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[54] **BUFFERED ALDEHYDE FIXATION
COMPOSITION**
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ABSTRACT: The process of fixing formaldehyde on cellulose or a cellulose ester with the aid of a carbamate and using glycolic acid in the catalyst system is improved by including a buffering agent. Preferably there is also included a hexitol as a scavenger for unreacted formaldehyde.

BUFFERED ALDEHYDE FIXATION COMPOSITION

The present invention is directed to an improvement in the process of fixing an aldehyde on cellulose using a carbamate carrier as disclosed in Cotton et al. application 451,033 filed Apr. 26, 1965 now U.S. Pat. No. 3,420,696, issued Jan. 7, 1969.

As set forth in Cotton et al. the fixation is normally carried out using an acid catalyst. Glycolic acid works well in the Cotton et al. procedure but unfortunately has a tendency to deteriorate cellulosic materials such as cotton for example.

Accordingly it is an object of the present invention to develop an improved procedure for utilizing glycolic acid as the acid catalyst in the fixation of an aldehyde on a cellulosic material.

Another object is to prepare cellulose containing fabrics which retain their wash and wear properties to an outstanding degree.

A further object is to scavenge unreacted formaldehyde from the treated cellulosic fabric.

Still further objects and the entire scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, however, that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

It has now been found that these objects can be attained by treating cellulose or a cellulose ester having residual hydroxyl groups with an aqueous mixture of formaldehyde and a carbamate together with glycolic acid and a buffer to control the acidity of the solution. To scavenge the formaldehyde there can be employed a hexitol, pentitol or other polyol. The addition of dimethyl sulfone further aids in controlling the acidity of the finished fabric and aids in odor control.

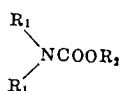
As stated above it is critical that the system contain a buffer. Glycolic acid is known to self condense to form polyglycolides of reduced acidity. However, when glycolic acid is used in the Cotton et al. process without buffering, the fabric becomes highly acidic on standing as moisture is absorbed. Pickup of moisture apparently allows hydrolysis of the polyglycolides to glycolic acid. This hydrolysis appears to be self-catalyzing. The use of the buffers prevents the generation of acidity on standing.

The cellulose can be in the form of cotton, alpha cellulose, regenerated cellulose or rayon, e.g. cuprammonium rayon or viscose rayon, or paper. As cellulose esters, there can be employed cellulose acetate, e.g. 38 percent acetate content, cellulose acetate butyrate and cellulose acetate-propionate.

The cellulose material can be blended with synthetic fibers such as polyesters, e.g. polyethylene terephthalates, acrylic fibers, e.g. polyacrylonitrile, acrylonitrile-vinyl chloride (85:15 or 15:85), nylon, e.g. polymeric hexamethylene adipamide or polymeric caprolactam, polypropylene, ethylenepropylene copolymer, spandex (polyurethane fibers), vinyl chloride-vinyl acetate (e.g. 87:13). A particularly preferred blend is either cotton or viscose rayon with polyethylene terephthalate, for examples blends of 50:50, 65:35, 35:65, 70:30 and 80:20 (the percent of cellulosic material being set forth first).

The formaldehyde source can be paraformaldehyde, trioxane, aqueous formaldehyde or hexamethylene tetramine. Paraformaldehyde is preferred since it is easier to handle than a formaldehyde solution.

As the carbamate there can be employed materials having the formula



where R_1 is hydrogen, hydrocarbyl, e.g. alkyl or carbocyclic and R_2 is hydrocarbyl, e.g. alkyl or aryl or hydroxy lower alkyl or lower alkoxy lower alkyl. Both R_1 groups can be employed methyl carbamate, ethyl carbamate, propylcarbamate, isopropyl carbamate, butyl carbamate, amyl carbamate, hexyl carbamate, isooctyl carbamate, octyl carbamate, decyl carbamate, isodecyl carbamate, dodecyl carbamate, cyclohexyl carbamate, octadecyl carbamate, phenyl carbamate, o-tolyl carbamate, p-tolyl carbamate, m-tolyl carbamate, p-butylphenyl carbamate, 2-naphthyl carbamate, beta-naphthyl carbamate, 2,4-xylyl carbamate, N-phenyl isopropyl carbamate, N-phenyl phenyl carbamate, N-p-tolyl ethyl carbamate, N-phenyl methyl carbamate, N-phenyl ethyl carbamate, N-methyl phenyl carbamate, N-ethyl phenyl carbamate, N-methyl methyl carbamate, N-methyl ethyl carbamate, N-methyl decyl carbamate, N-ethyl methyl carbamate, N-ethyl ethyl carbamate, N-dodecyl methyl carbamate, N-butyl cyclohexyl carbamate, N,N-diethyl ethyl carbamate, N,N-dimethyl ethyl carbamate, N,N-diethyl methyl carbamate, N,N-diphenyl methyl carbamate, 2-hydroxyethyl carbamate, 3-hydroxypropyl carbamate, 2-hydroxypropyl carbamate, 2-hydroxybutyl carbamate, 4-hydroxybutyl carbamate, methoxyethyl carbamate, ethoxyethyl carbamate, propoxyethyl carbamate, methoxypropyl carbamate. Of course mixtures of carbamates can be employed, e.g. the eutectic mixture of 52 percent ethyl carbamate and 48 percent methyl carbamate. The preferred carbamates are methyl carbamate and ethyl carbamate.

The temperature of heating the product in order to fix the formaldehyde to the cellulosic material can be widely varied, e.g. from 125° to 400° F. Temperatures of 180° to 300° F. are usually employed but the temperature is not critical. The use of a partial vacuum is recommended when drying and curing at temperatures in the order of 125° F.

Normally there is employed at least 0.1 percent, and more preferably at least 0.3 percent by weight of methyl carbamate, for example in the aqueous treating solution or dispersion, or equivalent molar percentage of other carbamates. Desirably at least 0.5 percent of methyl carbamate is employed. Higher amounts of carbamate, e.g. 1 to 5 percent or more of methyl carbamate in the aqueous mixture can be used but normally the improvement obtained by using over 1 percent of the carbamate does not justify the increase in cost.

Unless otherwise indicated all parts and percentages are by weight.

The aldehyde is employed in the aqueous system in an amount normally between 1 and 8 percent thereof although as much as 10 or 15 percent or more of formaldehyde can be used if relatively large amounts of formaldehydes are to be fixed onto the cellulose. Desirably the formaldehyde is employed in an amount of at least 2 moles and preferably at least 3 moles per mole of carbamate and can be employed in an amount of at least 3 moles per mole of carbamate and can be employed in an amount as much as 60 moles or even 100 moles per mole of carbamate.

