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[54] **PROCESS FOR CREASEPROOFING CELLULOSE-CONTAINING FABRIC WITH GLYOXAL-UREA-FORMALDEHYDE REACTION PRODUCT AND A BORON COMPOUND**

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[58] **Field of Search** 8/116.3, 133, 115.7; 260/29.4; 117/139.4

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[57] **ABSTRACT**

In combination glyoxal and a boron containing compound selected from the group consisting of the alkali metal borates and boric acid, the combination being incorporable into a resin forming reaction mixture to produce a durable press finish, and an improved process for treating textile fabrics with the same thereby yielding a treated fabric of significantly less discoloration then heretofore known and one exhibiting at the same time, improved strength properties.

5 Claims, No Drawings

PROCESS FOR CREASEPROOFING CELLULOSE-CONTAINING FABRIC WITH GLYOXAL-UREA-FORMALDEHYDE REACTION PRODUCT AND A BORON COMPOUND

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates generally to textile fabrics possessing durable press properties and, more particularly, to cellulose and cellulose-containing textile fabrics treated with novel glyoxal durable press finishes in combination with a boron containing compound, whereby the treated textile fabrics exhibit substantially reduced discoloration and improved strength properties.

2. Description of the Prior Art

Crease-resistant finishing agents, i.e., durable press finishes for fibrous material, particularly cellulose and cellulose-containing textile fabrics, have recently become widely used, and a large proportion of such materials now provided to the trade have been treated with such finishes for the production of fabrics having durable press characteristics. Cellulose reactants have been among such agents and one currently used durable press finish comprises the reaction product of glyoxal, urea and formaldehyde in an aqueous medium. While this prior art finish, when applied to a cellulose substrate, does impart certain durable press characteristics, to the treated substrate, e.g., dry wrinkle recovery and durable press qualitites, the latter properties have been sometimes offset by the discoloration of the treated product and by the loss of fabric strength.

In the past, an attempt has been made to employ boron-containing compounds to overcome the discoloration problem (yellowing) of cellulose attendant upon the alkaline degradation of cellulose in the alkaline treatment thereof with sulfone-type cross linking agents. Moreover, while borate compounds have been known to be used as cross linking catalysts for cellulosics, in amounts ranging between about 2 to 4 percent, no color improvement has been achieved therewith. Thus, it would be entirely unexpected that a boron-containing compound, when used in an acidic media or with acidic catalysts in conjunction with glyoxal based cross linking agents (finishes) applied to cellulosic fabrics, would overcome the prior art problems of discolored and weakened fabrics.

SUMMARY OF THE INVENTION

In accord with the invention, it has been discovered that a combination of glyoxal-based durable press finishes, formed by reacting glyoxal, urea and formaldehyde in an aqueous medium, and a boron-containing compound selected from the group consisting of alkali metal borates and boric acid in aqueous medium when applied to a suitable cellulose or cellulose containing textile fabric produces a fabric of significantly less discoloration and of improved strength.

The boron-containing additive may be incorporated into the glyoxal solution per se, in amounts between about 0.5 and about 20 weight percent, preferably between about 6 and about 14 weight percent, and still more preferably, about 9 weight percent; or, it may be added to an aqueous mixture of glyoxal, urea and formaldehyde or, alternatively, the additive may be placed into a suitable pad bath solution for application to the desired fabric. It has been found that the boron additive

in the pad bath solution should be in amounts ranging between about 0.01 and about 1.5 weight percent. Maximum reduction of discoloration of the finished treated fabric has been found to be at the level of about 0.5 weight percent boron additive in the pad bath solution. Indeed, amounts below 0.01 percent boron additive and above 1.5 percent boron additive show sharply increased discoloration levels. Thus, the concentration of boron additives is critical.

In terms of fabric content, the boron compound is normally present in amounts ranging between about 0.05 and 1.5 weight percent, based on the weight of fabric, preferably between about 0.125 and about 1.0 weight percent, and still more preferably between about 0.25 and about 0.5 weight percent, using a wet pick-up of 50 to 100 percent.

