 months' storage. Effect of FA on maintaining WI retentions after 8 months' storage were more prominent on

cotton treated with glutaraldehyde in the presence of SP and BO than those of cotton treated with glyoxal. Addition of these additives also improved retentions of mechanical properties of cotton treated with dialdehyde. Results also indicated that reduction in WRA by addition of boron compounds in the absence of FA was greater with glutaraldehyde than glyoxal.

At low temperature curing, no additives may not be needed to obtain desirable WI of the treated cotton, but at high curing temperature addition of additives was required. Additives, however, could be employed to enhance mechanical properties of cotton treated at low curing temperature. FTIR analysis showed that the presence of residual aldehyde group was a main cause of fabric yellowing. No carbonyl-stretching peak, on the other hand, was shown in the fabrics treated with additives. Therefore, additives such as SP and BO along with FA can be used to obtain superior performance properties of cotton treated with dialdehyde for nonformaldehyde wrinkle resistant finishing.

### References

1. H. Choi, M. J. Park, and K. W. Oh, *Text. Chem. Color.*, **30**(12), 41 (1998).
2. H. Choi, J. H. Kim, and S. Shin, *J. Appl. Polym. Sci.*, **73**, 2691 (1999).
3. H. Choi, *Text. Res. J.*, in press (2001).
4. H. Choi and J. H. Kim, *Cell. Chem. Tech.*, **34**(5-6), 557 (2000).
5. J. G. Frick and R. J. Harper, *J. Appl. Polym. Sci.*, **27**, 983 (1982).
6. J. G. Frick, *J. Appl. Polym. Sci.*, **30**, 3467 (1985).
7. J. G. Frick, *Text. Res. J.*, **56**(2), 124 (1986).
8. J. E. Hendrix, K. J. Lane, and J. Y. Daniels, *U. S. Patent*, 4623356 (1986).
9. K. W. Oh, E. J. Jung, and H. Choi, *Text. Res. J.*, **71**(3), 225 (2001).
10. C. M. Welch and G. F. Danna, *Text. Res. J.*, **52**(3), 149 (1982).
11. C. M. Welch, *Text. Res. J.*, **53**(3), 181 (1983).
12. C. M. Welch, *U. S. Patent*, 4472167 (1984).
13. C. M. Welch and J. G. Peters, *Text. Res. J.*, **57**(6), 351 (1987).
14. C. M. Welch, *Rev. Prog. Coloration*, **22**, 32 (1992).
15. J. McMurry, "Fundamentals of Organic Chemistry", 4th ed., Brooks/Cole Publishing Company, New York, 1998.
16. D. L. Worth and E. A. Freeman, *U. S. Patent*, 4269602 (1981).
17. E. B. Whipple, *J. Am. Chem. Soc.*, **92**(24), 7183 (1970).