When treating cotton, alpha cellulose and paper there is usually employed an aqueous mixture containing 1.25-4 percent formaldehyde, in order to fix 0.25-1.25 percent formaldehyde, on the treated material. When treating rayon and cellulose esters there usually is employed an aqueous mixture containing 2.5-8.0 percent formaldehyde, in order to fix 0.5-2.5 percent formaldehyde, onto the treated material.

The glycolic acid bath through which the cotton or other cellulosic material is passed generally contains a water soluble polyvalent metal salt catalyst as well to accelerate the reaction of the formaldehyde and cellulose although such salts can be omitted. Typical examples of such salts are magnesium chloride, calcium chloride, zinc nitrate, zinc chloride, zinc fluoborate (which gives excellent scorch protection), zinc silicofluoride, magnesium nitrate, magnesium fluoborate, aluminum chloride, aluminum bromide, magnesium sulfate, aluminum sulfate, potassium aluminum sulfate, paper maker's alum, zinc bromide, magnesium bromide, zinc iodide, magnesium iodide, zinc fluoride, zirconium oxychloride, zirconi-

um oxybromide, titanium tetrachloride, titanium tetrabromide, zinc sulfate, calcium sulfate, barium chloride, strontium chloride, barium bromide, chromic chloride, ferric chloride, ferric sulfate, cupric chloride, ferric bromide, chromic sulfate, cobaltic chloride, nickelous chloride, stannic chloride. Only a small amount of such salts is normally used.

The normal procedure for applying the formaldehyde and carbamate containing aqueous mixture to the material is to pass a fabric, fibers, sheet or continuous yarn through the aqueous mixture, and then to run the thus impregnated material through squeeze rolls to remove excess solution. In the case of yarn, the procedure employed can be to pass the aqueous mixture through packages of the yarn in a kier.

Of course there can be added to the aqueous mixture conventional additives such as wetting agents, hand modifier, softeners, lubricants, brighteners, and the like.

The process fixes formaldehyde on the base material, for example on cotton yarn or fabric with considerable reduction in loss of strength as compared with conventional resin finishing processes. Drying need not be carried to the end point of zero moisture and excellent results are obtained with drying to a residual moisture content of 2-4 percent measured with a resistance type moisture measuring device. Of course the fabric can be bone dried if desired.

The process of the present invention imparts better whiteness retention to cellulosic fabrics, e.g. viscose rayon and cotton fabrics and fabrics containing blends of synthetic and cellulosic fibers. Greatly reduced swelling properties are also imparted to cellulosic fabrics either alone or blended with synthetic fibers. The reaction occurs rapidly so that the cellulosic fibers are not collapsed or highly swollen but are in their normal state. If the fibers were collapsed before the curing with the formaldehyde there would be a reduction in regain and an embrittlement of the fibers.

The present treatment also eliminates the chlorine pickup encountered when cellulosic fabrics are treated with aminoplasts including formaldehyde-carbamate resins to bond nitrogen to the cellulose through methylene bridges.

The cellulose fabrics treated according to the invention are extremely durable to laundering and retain wash-wear properties for extended periods of time.

As the hexitol or pentitol formaldehyde scavenger there can be used mannitol, sorbitol, arabitol, xylitol, heptitols, rhamnitols, or mixtures such as Sutro 170-D which is hydrogenated invert sugar. This latter mixture has the same effect as mannitol and a much greater effect than sorbitol yet costs about one-tenth as much as mannitol. Commercially available polyols may also be used.

Conventional surfactants can be added to get good penetration of the fabrics.

The preferred buffer system contains monosodium phosphate and trisodium phosphate. The ratio of monosodium phosphate to trisodium phosphate can range from 1:1 to 10:1. A portion of the monosodium phosphate, e.g. up to 50 percent, can be replaced by disodium phosphate. When employing paraformaldehyde it is desirable to employ the trisodium phosphate but when employing formalin disodium phosphate can be used to replace the trisodium phosphate. There can also be employed tetrasodium pyrophosphate and sodium tripolyphosphate but they are not as effective as the mixture of monosodium phosphate and trisodium phosphate. The corresponding potassium compounds can be used in place of the sodium compounds. Thus there can be used monopotassium phosphate, dipotassium phosphate and tripotassium phosphate.

Sodium bisulfite can be employed to reduce the formaldehyde odor and to aid in preventing yellowing. It is employed in an amount of from 0.25 percent of the aqueous mix up to one part for two parts of formaldehyde. As stated it can be omitted, particularly with industrial fabrics which do not require as outstanding properties as better quality fabrics. In place of sodium bisulfite there can be used potassium bisulfite, sodium sulfite, potassium sulfite, sodium metabisulfite, sodium bisulfite-acetone.

When tetrasodium pyrophosphate is employed there is a tendency to precipitate magnesium pyrophosphate from the mix if magnesium salts, e.g. magnesium chloride, are employed unless the tetrasodium pyrophosphate and glycolic acid are added prior to the magnesium salts.

While the buffer is employed to increase the pH, normally it is not raised above about a pH of 7. When dimethylsulfone is employed to bring the pH from the acid side up to 7, it is used in an amount of 0.1 to 5 percent of the total mix. The use of higher amounts is not precluded but is wasteful of an expensive material. For one type of fabric, when impregnated fabric is dried in a tenter, the first 25 seconds is for drying and only the last 5 seconds are required for the formaldehyde reaction. Ammonium salts, e.g. ammonium chloride, can be used in place of the metal salt.

Based on the total weight of the aqueous mixture the following table gives the usual range of materials:

TABLE 1

Trisodium phosphate (as the decahydrate, percent)	0. 1-0. 75
Paraformaldehyde, percent	1. 5-15
Monosodium phosphate, percent	0. 1-1
Sodium bisulfite (can be omitted)	0. 25
to 1/2 of the formaldehyde	
Sutro 170-D or other hesitol (can be omitted), percent	1-8
Surfactant (can be omitted) percent	0. 1-1
Softener (varied to get desired hand, can be omitted), percent	1-8
Dimethyl sulfone (can be omitted), percent ..	0. 1-5
Carbamate (e.g. methyl carbamate), percent ..	0. 1-5
Glycolic acid, percent	0. 1-1. 5
Salt (e. g. magnesium chloride, can be omitted), percent	0. 1-2. 5

Higher amounts of glycolic acid and the salt can be added but no extra advantage is gained to justify the increase in cost.