DESCRIPTION OF THE SPECIFIC EMBODIMENTS

In the usual practice of the invention, a durable press resin forming solution is prepared by reacting glyoxal, urea and formaldehyde in any desired sequence, and employing the resultant reaction product 1,3-dimethylol-4,5-dihydroxy-2-imidazolidone in an aqueous medium. This is a typical commercial resin forming solution presently utilized for durable press finished products.

As earlier indicated, an additive comprising a boron-containing compound selected from the group consisting of alkali metal borates and boric acid may be added either to the glyoxal solution per se or to the glyoxal, urea, formaldehyde reaction mixture in aqueous solution. Illustrative of the boron-containing compounds are the following: boric acid, sodium tetraborate, sodium metaborate, sodium perborate, potassium tetraborate and potassium pentaborate, merely to name a few. Particularly preferred additives on the basis of over-all performance have been found to be sodium tetraborate and sodium metaborate.

Alternatively, the boron additive is incorporated into a standard pad bath solution containing the finish, i.e., the glyoxal-based resin forming solution just described; a catalyst, such as zinc nitrate or magnesium chloride; a surfactant, such as Tergitol 15—S—9; a softener, such as Mykon SF; a hand builder such as polyvinyl acetate; and water. The fabric to be treated is passed through the solution for the purpose of wet pickup of the pad solution ingredients and then placed on pin tenter frames. Thereafter, the fabric is usually dried at an elevated temperature to remove the water therefrom. While drying can be accomplished by simply allowing the fabric to remain in contact with air at room temperature, it is preferred to dry the fabric at a temperature of from about 250° to 300°F. for a period of about 30 to about 120 seconds, the higher temperatures generally being employed in conjunction with the shorter drying times.

After this drying step, the fabric is cured by heating at a temperature sufficient to promote the reaction of resin forming solution with the fabric so as to bind it chemically thereto and thereby impart the durable press properties. The curing can be carried out at temperatures from about 300°to 360°F. for periods ranging from about 30 seconds to 15 minutes, with the higher temperatures generally being employed in conjunction with the shorter heating times. Subjecting the treated textile fabric to a two-step curing procedure involving a pre-curing period and a post-curing period, as herein-

before described, is preferred, but it is not a critical feature of the process of this invention. It will be obvious to one skilled in the art that both drying and curing could be accomplished in a single operation or sequential operation.

The term "cellulose and cellulose-containing textile fabrics" as used throughout the specification and claims is intended to include fabrics, whether woven, non-woven or knitted, and garments or other articles made from such fabrics. Fabrics containing cellulose, regenerated cellulose, and mixtures of the two are intended to be within the scope of the present invention. Illustrative of the textile fabrics to which this invention relates are cotton fabrics, linen fabrics, rayon fabrics, fabrics consisting of blends of cotton, linen or rayon fibers, and fabrics consisting of blends of cellulosic fibers and non-cellulosic fibers, such as, for example, polyester or polyamide fibers.

Thus, by subjecting any one of the above listed fabrics to a textile treating solution containing the glyoxal based resin forming mixture and the boron containing compound, it has been found that textile fabrics are obtainable, which exhibit substantial reduction in discoloration of the treated fabric, a stronger fabric and, as an incidental feature, reduction in shade change. As indicated, the amount of boron-containing additive is in the range between about 0.01 and about 1.5 weight percent of the pad bath treating solution. Maximum reduction of discoloration of the finished treated product has been found to be at the level of about 0.5 weight percent additive.

