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3,546,006

WET-FIXATION PROCESS FOR CELLULOSIC FABRICS USING LOW ADD-ONS OF RESINS

Gerald B. Verburg and Alvin H. P. Young, Metairie, and Frank W. Snowden and Sidney L. Vail, New Orleans, La., assigns to the United States of America as represented by the Secretary of Agriculture
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3 Claims

ABSTRACT OF THE DISCLOSURE

The purpose of this invention is to fix or deposit N-methylol resins in cellulosic fabrics by the action of heat and steam to produce abrasion resistant fabrics capable of resisting wrinkling or musing throughout a reasonable wear life. Fixation is accomplished in a rapid one-step process. Fixed fabrics may be cured immediately or may be used in a delayed cure.

A non-exclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

This patent application is a continuation-in-part of our copending application Ser. No. 712,392 (filed Mar. 12, 1968).

Because of their poor resiliency, cellulosic textile fabrics tend to become wrinkled in use and acquire a mussed or unpleasant appearance. To overcome this objectionable property, it has been the practice to treat or finish cellulosic textiles with the methylol derivatives of organic nitrogen compounds, such as urea, ethyleneurea, guanidine, melamine, and the like. These agents produce a finished cellulosic fabric that has improved resistance to wrinkling or musing and give to the fabric the ability to dry smooth after laundering. A serious drawback results since the cellulosic fabrics are damaged during these treatments, thereby reducing the wear life of the treated cellulosic fabrics.

A wet fixation method was devised by workers of the National Cotton Council and Harris Research Laboratories to overcome damage to the cellulosic fabrics during treatment. In this method of treatment, the cellulosic fabrics are impregnated with a solution which deposits resins inside the wet, swollen cellulosic fiber. This is accomplished by padding an N-methylol resin-mineral acid solution on the fabric, heating the fabric in a closed or semi-closed container for 15 minutes at 180° F., neutralizing in sodium carbonate solution, washing, and drying. The heating results in deposition of the resin in the fiber in such a manner that it is not removed by washing and so sustains the fibers in a somewhat swollen condition. Subsequent application of a crosslinking catalyst, followed by a high-temperature dry cure, results in only modest loss of fabric strength as compared with conventional pad-dry-cure processing and a relatively high add-on of resin, i.e., above 8%.

According to the developers of the above wet-fix process, an important factor in arriving at good strength

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properties of the finished fabric and high levels of fabric performance, both in resistance to wrinkling and good crease retention, is the use of two resin components. One of these, called a polymer builder, acts to swell the fibers and keeps them in a swollen state (melamine derivatives are particularly effective in this regard); the second component, primarily a crosslinking agent for cotton, contributes only moderately to the swelling of the fiber but enhances wet wrinkle recovery and, once catalyzed and dried, imparts high dry wrinkle recovery to the fabric (dimethyldihydroxyethylene urea and dimethylol carbamates are good examples of this class).

In mill trials cellulosic fabric is impregnated with the resin solution, made into rolls, sealed, and rotated overnight while wet fixation is taking place. Difficulty has been experienced in achieving good yarn penetration in plant runs. In short, the laboratory procedure of wet fix is not directly applicable to continuous mill runs; in addition, it requires several more steps than the procedures normally used in the prior art to achieve resistance to wrinkling and musing.

For the purposes of the instant invention, the fabric used was a white, 7 oz., cotton twill from Testfabrics, Inc. (#413) unless mentioned otherwise.

In the copending application it was demonstrated that a press could be used to fix resins in the fibers in a much more efficient manner than previously disclosed. Not only were the resins fixed rapidly but it was shown that the so-called one-step padding method had the added advantage that one of the padding, washing, and drying steps was eliminated. In addition, the fixation of resins was carried out over a range of pH values, that is, from pH 2 to 6. However, the padded cellulosic fabric had to be enrobed within a flexible, moisture impervious material or the fabric would become stiff and brittle from what appeared to be polymer formation on the surface.