The surfactants and softeners used in the examples below are identified as follows.

Surfactant FW is a wetting agent of the ethylene oxide condensate type. It can be replaced by an equal weight of nonylphenol-ethylene oxide condensate having 10 ethylene oxide units in any examples in which Surfactant FW is employed. Any nonionic or anionic surfactant can be used which doesn't precipitate the salt, e.g. magnesium chloride is used.

Mykon SF is polyethylene emulsified in water, 30 percent solids.

Finish No. 4 is a softener emulsion of a higher fatty acid ester made by Proctor and Gamble. It can be replaced by glycerol monostearate.

Except as noted, the process is normally carried out as a one step operation.

Appearance ratings are according to the American Association of Textile Chemists and Colorists (AATCC) Test Method 88-A-1964T-Procedure III C-1 wherein 1 is the poorest appearance.

EXAMPLE 1

The procedure employed was to pad the aqueous mixture on the white twill cotton fabric (8 oz./sq. yd.), dry at 270° F. for 4 minutes and evaluate for wash-wear properties. The appearance, shrinkage, nitrogen and formaldehyde were measured after five home launderings. All of the mixes had a pH between 2.4 and 2.8. Under each mixture number is the parts of carbamate employed.

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TABLE 2

Sample Number														
Carbamate	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Methyl	1	2	3											
Methoxyethyl				1	2	3								
Hydroxypropyl					1	2	3							
Hydroxyethyl								1	2	3				
Hydroxypropyl)											1	2	3	
Hydroxyethyl)													1	2

Each mix also contained 4 parts formaldehyde (added as formalin originally), 0.83 part glycolic acid, 0.83 part magnesium chloride, 0.75 part sodium bisulfite-acetone adduct (made from 120 grams acetone in 1,500 grams of water and slowly adding 200 grams of sodium bisulfite), 0.75 part tetrasodium pyrophosphate and 0.2 part Surfactant FW (nonionic wetting agent). Sufficient water was added to make 100 parts.

TABLE 3

Sample	Percent fixed formaldehyde	Percent nitrogen	Appearance	Percent warp shrinkage	Grab breaking strength (lbs./in.) warp times filling
Control	0.08	1.0		7.1	168X100
1	0.64	0.13	4.6	2.2	109X50
2	0.72	0.17		2.3	104X54
3	0.76	0.21		2.2	108X58
4	0.70	0.11	4.5	2.3	100X51
5	0.72	0.15		2.1	110X54
6	0.76	0.17		2.0	108X58
7	0.74	0.12	5.0	1.7	106X56
8	0.82	0.17		1.9	108X58
9	0.86	0.21		1.8	108X58
10	0.76	0.12	4.6	2.0	104X54
11	0.80	0.19		1.9	106X63
12	0.84	0.20		1.9	105X61
13	0.70	0.12	4.6	2.1	102X54
14	0.76	0.16		2.0	108X55
15	0.80	0.21		2.0	105X63

EXAMPLE 2

The fabric employed was polyethylene terephthalate-viscose rayon. The formulation employed was as follows:

TABLE 4

Component	Weight/200 gal. mix	Concentration weight, percent
Trisodium phosphate	3.5	10.2
Paraformaldehyde	100	6.0
Monosodium phosphate	8.5	0.5
Sodium bisulfite	12.5	0.75
Dimethyl sulfone	4.0	0.25
Methyl carbamate	16.5	1.00
Glycolic acid, 70%	17.5	2.0
30% magnesium chloride	53	3.00
Sutro 170 D	50	3.00
Surfactant FW	3.5	0.2
Mykon SF	33	2.0
Procter & Gamble Finish No. 4	33	2.0
Water sufficient to make 200 gallons.		

¹ As decahydrate.

² As glycolic acid.

³ As MgCl₂.

The mix was prepared as follows. 50 gallons of water was heated to 160° F. Then the trisodium phosphate decahydrate was dissolved therein. The paraformaldehyde was added at 160° F. The mixture was diluted to 100 gallons and then the monosodium phosphate, sodium bisulfite, dimethyl sulfone, methyl carbamate, glycolic acid, magnesium chloride, Sutro 170-D added in order with stirring. The mixture was diluted to 180 gallons and temperature brought to 100°-110° F. Surfactant FW, Mykon SF and Procter and Gamble Finish No. 4 was added with stirring. The mixture was diluted to 200 gallons.

The mixture is applied to the fabric and the fabric dried at a frame temperature of about 330° F. The fabric temperature is about 240°-280° F. The amount of formaldehyde fixed was about 0.8 percent.

EXAMPLE 3

The procedure of example 2 was repeated using polyethylene terephthalate/cotton blend (50:50) sheeting. The amount of paraformaldehyde was reduced to 60 pounds (3.3 percent) but all other components were unchanged except for the use of slightly more water to bring the mix to 200 gallons.

EXAMPLE 4

65 percent polyester (polyethylene terephthalate) 35 percent cotton broadcloth shirting fabrics were passed through the following mix in which percentages are by weight of the total mix.

TABLE 5

Polyvinyl alcohol	0.5%
Tetrasodium pyrophosphate	0.38%
Formaldehyde	5.00%
Methyl carbamate	1.00%
Sodium bisulfite	0.75%
Glycolic acid	0.83%
Magnesium chloride	0.83%
Nonylphenol-ethylene oxide condensate (wetting agent)	0.25%
Polyethylene emulsion (30% solids, softener)	3.00%
Rhoplex E-32 (ethyl acrylate, methyl methacrylate, acrylic acid, acrylamide tetrapolymer) emulsion	2.20%
Water	balance

In a continuous operation the fabrics were padded through the mix and dried in a tenter dryer under the following conditions.

Dryer temperature	318°-338° F.
Operating speed	100 yards/min.
Total dwell time in drying	15 seconds
Fabric temperature at the exit end of the dryer as measured with an optical pyrometer	255°-260° F.

Portions of fabric were taken at this point for testing. They are designated with an A in the table below. The balance of the fabrics were given an alkaline process wash, dried and then treated in the following mix.