Evaluation of the textile fabrics subjected to the process of this invention was made in accordance with accepted test procedures of the ASTM (American Society For Testing Materials), or the AATCC (American Association of Textile Chemists and Colorists), or by procedures that are fully described herein. The following tests were conducted:

Dry Wrinkle Recovery	– AATCC Tentative Test Method 66–1959T
Tensile Strength	– ASTM Method D–1682 (Grab)
Tear Strength	– ASTM Method D–1424–59 (Elmendorf)
Damage Caused by Retained Chlorine	– AATCC Tentative Test Method 114–1967
Durable Press Rating	– AATCC Test Method – 124
Yellowness Index	– Measured with a Hunterlab Model D–40 reflectometer excluding filter $\text{Yellowness} = \frac{\text{Green-Blue Reflectance}}{100}$ Increasingly positive numbers indicate increasingly yellow fabric

The following definitions will also be applicable in the Examples and wherever appearing in the specification:

Catalyst X–4	– An acetic acid solution of zinc nitrate hexahydrate.
Mykon SF	– A nonionic paraffin-free polyethylene emulsion softening agent.
Tergitol 15–S–9	– A C_{11} to C_{15} linear chain alcohol-ethylene oxide adduct used as a wetting agent.

Boron salts used to effect improvements in the fabric performance properties can be introduced into the treating bath by a number of different procedures, as have already been indicated. One method involves the addition of the boron additive directly to the pad bath solution along with the other pad bath components. No adverse effects have been observed when the boron salts have been added to the durable press finish. Alternatively, another method involves the addition of the boron-containing material to a durable press finish and marketing the finish containing the additive, whence it will be eventually added to the pad bath solution. In yet another alternative method of introducing the boron salt to a durable press treating bath, the boron salt is added to a raw material, e.g., glyoxal, that is used to make a durable press finish. In other words, sodium metaborate can be added to glyoxal, the glyoxal used to make a durable press finish and the finish used to treat the fabric to impart durable press properties thereto. The various alternative methods of introducing the boron containing additive will be better understood in conjunction with the following specific examples. 1–7.

Example 1

The pH of a mixture of 170 grams of 40 percent glyoxal (1.17 moles) and 187.6 grams of 37.5 percent unhibited formaldehyde (2.34 moles) was adjusted to 5.5 with 7.4 grams of a 30 percent aqueous sodium acetate solution; 70.4 grams of urea (1.17 moles) was then added and the pH of the mixture raised to 7.0 with 1.3 grams of a 10 percent aqueous sodium hydroxide solution and maintained at this pH for the remainder of the reaction with the periodic addition of 14.8 grams of the 10 percent aqueous sodium hydroxide solution. The temperature of the reaction mixture was raised to 60°C. and maintained at this temperature for 1½ hours. The reaction product, 1,3-dimethylol-4,5-dihydroxy-2-imidazolidone, was cooled to room temperature and 24.0 grams of distilled water was added to make a 45 percent solution. The formaldehyde content of the 45 percent product was 0.59 percent, and pH 7.0 and the color 100 (Pt), ASTM D1209-62.

Example 2

The pH of a mixture of 170 grams of 40 percent glyoxal (1.17 moles) and 187.6 grams of 37.5 percent uninhibited form-aldehyde (2.34 moles) was adjusted to 5.5 with 7.6 grams of a 30 percent aqueous sodium acetate solution; 70.4 grams of urea (1.17 moles) was then added and the pH of the mixture raised to 7.0 with 1.3 grams of a 10 percent aqueous sodium hydroxide solution. Six and one-half grams of sodium tetraborate was added intermittently with 13.3 grams of 24 percent sodium hydroxide. The intermittent addition of base is necessary to maintain the pH of the reaction mixture between 6.0 and 7.0. After the sodium tetraborate had been added, the temperature of the reaction was raised to 60°C. and the pH maintained at 7.0 for 2 hours, after which another 6.5 grams of sodium tetraborate was added. The pH of the reaction was maintained at 7.0 during and after the reaction period by the addition of 31.8 grams of 10 percent sodium hydroxide. The formaldehyde content of the reaction product was nil, the color 65 (Pt) and the pH 7.0.

Example 3

The procedure used to make the finish of this example was the same as that used to make the finish of Example 2 with the exception of boric acid being used instead of sodium tetraborate. The formaldehyde content of the present finish was nil, the color 60 (Pt) and the pH 7.0.