It has now been discovered that fixation of resins over a range of pH values can be achieved without the use of a plastic bag for an enrobing material. The necessary fixation is achieved by subjecting the padded fabric to steam atmospheres above 100° C. for a few minutes. If the fixation is carried out at pH values above 3, it may not be necessary to wash the fabrics. In fact, under the conditions described in this invention washing of the fixed fabrics would remove a large proportion of the reactants.

There are various opinions concerning the actual chemical reactions and physical changes occurring during fixation of reactants. However, it appears that during the fixation described in this invention only a limited amount of crosslinking of the cellulosic fiber (cotton in this case) occurs. Further, it appears that the reactants which are not involved in the crosslink are attached or polymerized in some manner such that there is little or no formaldehyde vapor evolved from the dry, fixed fabrics. Furthermore, the reactants are fixed in such a manner that on conventional curing, a highly wrinkle resistant fabric is obtained which also possesses a high resistance to abrasion.

The properties of the cellulosic fabrics which were finished by the methods of this invention are illustrated in the following discussion and examples. All parts and percentages described are by weight. The degree of wrinkle resistance in the finished textile is shown by the wrinkle recovery angle, determined by the American Society for Testing Materials Test D1424-56T. The tearing

strength of the fabrics is obtained by the Elmendorf method, American Society for Testing Materials Test D1424-63. The Stoll flex abrasion resistance of the fabrics is determined by the American Society for Testing Materials Test D1175-64T. The laundry tests with simulated trouser cuffs were evaluated after repeated laundry wash-tumble dry cycles. A four-pound load was washed in 18 gallons of water on a normal laundry cycle using a commercial wetting agent as detergent. Water temperature control was set for a hot (approximately 60° C.) wash and warm rinse. After washing, the cuffs were tumble dried for 30 minutes in a gas-heated dryer at about 60° C. Each cuff was then inspected for abrasive damage. The breakage of either a warp or filling yarn was classified as minor damage. The breakage of four yarns in one direction or of both a warp and a filling yarn to form a detectable hole was classified as major damage. Wash-wear ratings were obtained by the Monsanto Overhead Lighting method. Crease retention angles were obtained as described in Textile Industries, 131, No. 1, 116 (1967).

An object of this invention is to provide an improved method for treating or finishing cellulosic textiles whereby the textile is rendered resistant to wrinkling or musing during use and will have improved smooth drying properties after laundering without impairing the wear life of the cellulosic fabric. This was accomplished by padding cellulosic fabrics with solutions of the following compositions:

(1) About from 5 to 10 percent solids of a polymer builder of the methylated methylolmelamine type resin (abbreviated MMM) such as Aerotex 23S and Aerotex M-3, about from 2 to 10 percent solids of a crosslinking resin, dimethyloldihydroxyethyleneurea (Permafresh 183, abbreviated DMDHEU), about 4 percent magnesium chloride hexahydrate and most of the required water. The solution was adjusted to a pH between 4 and 6 with hydrochloric acid as desired and about 2 percent solids of a polyethylene emulsion and the remainder of the water was added. A pH of 6 was usually obtained without the addition of acid.

(2) About from 5 to 10 percent solids of a polymer

builder of the methylolated methylolmelamine type resin, such as Aerotex 23S and Aerotex M-3, about from 2 to 10 percent solids of a crosslinking resin, dimethyloldihydroxyethyleneurea (Permafresh 183), about 1 percent catalyst composed of a 1:1 molar ratio of citric acid to magnesium chloride hexahydrate and about 2 percent solids of a polyethylene emulsion and the required amount of water. No adjustment on pH was made for this solution. The pH was usually about 3.5-4.0.