Borax	0.5%
Sodium bisulfite	1.5%
Zinc fluoborate	1.0%
Ethylene oxide condensate (wetting agent)	0.25%
Fatty ester dispersion (softener)	3.0%
Water	balance

The fabric was padded through the mix and dried in a tenter dryer. Conditions for this application were

Dryer temperature	250°-260° F.
Operating speed	110 yards/min.
Total dwell time in drying	14 seconds
Fabric temperature at the exit end of the dryer	160° F.

The purpose of this latter treatment was to provide a soft, supple hand to the fabric and to provide an agent which would enhance the creasing of the fabric along seams when pressed after garment manufacture. As a final operation this fabric was compressively shrunk. The fabric given the additional processing is designated by the letter B in the table below

TABLE 6

Sample	Percent nitrogen as finished	Percent nitrogen after 5 launders	Percent formaldehyde after 5 launders	Molar ratio of formaldehyde to nitrogen after laundering
1A.....	0.09	0.05	0.56	5.2
1A ¹		0.03	0.62	9.7
1B.....	0.08	0.07	0.64	4.3
1B ¹		(²)	0.64	Infinite
2A.....	0.08	0.03	0.58	9.1
2A ¹		0.07	0.66	4.4
2B.....	0.09	0.02	0.62	14.4
2B ¹		0.02	0.58	13.5

¹ Indicates the sample was pressed in a garment press for 15 seconds including 5 seconds steaming, 5 seconds.

² None found.

heating at a steam pressure of 100 p.s.i.g. and 5 seconds vacuum extraction.

Shirts were made from samples 2A and 2B. After being laundered 50 times the shirts were analyzed for nitrogen and formaldehyde. For comparison a Manhattan Shirt Company 65 percent polyester (polyethylene terephthalate), 35 percent cotton broadcloth shirt indicated to be treated with a carbamate-formaldehyde precondensate was analyzed after 50 launders.

TABLE 7 (after 50 launders)

Sample	% Nitrogen	% Formaldehyde	Formaldehyde to Nitrogen ratio
2A	0.04	0.58	6.8
2B	0.04	0.58	6.8
Manhattan	0.44	1.30	1.38

EXAMPLE 5

The fabric employed was a cotton twill dyed and prepared for treatment. The mixes employed were as follows. The solutions of materials employed to make the mix, e.g. 10 percent methyl carbamate, were solutions in water.

TABLE 8

Compound	Base Mix	Post Catalyst Mix				
		A	B	C	D	E
10% methyl carbamate	100	25	31	38	44	50
37% formaldehyde	108	27	34	41	47	54
30% magnesium chloride	28	28	28	28	28	28
10% glycolic acid	83	83	83	83	83	83
10% sodium bisulfite	75	75	75	75	75	75
5% tetrasodium pyrophosphate	75	75	75	75	75	75
10% Surfactant FW	20	20	20	20	20	20
Rhoplex E-32	22					

Water to make 1,000 parts in all mixes

The procedure was to pad the base mix on at room temperature and vacuum extract. Then the fabric was dried in a 250° F. oven to obtain a fabric temperature of 160°-170° F. and then the fabric was cured at 230° F. for 20 minutes. Next it was given an X-0² (sodium meta bisulfite treatment as set forth in Waddle U.S. Pat. 2,870,041) treatment at 180° F., washed twice at 140° F. and dried in an oven at 250° F., washed twice at 140° F. and dried in an oven at 250° F. to a fabric temperature of 160°-170° F. Next the post catalyst mix was applied, the fabric vacuum extracted and dried in a 250° F. oven to a fabric temperature of 160°-170° F.

The treated fabrics along with a control (untreated) sample of the fabric were then tested. The results are set forth below.

TABLE 9

Sample	Appearance rating	Crease rating	Percent shrinkage, warp	Grab strength (lbs.) after 5 home launderings
Control.....	2.7	1.0	6.25	150X139
A.....	4.6	3.9	1.8	105X95
B.....	4.5	4.2	1.8	100X102
C.....	4.6	4.6	1.8	99X97
D.....	4.2	4.3	1.8	103X98
E.....	4.3	4.1	2.0	108X105

EXAMPLE 6

Using several different buffers, 9 inches warpwise strips cut from 48 inches 1.39 yd./lb. cotton twill fabric was padded with the mixes and vacuum extracted, dried and cured in an oven at 330° F. 1,000 4 minutes.

All of the samples contained 100 grams of 10 percent methyl carbamate in water and 108 grams of 37 percent aqueous formaldehyde. All samples also contained sufficient water to make the samples up to 1000 grams. Samples 1-15 contained 32 grams of 30 percent magnesium chloride and sample 22 contained 28 grams of 30 percent magnesium chloride. Samples 16-19 contained 28 grams of 30 percent magnesium chloride. Samples 16-19 contained 15 grams of zinc nitrate hexahydrate, samples 20 and 21 contained 120 grams of 5 percent aqueous zinc fluoborate, samples 1-21 contained 76 grams of 10 percent glycolic acid and sample 22 contained 83 grams of 10 percent glycolic acid, samples 1-10, 15-18, 20 and 21 contained 78 grams of 10 percent aqueous sodium bisulfite and sample 22 contained 75 grams of 10 percent aqueous sodium bisulfite. The samples also contained the ingredients set forth in the table and the indicated properties.

TABLE 10

Sample	Ingredient	Amount (grams)	Mix pH	Fabric pH
1.....			1.6	4.0
2.....	5% aqueous TSPP.....	128	2.6	4.5
3.....	5% Na ₃ PO ₄	82	2.2	4.2
4.....	5% sodium tripolyphosphate.....	180	2.7	4.5
5.....	5% sodium hexametaphosphate.....	337	2.3	4.2
6.....	5% borax (Na ₂ B ₄ O ₇ ·10H ₂ O).....	186	3.2	5.0
7.....	2.5% boric acid.....	62	1.7	3.9
8.....	5% sodium metasilicate.....	51	2.2	4.0
9.....	5% sodium silicate (commercial).....	89	2.1	4.1
10.....	5% NaOH.....	20	2.4	4.3
11.....	5% Na ₃ PO ₄	164	2.8	4.4
12.....	2.5% boric acid.....	248	1.6	3.9
13.....	5% sodium tripolyphosphate.....	180	2.7	4.3
14.....	5% NaOH.....	40	2.7	4.4
15.....	2.5% boric acid.....	248	1.7	3.9
16.....	5% sodium tripolyphosphate.....	180	2.9	4.1
17.....	2.5% boric acid.....	62	1.85	4.1
18.....	5% sodium metasilicate.....	51	2.35	4.5
19.....	5% NaOH.....	40	2.8	5.0
20.....	2.5% boric acid.....	62	2.2	3.5
21.....			2.4	4.2