Example 4

The pH of a mixture of 170 grams of 40 percent

glyoxal (1.17 moles) and 187.6 grams of 37.5 percent uninhibited formaldehyde (2.34 moles) was adjusted to 5.5 with 7.3 grams of a 30 percent aqueous sodium acetate solution; 70.4 grams of urea (1.17 moles) was then added and the pH of the mixture raised to 7.0 with 1.3 grams of a 10 percent aqueous sodium hydroxide solution and maintained at this pH for the remainder of the reaction with the periodic addition of 14.8 grams of the 10 percent aqueous sodium hydroxide solution. The temperature of the reaction mixture was raised to 60°C. and maintained at this temperature for 2 hours. The reaction product, 1,3-dimethylol-4,5-dihydroxy-2-imidazolidone, was cooled to room temperature and enough distilled water was added to make a 45 percent solution. The formaldehyde content of the 45 percent product was 0.30 percent, the pH 7.0 and the color 65 (Pt).

Example 5

To 170 grams of 40 percent glyoxal (Union Carbide Corporation) was added 15 grams of sodium metaborate. The mixture remained at ambient temperature (24°C.) for 3 days and was then used in the following manner to make a glyoxal-based finish. The pH of a mixture of 170 grams of 40 percent glyoxal (1.17 moles), to which had been added 15 grams of sodium metaborate and 187.6 grams of 37.5 percent uninhibited formaldehyde (2.34 moles) was adjusted to 5.5 with 5.3 grams of a 30 percent aqueous sodium acetate solution; 70.4 grams of urea (1.17 moles) was then added and the pH of the mixture raised to 7.0 with 1.0 gram of a 10 percent aqueous sodium hydroxide solution and maintained at this pH for the remainder of the reaction with the periodic addition of 12.2 grams of the 10 percent aqueous sodium hydroxide solution. The temperature of the reaction mixture was raised to 60° C. and maintained at this temperature for 1½ hours. The reaction product, 1,3-dimethyl-4,5-dihydroxy-2-imidazolidone, was cooled to room temperature and enough distilled water was added to make a 45 percent solution. The formaldehyde content of the 45 percent product was 0.20 percent, the pH 7.0 and the color 45 (Pt).

In order to illustrate that this invention is applicable to other commercially available glyoxal material, the finishes of Examples 6 and 7 were prepared using Nobel Bozel glyoxal, another commercially available glyoxal.

Example 6

The pH of a mixture of 170 grams of Nobel Bozel 40 percent glyoxal (1.17 moles) and 187.6 grams of 37.5 percent uninhibited formaldehyde (2.34 moles) was adjusted to 5.5 with 6.8 grams of a 30 percent aqueous sodium acetate solution; 70.4 grams of urea (1.17 moles) was then added and the pH of the mixture raised to 7.0 with 1.2 grams of a 10 percent aqueous sodium hydroxide solution and maintained at this pH for the remainder of the reaction with the periodic addition of 14.4 grams of the 10 percent aqueous sodium hydroxide solution. The temperature of the reaction mixture was raised to 60°C. and maintained at this temperature for 2 hours. The reaction product 1,3-dimethylol-4,5-dihydroxy-2-imidazolidone, was cooled to room temperature and enough distilled water was added to make a 45 percent solution. The formaldehyde content of the 45 percent product was 0.30 percent, the pH 7.0 and the color 90 (Pt).

Example 7

The procedure used to make the finish of Example 6 was the same used to make the finish of the present example with the exception that 15.0 grams of sodium metaborate had three days previously been added to 170.0 grams of Nobel Bozel glyoxal and this material was used to make the present finish. The formaldehyde content of the finish was 0.2, the color 70 (Pt) and the pH 7.0.

Each of the finishes made by the procedures of 1 through 7 were then individually applied to white cotton 136 × 64 broadcloth which had been desized, scoured, bleached and mercerized by padding through a laboratory padder at a wet pick-up of 75 percent. The fabrics were placed on pin tenter frames, dried at 300°F. for 1.5 minutes and cured for 1.5 minutes at 340°F. or 15 minutes at 320°F. in a laboratory oven.