After padding to about 60-80 percent wet pick-up with the selected treating solution, the wetter fabrics were passed in to a steam filled chamber at a temperature between 100° C. and 140° C. for a period of 1 to 10 minutes. Conditions were selected such that the fabric generally emerged from the chamber with more than 10% moisture. Temperatures between 115° C. and 140° C. for about 1 to 3 minutes are generally preferred. The fixed fabrics were then dried (overdrying avoided at this point) and cured when desired. Fabrics were cured in a forced draft oven or in a steam atmosphere over a range of conditions depending upon the acidity of the pad bath and the catalyst used. Temperatures from 120° C. to 160° C. for 1 to 10 minutes were employed.

Using these general conditions fabrics were treated and some were made into cuffs. Further details on the process and fabric properties are given in Tables I and II. Standards or controls are given in Table III. Magnesium chloride was used as catalyst in Table I and citric acid-magnesium chloride mixed catalyst was used in Table II. Fixation time in the presence of steam for all samples in Tables I and II were the same, i.e., 3 minutes at 138° C. The pH values recorded are approximate. Aqueous solutions of MMM, DMDHEU, and $MgCl_2 \cdot 6H_2O$ generally produce a solution of about pH 6; therefore, all solutions are reported to be this value. No attempt was made in any case to adjust the pH to 6. Dilute hydrochloric acid was used to lower the pH to 4.5 or 4.0. The solutions used to obtain the results reported in Table II were not adjusted in pH. The mixed catalyst was added to give a 1% concentration which generally reduced the pH to 3.5 to 4.0.

TABLE I¹.—ONE-STEP PADDING METHOD OF STEAM WET-FIXATION ON COTTON TWILL (MAGNESIUM CHLORIDE CATALYST)

Treatment	Padbath		Fabric properties					Cuff data		
	Agent concentration, MMM/DMDHEU	pH	Cure (° C./min.)	Conditioned WRA (W+F)	Wet WRA (W+F)	Tearing strength (W) (percent of original)	Stoll flex (W) (percent of original)	Cycles to damage		Crease retention (degrees)
								Slight	Major	
1.....	5/5	6	160/5	292	253	100	70	13	24	38
2.....	5/5	4.5	160/3	280	257	100+	100+	20	30+	42
3.....	7/3	6	160/5	296	279	90	40	14	25	36
4.....	7/3	6	160/8	284	283	-----	-----	13	21	37
5.....	7/3	4	160/3	282	281	90+	80	22	30+	48
6.....	7/3	4	160/5	295	284	80+	50	16	27	41
7.....	7/3	4	160/8	296	285	-----	-----	11	20	39
8.....	10/5	6	160/3	294	269	90	30	15	27	39
9.....	10/5	4.5	160/3	281	275	90	30	14	26	37
10.....	10/5	4.5	160/5	301	272	80	40+	14	19	37
11.....	10/5	4.5	160/8	300	287	-----	-----	9	11	30

¹ All wrinkle recovery angles (WRA) are reported in degrees. Tearing strengths and Stoll flex abrasion values are reported as percent of the original, laundered fabric. These values are reported to the nearest 10% with plus signs to indicate a value up to 5 to be added to the given number, except for the value of 100+ which indicates a value of 5 or more in excess of 100. Properties of the original, laundered fabric are given in Table III. All fabrics were laundered once before their properties were determined except crease retention values for the cuffs were determined after the 30th laundry cycle. A 7 oz. twill fabric was used in all treatments. In the cuff data a value of 30+ indicates that the cuff did not suffer major damage during the 30 cycle laundering tests.

TABLE II¹.—ONE-STEP PADDING METHOD OF STEAM WET-FIXATION ON COTTON TWILL (CITRIC ACID-MAGNESIUM CHLORIDE CATALYST)

Treatment	Agent concentration, MMM/DMDHEU		Cure (° C./min.)	Conditioned WRA (W+F)	Wet WRA (W+F)	Tearing strength (W) (percent of original)	Stoll flex (W) (percent of original)	Cuff data		
								Cycles to damage		Crease retention (degrees)
								Slight	Major	
12.....	5/5		160/5	293	273	100+	60+	20	27	38
13.....	5/5		160/10	300	269	-----	-----	14	20	38
14.....	5/5		140/8	296	251	100+	100+	-----	-----	-----
15.....	7/3		160/5	292	251	100+	80+	16	23	40
16.....	7/3		160/10	287	258	-----	-----	9	20	39
17.....	7/3		140/10	299	239	100+	100+	-----	-----	-----

¹ See footnote number 1 in Table I.