TABLE 11

Sample	Percent formaldehyde		Warp shrinkage, percent	Grab strength (lbs.) after 5 launders
	Original	after 5 launders		
Control.....			6.78	177X122
1.....	0.78	0.67	0.89	86X45
2.....	0.78	0.57	1.44	98X59
3.....	0.79	0.64	1.17	98X51
4.....	0.72	0.55	1.44	109X64
5.....	0.67	0.53	1.94	115X68
6.....	0.71	0.44	1.94	122X68
7.....	0.78	0.67	0.83	77X43
8.....	0.84	0.59	0.95	96X63
9.....	0.10	0.61	0.72	82X51
10.....	0.71	0.53	1.11	101X61
11.....	0.92	0.73	0.47	74X61
12.....	0.68	0.21	0.67	64X35
13.....	0.78	0.67	1.06	77X46
14.....	0.48	0.71	0.95	73X43
15.....	0.78	0.71	1.00	83X46
16.....	0.70	0.28	2.66	149X84
17.....	0.62	0.39	1.89	118X72
18.....	0.72	0.38	2.00	123X79
19.....	0.78	0.52	1.33	110X63
20.....	0.72	0.28	2.66	153X92
21.....	0.92	0.62	1.11	100X50

EXAMPLE 7

The following formulations were made and the buffered solutions prepared were padded on 65 percent polyester, 35 percent cotton twill fabric and dried at 350° F. for 1.5 minutes.

Material	Sample																	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
37% formaldehyde.....	217	217	217	217	217	217	217	217	217	217	217	217	217	325	217	217	217	217
Methyl carbamate.....	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20
10% NaHSO ₃	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150
Sutro 170-D.....	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60
10% glycolic acid.....	100	100	100	100	100	80	60	60	60	60	60	60	60	60	60	60	60	60
MgCl ₂ , 30%.....	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56
10% NaH ₂ PO ₄	100	80	60	40	20	40	40	40	40	40	40	40	40	40	40	40	40	40
10% surfactant FW.....	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40

TABLE 12

Material, grams:	1	2	3	4	5	6	7	8	9	20
37% formaldehyde.....	217	217	217	217	217	217	217	217	217	217
Methyl carbamate.....	20	20	20	20	20	20	20	20	20	20
10% sodium bisulfite.....	350	350	350	350	350	350	350	350	350	350
Mannitol.....	60	60	60	60	60	60	60	60	60	60
10% glycolic acid.....	166	166	166	166	166	166	166	166	166	166
10% magnesium chloride.....	56	56	56	56	56	56	56	56	56	56
10% NaH ₂ PO ₄	50	50	100	50	100	100	100	100	100	100
10% surfactant FW.....	50	50	50	50	50	50	50	50	50	50
50% sorbitol.....										
Water balance to make 2,000 grams in each of samples 1-9.										

TABLE 13

Sample	Percent formaldehyde after 5 launders	Fabric pH	Colorimetric odor rating (AATCC 113-1965T)	Percent warp shrinkage
1.....	0.56	5.9	0.5	1.3
2.....	0.60	5.7	1.0	1.2
3.....	0.60	6.2	0.5	1.2
4.....	0.62	4.5	1.0	1.3
5.....	0.60	4.4	1.5	1.1
6.....	0.60	4.7	2.5	1.2
7.....	0.60	4.6	1.0	1.2
8.....	0.70	4.6	2.0	1.0
9.....	0.64	5.4	2.5	1.0

EXAMPLE 8

Sodium hexametaphosphate was employed as a buffer in the following formulations. The mixes were padded on (a) a cotton twill fabric and (b) a 50 percent polyester, 50 percent cotton twill fabric. The cotton twill fabric was dried at 350° F. for 1.5 minutes and the polyester, cotton twill fabric was dried at 350° F. for 2 minutes. To simulate usual procedure for durable press garment manufacture, the treated fabrics were creased by pressing 15 seconds at 120 p.s.i.g. steam pressure in a garment press and heated in an oven at 325° F. for 8 minutes.

TABLE 14

Material, grams:	1	2	3
37% formaldehyde.....	217	217	217
Methyl carbamate.....	20	20	20
10% sodium bisulfite.....	150	150	150
Mannitol.....	60	60	60
10% glycolic acid.....	100	100	100
30% MgCl ₂	56	56	56
10% sodium hexametaphosphate.....	40	100	200
10% surfactant FW.....	50	50	50
Water sufficient to make 2000 grams.....			

TABLE 15

	Cotton twill				Tanker twill			
	Control	1	2	3	Control	1	2	3
Percent hexametaphosphate.....		0.1	0.25	0.40		0.1	0.25	0.50
Appearance (5 launders).....	2.5	3.5	3.5	3.5	3.0	4.0	4.0	4.1
Crease rating.....	1.0	3.0	3.0	3.0	3.5	4.0	3.8	3.8
Fabric pH.....		4.22	4.37	4.38		4.28	4.23	4.35
Fixed formaldehyde (5 launders).....		0.68	0.70	0.68		0.44		0.44
Shrinkage.....	7.39	1.61	1.89	1.89	4.38	2.23	2.28	2.17

EXAMPLE 9

A series of 18 samples were prepared using the formulations in the table. The solutions made up to 2,000 grams were padded on 67 percent viscose rayon, 33 percent cotton tablecloth fabric and dried at 350° F. (oven temperature) for 1.5 minutes to obtain a fabric temperature of 292°-294° F.