Table 1 constituting Examples 8 to 14 lists the treating or pad bath formulations.

Tables II and III detail the fabric performance properties shown by fabrics treated by the bath formulations illustrated by Examples 8 to 14 respectively. In Tables II and III, the series denoted by Examples 8*a*, 9*a*, 10*a* show the effect of adding the sodium tetraborate or boric acid to the reaction of glyoxal, urea and formaldehyde to make the glyoxal-based finish used to impart durable press properties to the fabrics. The series denoted by Examples 11*a*, 12*a*, 13*a* and 14*a* show that sodium metaborate can be added to glyoxal which is subsequently used to make a glyoxal-based durable press finish and still yields the desired improvements in the fabric performance properties.

TABLE I.—TREATMENT BATH FORMULATION

Ingredients	Additive	Composition, percent by weight						
		Ex. 8	Ex. 9	Ex. 10	Ex. 11	Ex. 12	Ex. 13	Ex. 14
Durable press finish	None	17.8						
Do	Sodium tetraborate		17.8					
Do	Boric acid			17.8				
Do	None				17.8			
Do	Sodium metaborate					17.8		
Do	None						17.8	
Do	Sodium metaborate							17.8
Catalyst X-4 (mod. zinc nitrate, Sun Chem. Co.)		3.2	3.2	3.2	3.2	3.2	3.2	3.2
Mykon SF (polyethylene softener, Sun Chem. Co.)		1.8	1.8	1.8	1.8	1.8	1.8	1.8
Tergitol 15-S-9 (nonionic surfactant, U.C.C.)		0.1	0.1	0.1	0.1	0.1	0.1	0.1
Distilled water		77.1	77.1	77.1	77.1	77.1	77.1	77.1

TABLE II.—FABRIC PERFORMANCE PROPERTIES AT A CURE TIME OF 1.5 MINUTES AT 340° F.

Property	Treatment						
	Ex. 8a	Ex. 9a	Ex. 10a	Ex. 11a	Ex. 12a	Ex. 13a	Ex. 14a
Yellowness index	.067	.026	.030	.036	.029	.037	.032
Tear strength (warp/fill) grams	400/208	704/464	672/448	560/352	592/400	528/352	608/416
Tensile strength (fill) pounds	19	26	25				
Dry wrinkle recovery (warp-fill) degrees	300	283	278	298	302	297	274
Durable press rating tumble dry/spin dry	3.5/3.1	3.4/3.0	3.4/3.0				
Damage caused by retained chlorine, percent loss in strength	2.75	4.30	Nil				

TABLE III.—FABRIC PERFORMANCE PROPERTIES AT A CURE TIME OF 15 MINUTES AT 320° F.

Property	Treatment						
	Ex. 8a	Ex. 9a	Ex. 10a	Ex. 11a	Ex. 12a	Ex. 13a	Ex. 14
Yellowness index	.124	.067	.066	.074	.048	.081	.053
Tear strength (warp/fill) grams	320/128	640/416	576/368	416/288	512/352	352/272	512/352
Tensile strength (fill) pounds	75	22	24				
Dry wrinkle recovery (warp-fill) degrees	296	293	292	307	309	293	293
Durable press rating tumble dry/spin dry	3.7/3.3	3.4/3.2	3.4/3.1				
Damage caused by retained chlorine, percent loss in strength	4.95	Nil	Nil				

A convenient method for introducing the boron salts is to add them directly to the treating bath. However, no loss in efficiency was noted when the salt was added either to the durable press finish or to the glyoxal or to the other of the raw materials used to make the durable press finish, i.e., urea or formaldehyde.

Effect of Boric Acid Salts on Fabric Discoloration and Fabric Strength

series, Examples 15*a*–17*a*, showed the effect of sodium tetraborate and boric acid on fabric properties and the second series, Examples 25*a* and 26*a*, showed the effect of sodium tetraborate on fabric properties using another commercial durable press finish, American Cyanamid Company's "Aerotex" 101.