TABLE 16

Material	Sample																	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
37% formaldehyde.....	217	217	217	217	217	217	217	217	217	217	217	217	217	325	217	217	217	217
Methyl carbamate.....	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20
10% NaHSO ₃	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150	150
Sutro 170-D.....	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60
10% glycolic acid.....	100	100	100	100	100	80	60	60	60	60	60	60	60	60	60	60	60	60
MgCl ₂ , 30%.....	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56	56
10% NaH ₂ PO ₄	100	80	60	40	20	40	40	40	40	40	40	40	40	40	40	40	40	40
10% surfactant FW.....	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40	40

TABLE 17

Sample:	Mix pH	Fabric pH	Percent formaldehyde fixed	Percent shrinkage
1.....	2.5	4.3	0.94	5.78
2.....	2.5	4.3	0.96	5.56
3.....	2.4	4.3	0.94	5.56
4.....	2.4	4.3	0.88	5.50
5.....	2.3	4.1	0.88	6.23
6.....	2.2	4.3	1.02	6.12
7.....	2.3	4.5	0.96	6.23
8.....	2.4	4.8	0.90	6.39
9.....	2.6	5.1	0.70	8.17
10.....	2.0	4.3	1.14	5.27
11.....	2.1	4.2	1.28	4.38
12.....	2.1	4.1	1.28	4.16
13.....	2.1	4.0	1.48	3.27
14.....	2.2	4.2	1.54	3.94
15.....	2.2	4.2	1.34	4.89
16.....	2.2	4.3	1.14	5.56
17.....	2.2	4.2	1.08	5.38
18.....	2.2	4.1	1.56	4.67
Untreated.....				18.73

EXAMPLE 10

The following mixes (all parts are in grams) were padded on a rayon-cotton tablecloth fabric at room temperature. The fabric was then dried at 330° F. (oven temperature) for 1.5 minutes.

TABLE 18

Material:	Sample			
	1	2	3	4
37% formaldehyde.....	217	217	217	217
Methyl carbamate.....	20	20	20	20
10% sodium bisulfite.....	150	150	150	150
50% sutro 170 D.....	120	120	120	120
10% glycolic acid.....	80	80	80	80
40% magnesium fluoborate.....	49	35	24	12
10% NaH ₂ PO ₄	40	40	40	40
10% surfactant FW.....	40	40	40	40
Water sufficient to make 2000 grams in all samples.				

EXAMPLE 11

The following mixes were padded on a bleached 50 percent polyester 50 percent cotton fabric at room temperature and dried in an oven (a) at 330° F. for 1 minute and (b) at 330° F. for 1.5 minutes. The mixes all contained enough added water to make a total of 2,000 grams of mix.

TABLE 19

	1	2	3	4	5	6	7
Material, grams:							
10% monosodium phosphate.....	100	92	85	77	70	62	92
10% trisodium phosphate.....	76	152	228	304	380	76	
10% sodium bisulfite.....	100	100	100	100	100	100	100
Methyl carbamate.....	20	20	20	20	20	20	20
37% formaldehyde.....	216	216	216	216	216	216	216
Dimethyl sulfone.....	20	20	20	20	20	20	20
10% glycolic acid.....	100	100	100	100	100	100	100
30% MgCl ₂	56	56	56	56	56	56	56
10% surfactant FW.....	50	50	50	50	50	50	50

The results obtained are set forth in the following table.

TABLE 20

Sample:	Mix/pH	330° oven		350° oven	
		Fabric pH	Percent CH ₂ O	Fabric pH	Percent CH ₂ O
1.....	2.0	4.28	0.52	4.79	0.56
2.....	2.2	4.50	0.48	5.03	0.66
3.....	2.2	4.90	0.68	5.10	0.42
4.....	2.4	4.76	0.52	5.30	0.52
5.....	2.3	4.41	0.46	5.47	0.76
6.....	2.5	4.48	0.54	5.40	0.86
7.....	2.2	4.00	0.44	5.19	0.42

In the steam tube test for free formaldehyde in which steam is passed through a fabric sample placed in a stainless steel tube in the manner of AATCC Test method 113-1965T and the effluent steam is smelled, after the 350° F. oven treatment sample 1 showed a strong formaldehyde odor, sample 2 a moderate formaldehyde odor and samples 3-7 a slight formaldehyde odor.

EXAMPLE 12

A series of eight mixes were prepared and 50 percent polyester, 50 percent cotton sheeting padded there through at room temperature and dried at 330° F. for 1 minute.

Each mix contained 80 grams of 10 percent monosodium phosphate, 40 grams of 5 percent trisodium phosphate, 200 grams of 10 percent sodium bisulfite, 20 grams of dimethyl sulfone, 216 grams of 37 percent formaldehyde, 120 grams of 50 percent Sutro 170 D 100 grams of 10 percent glycolic acid, 56 grams 30 percent magnesium chloride, 50 grams of 10 percent Surfactant FW, 20 grams of carbamate and water to make 2,000 grams. The carbamates employed were as follows: (1) methyl carbamate, (2) ethyl carbamates, (3) N-ethyl methyl carbamate, (4) N-ethyl ethyl carbamate, (5) N-butyl carbamate, (6) hydroxyethyl carbamate, (7) hydroxypropyl carbamate, (8) hydroxypropyl-hydroxyethyl carbamate mixture. Except for the mix pH all tests were after 5 home laundings. The results are set forth below.

TABLE 21

Sample	Mix pH	Fabric pH	% CH ₂ O	% Shrinkage
1	2.4	6.08	0.66	1.00
2	2.4	5.80	0.76	1.00
3	2.4	5.29	0.64	1.00
4	2.4	5.30	0.64	0.89
5	2.4	5.90	0.60	0.95
6	2.4	5.91	0.76	0.78
7	2.4	6.20	0.64	0.78
8	2.4	6.13	0.72	0.95
untreated				3.12

EXAMPLE 13

A series of mixes were prepared and 50 percent polyester, 50 percent cotton sheeting padded there through at room temperature and dried at 330° F. for 1 minute.

Each mix contained 80 grams of 10 percent monosodium phosphate, 40 grams of 5 percent trisodium phosphate, 20 grams of dimethyl carbamate, 216 grams of 37 percent formaldehyde, 120 grams of 50 percent Sutro-170-D, 100 grams

of 10 percent glycolic acid, 56 grams of 30 percent magnesium chloride, 50 grams of 10 percent surfactant solution, 10 percent sodium bisulfite and 10 percent dimethyl sulfone (DMSO₂) in the amounts in grams set forth below and water to make 2,000 grams.