Fabric performance properties are summarized in Table V below.

TABLE IV.—TREATING BATH FORMULATION

Ingredients	Composition, percent by weight											
	Ex. 15	Ex. 16	Ex. 17	Ex. 18	Ex. 19	Ex. 20	Ex. 21	Ex. 22	Ex. 23	Ex. 24	Ex. 25	Ex. 26
Durable press finish (Permafresh 183, Sun Chemical Company)	17.8	17.8	17.8	17.8	17.8	17.8	17.8	17.8	17.8	17.8		
Modified zinc nitrate catalyst (Catalyst X–4, Sun Chemical Company)	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2
Polyethylene softener (Mykon SF, Sun Chemical Company)	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8
Nonionic surfactant (Tergitol 15–S–9, Union Carbide Corp.)	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sodium tetraborate		0.5										0.5
Sodium metaborate					0.5							
Sodium perborate						0.5						
Potassium tetraborate							0.5					
Potassium pentaborate								0.5				
Ammonium biborate									0.5			
Ammonium pentaborate										0.5		
Boric acid			0.5									
Distilled water	77.1	76.6									77.1	76.6
Durable press finish (Aerotex 101, American Cyanamid Company)											17.8	17.8

TABLE V.—FABRIC PERFORMANCE PROPERTIES

Property	Examples											
	15a	16a	17a	18a	19a	20a	21a	22a	23a	24a	25a	26a
Yellowness index	.134	.080	.084	.083	.069	.069	.075	.078	.148	.135	.041	.032
Tear strength warp/fill (grams)	384/256	496/336	432/272	416/288	512/336	496/320	480/352	448/320	384/288	368/240	608/415	656/464
Dry wrinkle recovery (degrees)	297	285	289	288	277	284	280	285	283	280	280	258

NOTE.—The curing temperature for Examples 25a and 26a was 320° F.

In accord with the invention, the presence of certain boric acid salts in treating baths, containing commercial glyoxal-based durable press finishes, beneficially reduces fabric discoloration that is normally associated with this type of fabric treatment. They also significantly improve the fabric strength properties.

White cotton 136 × 64 broadcloth desized, scoured, bleached and mercerized was padded with the treating baths detailed in Table IV, and identified as Examples 15–26, using a laboratory padder at a wet pick-up of 75 percent. The fabrics were then placed on pin tenter frames, dried at 300°F. for 1.5 minutes and cured at 340°F. for 15 minutes, except for the fabrics of Examples 25*a* and 26*a* which were cured at 320°F. for 15 minutes in a laboratory oven.

Three series of experiments were performed to illustrate the degreee of improvement rendered. The first

With the exception of the treatments containing the ammonium borates, the difference in each series between the control treatment and the treatments containing boron salts show the significant decrease in fabric discoloration and the increase in fabric strength that are effected by the presence of boron-containing materials. On the basis of over-all performance, sodium tetraborate and sodium metaborate are to be preferred. Although there is a slight decrease in durable press properties (dry wrinkle recovery), this deficiency can be overcome by altering the composition of the treating bath as will be seen subsequently.

Effect of Concentration of Sodium Tetraborate and Sodium Metaborate on Fabric Discoloration and Fabric Strength

The degree of improvement in fabric discoloration and fabric strength is effected by the concentration of

sodium tetraborate or sodium metaborate in the treating bath. Following are examples which show the degree by which discoloration and strength are effected.

White cotton broadcloth desized, scoured, bleached and mercerized was padded (Examples 27 through 36; summarized in Table VI) through a laboratory padder at a wet pick-up of 75 percent. The fabrics were then placed on pin tenter frames, dried at 300°F. for 1.5 minutes and cured to 1.5 and 15.0 minutes at 340°F. in a laboratory oven. "Permafresh" 183 was the commercial glyoxal-based durable press finish used in these experiments.