TABLE 22

Sample	10% NaHSO ₃	10% DMSO ₂	Mix pH	Fabric pH after drying
1	0	0	2.3	3.90
2	30	0	2.5	3.81
3	60	0	2.7	3.90
4	90	0	2.8	4.06
5	120	0	2.5	4.20
6	150	0	2.4	4.38
7	0	20	2.5	3.91
8	30	20	2.5	3.91
9	60	20	2.5	4.02
10	90	20	2.5	4.07
11	120	20	2.5	4.23
12	150	20	2.5	4.42
13	0	40	2.4	3.90
14	30	40	2.4	4.01
15	60	40	2.4	4.04
16	90	40	2.4	4.01
17	120	40	2.4	4.23
18	150	40	2.5	4.50
19	0	60	2.4	3.87
20	30	60	2.4	3.90
21	60	60	2.4	4.10
22	90	60	2.4	4.07
23	120	60	2.4	4.25
24	150	60	2.4	4.61
25	0	100	2.4	3.88
26	30	100	2.4	3.92
27	60	100	2.7	4.03
28	90	100	2.5	4.18
29	120	100	2.5	4.47
30	150	100	2.4	4.72
31	0	100	2.4	4.00
32	30	200	2.4	4.10
33	60	200	2.4	4.19
34	90	200	2.4	4.22
35	120	200	2.4	4.51
36	150	200	2.4	5.05

EXAMPLE 14

A series of mixes were prepared with varying amounts of 10 percent dimethyl sulfone. The mixes were prepared by diluting 40 grams of 5 percent trisodium phosphate, 50 grams of 10 percent Surfactant FW and 60 grams of paraformaldehyde with 500 grams of water at 140° F. with stirring for 5 minutes. The mixture was cooled by the addition of 500 grams of cold water and then there were added 80 grams of 10 percent monosodium phosphate, 100 grams of 10 percent glycolic acid, 56 grams of 30 percent magnesium chloride, 120 grams of 50 percent Sutro 170-D, the indicated amounts of 10 percent dimethyl sulfone and cold water sufficient to make 2,000 grams. The mix was padded on sheeting which was 50 percent cotton—50 percent Dacron (polyethylene terephthalate) at room temperature and dried at 350° F. in an oven for 1 minute. The results are shown in the following table. The percent shrinkage and percent formaldehyde were measured after five laundings.

TABLE 23

Samples:	10% dimethyl sulfone	Mix pH	Percent shrinkage	Percent CH ₂ O	Fabric pH
1.....	0	2.2	1.05	0.42	5.00
2.....	50	2.2	1.0	0.44	4.90
3.....	100	2.2	0.78	0.44	5.08
4.....	150	2.1	1.0	0.48	5.80
5.....	200	2.2	1.05	0.50	5.46
6.....	250	2.1	1.0	0.46	5.36
7.....	300	2.1	1.11	0.44	5.60
Control.....			4.22		

EXAMPLE 15

A series of runs were made with varying amounts of buffer to raise the pH of the fabric close to neutral. There were employed in the mixes the indicated amount of 5 percent trisodium phosphate, 60 grams of paraformaldehyde, the indicated amounts of 10 percent monosodium phosphate, 150 grams of 10 percent sodium bisulfite, 20 grams of methyl carbamate, 100 grams of 10 percent glycolic acid, 120 grams of 50 percent Sutro 170 D, 50 grams of Surfactant FW and sufficient water to make 2,000 grams. The mixture was padded on 50 percent cotton—50 percent Dacron sheeting at room temperature and the sheeting dried at 350° F. for 1 minute. The pH of the initial mix and fabric pH were measured and the percent shrinkage and percent formaldehyde were measured after five home launders. In this example and the other examples employing trisodium phosphate it was always measured as $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$, e.g. if the example states 40 grams of 5 percent trisodium phosphate was used this would mean that $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$, was dissolved in water to give a 5 percent solution containing 40 grams in all.

TABLE 24

Sample:	5% trisodium phosphate	10% NaH_2PO_4	pH	Percent shrinkage	Percent CH_2O	Fabric pH
1.....	40	80	2.1	0.67	0.48	5.42
2.....	60	120	2.3	1.11	0.45	6.20
3.....	80	160	2.5	1.17	0.40	6.10
4.....	100	200	2.6	1.05	0.41	6.31
5.....	100	50	2.4	1.22	0.48	6.00
6.....	150	75	2.5	1.11	0.44	6.58
7.....	200	100	2.7	1.17	0.41	6.72
8.....	250	125	3.0	1.17	0.40	6.98
Control.....				4.44		

EXAMPLE 16

50 percent Dacron—50 percent cotton sheeting was padded through the mixes set forth below at room temperature and dried at 350° F. for 30 seconds. The mixes contained enough water to make 2,000 grams of mix.

TABLE 25

Material	1	2	3
		Weight	
5% Trisodium phosphate	250	250	250
Paraformaldehyde	60	60	60
10% Monosodium phosphate	125	125	125
10% Sodium bisulfite	150	150	150
10% Glycolic acid	200	200	200
30% Magnesium chloride	112		
38% Zinc nitrate		185	
40% Zinc fluoborate			210
50% Sutro 170 D	120	120	120
10% Surfactant FW	50	50	50
Methyl carbamate	40	40	40

The mix pH and fabric pH were determined as well as percent shrinkage and percent formaldehyde after 5 launders. The results are shown below.

TABLE 26

Sample	Mix/pH	% Shrinkage	% CH_2O	Fabric pH
1	2.0	1.22	0.42	4.90
2	1.9	1.22	0.36	5.30
3	1.8	1.39	0.36	4.00
Control		3.72		

EXAMPLE 17

Mixes were prepared containing 40 grams of 5 percent trisodium phosphate, the indicated amounts of paraformaldehyde or 37 percent aqueous formaldehyde, 80 grams of 10 percent monosodium phosphate, 144 grams of 10 percent

sodium bisulfite, 20 grams of methyl carbamate, 100 grams of 10 percent glycolic acid, 56 grams of 30 percent magnesium chloride, the indicated amounts of 50 percent Sutro 170 D, 50 grams of 10 percent Surfactant FW and water sufficient to make 2,000 grams. The mix was padded on 50 percent cotton—50 percent Dacron sheeting at room temperature and the fabric dried at 350° F. for 1 minute.

TABLE 27

Material	1	2	3	4
	Amount			
Paraformaldehyde	60	60	60	
37% Formaldehyde			163	
50% Sutro 170 D	60	120		

The properties of the mix and finished fabrics are shown below.

EXAMPLE 18

The purpose of this experiment was to determine the minimum level of formaldehyde in the mix to yield 1 percent formaldehyde fixation. The mixes prepared were padded on 50 percent Dacron—50 percent viscose rayon sheeting. All of

TABLE 28

Sample:	Mix pH	As finished			After 5 launders, percent CH_2O
		Percent shrinkage	Percent CH_2O	Fabric pH	
1.....	2.0	1.0	0.60	4.39	0.47
2.....	2.1	1.05	0.67	5.57	0.53
3.....	2.1	0.89	0.57	3.89	0.49
4.....	2.3	1.0	0.68	4.00	0.56
Control.....		3.72			0.41

the mixes were made up to 2,000 grams. Each mix contained 80 grams of 5 percent trisodium phosphate, the indicated amount of paraformaldehyde, 100 grams of 10 percent monosodium phosphate, 150 grams of 10 percent sodium bisulfite, 5 grams of dimethyl sulfone, 20 grams of methyl carbamate, 150 grams of 10 percent glycolic acid, 67 grams of 30 percent magnesium chloride, 120 grams of 50 percent Sutro 170 D, 40 grams of 10 percent Surfactant FW, 48 grams of Mykon SF and 48 grams of Finish No. 4. The mix pH, formaldehyde fixation and fabric pH were recorded. The mix was padded on at room temperature and drying was at 350° F. for 1 minute.

TABLE 29

Sample:	Paraformaldehyde, grams	Mix pH	Percent CH_2O fixed	Fabric pH
1.....	120	2.1	1.43	3.51
2.....	110	2.2	1.62	3.41
3.....	100	2.3	1.34	3.67
4.....	90	2.3	1.40	3.72
5.....	80	2.4	1.03	4.10

EXAMPLE 19

The procedure and mixes were the same as in example 18 but the fabric was 100 percent viscose rayon. Samples 1, 2, 3, 4 and 5 had the same materials and proportions as the samples in example 18. The oven dwell times at 350° F. were varied. In the following table the letter A designates 1 minute dwell, the letter B 1.5 minutes dwell and the letter C 2 minutes dwell.

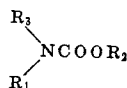
The values were recorded (with the exception of the initial pH of the mix and fabric pH) after 5 launders.

TABLE 30

Sample	Initial pH	% Shrinkage	% CH ₂ O	Fabric pH
1A	2.2	2.00	1.70	3.78
1B	2.2	1.22	2.30	3.86
1C	2.2	0.83	2.30	3.81
2A	2.2	2.27	1.70	3.90
2B	2.2	1.17	2.60	3.92
2C	2.2	1.22	2.40	4.02
3A	2.3	1.67	2.30	3.60
3B	2.3	1.11	2.30	3.92
3C	2.3	1.22	2.40	4.23
4A	2.3	2.56	1.49	3.93
4B	2.3	2.16	1.62	4.20
4C	2.3	2.05	1.70	4.27
5A	2.4	3.22	1.49	4.09
5B	2.4	2.56	1.34	4.12
5C	2.4	2.05	1.50	4.37
Control		17.13		

We claim:

1. In a process of fixing formaldehyde on a hydroxyl containing polymer selected from the group consisting of cellulose, cellulose acetate, cellulose acetate-propionate and cellulose acetate-butyrate by treating said polymer with an aqueous mixture comprising (1) formaldehyde, (2) a carbamate having the formula



where R₁ and R₃ are selected from the group consisting of hydrogen, alkyl and carbocyclic aryl and R₂ is selected from the group consisting of alkyl, carbocyclic aryl, hydroxy lower alkyl and lower alkoxy lower alkyl and (3) glycolic acid, the improvement comprising including in the aqueous mixture a buffer to counteract the tendency of the cellulosic material impregnated with said mixture to become highly acidic on standing.

2. A process according to claim 1 wherein the treating solution includes a water soluble salt of a polyvalent metal capable of catalyzing the reaction between cellulose and formaldehyde.

3. A process according to claim 1 wherein the treating solution includes water soluble sugar alcohol selected from the group consisting of pentitols, hexitols and heptitols.

4. A process according to claim 1 wherein the buffer includes an alkali metal phosphate, the aqueous mixture includes alkali metal bisulfite in an amount sufficient to reduce formaldehyde odor and also includes dimethyl sulfone in an amount of 0.1 to 5 percent to raise the pH.

5. A process according to claim 1 wherein the polymer is in the form of fibers.

6. A process according to claim 1 wherein the buffer in-

cludes an alkali metal phosphate.

7. A process according to claim 6 wherein the phosphate comprises monosodium phosphate.

8. A process according to claim 6 wherein the phosphate comprises disodium phosphate.

9. A process according to claim 5 wherein the fibers are in the form of a fabric.

10. A process according to claim 6 wherein the aqueous mixture includes dimethyl sulfone in an amount effective to raise the pH of the treated polymer.

11. A process according to claim 6 wherein the phosphate comprises trisodium phosphate.

12. A process according to claim 11 wherein the phosphate also comprises monosodium phosphate.

13. A process according to claim 6 wherein the aqueous mixture includes alkali metal bisulfite in an amount sufficient to reduce formaldehyde odor.

14. A process according to claim 13 wherein the bisulfite comprises sodium bisulfite.

15. A process according to claim 13 wherein the aqueous mixture includes dimethyl sulfone in an amount effective to raise the pH of the treated polymer.

16. A process according to claim 15 wherein the aqueous mix also contains mannitol as a formaldehyde scavenger and sodium bisulfite to reduce the formaldehyde odor.

17. A process according to claim 6 wherein the aqueous mix also contains sugar alcohol of the group consisting of pentitols, hexitols and heptitols as a formaldehyde scavenger.

18. A process according to claim 17 wherein the sugar alcohol comprises mannitol.

19. A process according to claim 16 wherein the carbamate is methyl carbamate.

20. A process according to claim 18 wherein the treating mixture comprises 0.1 to 0.75 percent trisodium phosphate calculated as the decahydrate, 1.5 to 15 percent formaldehyde, 0.1 to 1 percent monosodium phosphate, sodium bisulfite in an amount of 0.25 percent to not more than 1 part for each two parts formaldehyde, 0.1 to 5 percent of the carbamate, 0.1 to 1.5 percent of glycolic acid.

21. A process according to claim 1 comprising treating a cellulose fabric with an aqueous mixture of (1) formaldehyde, (2) methyl carbamate or ethyl carbamate, glycolic acid and an alkali metal phosphate buffer.

22. A process according to claim 21 wherein the buffer comprises monosodium phosphate.

23. A process according to claim 22 wherein the buffer includes trisodium phosphate.

24. A process according to claim 22 wherein the aqueous mixture also contains sodium bisulfite, magnesium chloride, dimethyl sulfone and mannitol.

25. A process according to claim 23 wherein the ratio of monosodium phosphate to trisodium phosphate is between 1:1 and 10:1.

26. A process according to claim 25 wherein the treating solution includes 0.1 to 2.5 percent of magnesium chloride.

* * * * *