The first series, Examples 27*a*–31*a*, showed the effect of the concentration of sodium tetraborate. The second series, Examples 32*a*–36*a*, showed the effects of sodium metaborate concentration. Fabric performance properties are summarized in Tables VII and VIII below.

TABLE VI.—TREATING BATH FORMULATION

Ingredients	Compositions, percent by weight									
	Ex. 27	Ex. 28	Ex. 29	Ex. 30	Ex. 31	Ex. 32	Ex. 33	Ex. 34	Ex. 35	Ex. 36
Durable press finish (Permafresh 183, Sun Chemical Company)	17.8	17.8	17.8	17.8	17.8	17.8	17.8	17.8	17.8	17.8
Modified zinc nitrate catalyst (Catalyst X-4, Sun Chemical Company)	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2
Polyethylene softener (Mykon SF, Sun Chemical Company)	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8
Nonionic surfactant (Tergitol 15-S-9, Union Carbide Corp.)	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sodium tetraborate		0.25	0.50	0.75	1.00					
Sodium metaborate							0.25	0.50	0.75	1.00
Distilled water	77.1	76.85	76.6	76.35	76.1	77.1	76.85	76.6	76.35	76.1

TABLE VII.—FABRIC PERFORMANCE PROPERTIES AT A CURE TIME OF 1.5 MINUTES AT 300° F.

Property	Treatment									
	Ex. 27a	Ex. 28a	Ex. 29a	Ex. 30a	Ex. 31a	Ex. 32a	Ex. 33a	Ex. 34a	Ex. 35a	Ex. 36a
Yellowness index	.040	.036	.041	.035	.044	.043	.049	.036	.039	.040
Tear strength (warp/fill) grams	560/368	576/416	688/480	816/544	864/624	560/345	650/460	740/500	805/570	895/705
Tensile strength (fill) pounds	20	25	25	27	32	19	22	25	28	31
Dry wrinkle recovery (warp-fill) degrees	293	286	274	269	239	295	282	283	260	244
Durable press rating (tumble dry/spin dry)	3.6/3.3	3.4/3.1	3.2/2.8	3.1/2.3	2.7/2.2	3.6/3.2	3.4/3.1	3.4/3.1	3.1/2.9	3.1/2.5
Damage caused by retained chlorine (percent loss in strength)	Nil	Nil	6.7	4.1	Nil	Nil	4.5	2.8	6.5	9.0

TABLE VIII.—FABRIC PERFORMANCE PROPERTIES AT A CURE TIME OF 15 MINUTES AT 340° F.

Property	Treatment									
	Ex. 27a	Ex. 28a	Ex. 29a	Ex. 30a	Ex. 31a	Ex. 32a	Ex. 33a	Ex. 34a	Ex. 35a	Ex. 36a
Yellowness index	.135	.099	.076	.092	.099	.012	.075	.071	.077	.084
Tear strength (warp/fill) grams	432/272	448/304	544/336	656/400	672/432	580/315	505/256	630/363	630/425	752/452
Tensile strength (fill) pounds	16	18	20	22	25	19	17	21	22	25
Dry wrinkle recovery (warp-fill) degrees	284	292	280	273	266	296	291	285	289	274
Durable press rating, tumble dry/spin dry	3.6/3.3	3.5/3.2	3.5/3.2	3.4/3.1	3.4/3.0	3.5/3.2	3.4/3.2	3.4/3.2	3.4/3.1	3.1/3.0
Damage caused by retained chlorine, percent loss in strength	Nil	2.9	5.0	2.5	Nil	Nil	4.8	9.8	Nil	Nil

Although the higher the concentration of the sodium borates in the treating bath the higher the strength properties of the fabric, the optimum concentration in relation to fabric discoloration is 0.5 weight percent. At this concentration, the minimum amount of fabric discoloration is obtained.

Effect of Catalyst Concentration On The Fabric Performance Properties of Fabric Treated With A Glyoxal-Based Durable Press Finish In The Presence of Sodium Tetraborate

White cotton broadcloth desized, scoured, bleached and mercerized was padded (Examples 37 through 40; summarized in Table IX) through a laboratory padder at a wet pick-up of 75 percent. The fabrics were then placed on pin tenter frames, dried at 300°F. for 1.5 minutes and cured for 1.5 and 15.0 minutes at 340°F. in a laboratory oven. "Permafresh 183" was the commercial glyoxal-based curable press finish used in these experiments.

Examples 37*a*–40*a*, showed the effect of catalyst concentration in the presence of sodium tetraborate. Fabric performance properties are summarized in Tables X and XI below.

TABLE IX

TREATING BATH FORMULATION

	EXAMPLES			
	37	38	39	40
Distilled Water	77.8	76.8	75.8	73.8
Permafresh 183	17.8	17.8	17.8	17.8

Tergitol 15-S-9	0.1	0.1	0.1	0.1
Mykon SF	1.8	1.8	1.8	1.8
Sodium Tetraborate	0.5	0.5	0.5	0.5
Modified Zinc Nitrate Catalyst (catalyst X-4)	2.0	3.0	4.0	6.0

TABLE X.—FABRIC PERFORMANCE PROPERTIES AT A CURE TIME OF 1.5 MINUTES AT 340° F.

Property	Treatment			
	Ex. 37a	Ex. 38a	Ex. 39a	Ex. 40a
Yellowness index	.026	.022	.030	.038
Tear strength (warp/fill) grams	944/768	800/656	704/512	608/448
Tensile strength (fill) pounds	37	32	25	23
Dry wrinkle recovery (warp-fill) degrees	248	260	280	295
Durable press rating tumble dry/spin dry	2.5/2.4	3.0/2.8	3.4/2.9	3.4/3.2
Damage caused by retained chlorine, percent loss in strength	3.5	Nil	12.5	Nil

TABLE XI.—FABRIC PERFORMANCE PROPERTIES AT A CURE TIME OF 15 MINUTES AT 340° F.

Property	Treatment			
	Ex. 37a	Ex. 38a	Ex. 39a	Ex. 40a
Yellowness index	.067	.053	.059	.130
Tear strength (warp/fill) grams	672/432	608/464	544/384	480/336
Tensile strength (fill) pounds	27	26	23	19
Dry wrinkle recovery (warp-fill) degrees	280	280	290	294
Durable press rating tumble dry/spin dry	3.2/2.9	3.3/2.9	3.4/3.0	3.3/3.2
Damage caused by retained chlorine, percent loss in strength	Nil	3.7	1.0	Nil

The use of the boron salts slightly effects the effectiveness of the catalyst. The slight loss in catalyst efficiency can be overcome by increasing the catalyst concentration by approximately 1 percent. The data indicated that 4 percent by weight of the catalyst is optionally employed. This will, of course, differ with different treating bath compositions and different glyoxal-based durable press finishes.

What is claimed is:

1. In the process for crease-proofing cellulose-containing textile fabric wherein the reaction product of glyoxal, urea and formaldehyde is applied to said fabric in an acidic medium and thereafter cured to impart crease-resistance to said fabric, the improvement in making said reaction product comprising adding sodium metaborate to an aqueous solution of glyoxal, said solution also containing formaldehyde and in the proportions of 1 mole of glyoxal to 2 moles of formaldehyde, adjusting the pH of said solution to pH 5.5, prior to forming said reaction product, and then adding proportionately 1 mole of urea to said solution and changing the pH of the solution of pH7 which is maintained during the formation of said reaction product, said sodium metaborate being added to said glyoxal in an amount ranging between about 0.5 and 20 weight percent based on the weight of the total solution.

2. The process of claim 1 wherein said sodium metaborate is added in an amount ranging between about 6 and 14 weight percent.

3. A process according to claim 1 wherein said textile fabric is a blend of polyester and cotton.

4. A process according to claim 1 wherein said textile fabric is cotton.

5. A process according to claim 1 wherein said textile fabric is rayon.

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