TABLE III¹.—PROPERTIES OF UNTREATED FABRIC AND CONVENTIONAL FINISHES

Treatment	Condi- tioned (W+F)	Wet (W+F)	Tearing strength (W) (percent of original)	Stoll flex (W) (percent of original)	Cuff data		
					Cycles to damage		Crease retention (degrees)
					Slight	Major	
18. Laundered, untreated fabric	160	170	² 1,600	² 900			
19.8% DMDHEU, 4% MgCl ₂ ·6H ₂ O, and 2% polyethylene ³	305	275	80+	20+	7	9	40

¹ See footnote number 1 in Table I.² These values represent 100% of the warp direction tearing strength (1,600 grams) and of the warp direction Stoll flex abrasion (900 cycles to failure).³ This treatment represents a conventional pad-dry-cure operation and is used as a standard to compare previous treatments.

The data in Tables I and II show that a wide range of conditions can be used to produce abrasion and wrinkle resistant fabrics by the processes described in this invention. The balance of properties of the treated fabric, that is, the retention of strength and abrasion resistance for cotton fabrics with high wrinkle resistance, is excellent. This excellent balance of properties becomes more significant when it is realized that the process is one which can be carried out simply and economically on a commercial scale.

Fixations for treatments 1–17 in this disclosure were carried out in the same piece of pilot plant equipment. However, these results have been checked satisfactorily on other equipment. Reproducibility has been found to be very good. Therefore, it is suggested that any chamber equipped with steam inlets and auxiliary heating (if necessary) such that temperatures over 100° C. can be maintained, would be satisfactory for this process. Condensation on fabrics must be minimized, but drying of fabrics which may occur during fixation does not appear to produce an unsatisfactory product. Cured fabrics have a soft, full hand comparable to that of untreated cotton. These and many other advantages should be obvious to those skilled in the art.

The use of low pH values in this disclosure does not produce an unsatisfactory fabric for delayed curing. Fabrics fixed at pH 3.5 (mixed catalyst) but not cured have been stored up to 30 days at 22–25° C. with only a moderate (30°) increase in conditioned wrinkle recovery angle (warp plus filling). The acidity used during fixation does, however, influence the conditions (time and temperature) used during curing. Curing at temperatures as low as 120° C. are in some cases satisfactory. Curing in a steam atmosphere has also produced fabrics with an excellent balance of properties.

If for some reason it is desirable to wash an intermediate (or fixed) fabric, this may be done (see Example XIII). However, if the fixation is carried out at pH of 3.5 or higher, it is likely that additional resin will have to be added to produce highly wrinkle resistant fabrics. If such a wash is necessary, the softener may be omitted from the first pad bath.

The process of this invention may be used to treat substantially any hydrophilic fibrous 100% cellulosic material or blends of cellulose and other fibers (wherein the cellulose is cotton, rayon, ramie, jute, and the like) which can be impregnated with a liquid, dried, and cured. The following examples are given by way of illustration and are not by way of limitation of invention. The detailed procedures given in the examples are illustrative, and are not the only specific conditions for the production of an acceptable finished textile. Many variations or additions within these procedures can be made as will be readily apparent to those skilled in the art. In the examples all parts and percentages are by weight unless noted otherwise. The fabrics were tested by conventional methods.

EXAMPLE 1

Wrinkle and abrasion resistant cellulosic fabric is produced in a process whereby Style #413 cotton twill (Testfabrics, Inc.) was padded at 20 p.s.i. pressure in a solution of the following composition: 7 percent solids of a polymer builder of a melamine type resin, methylated

methylolmelamine, 3 percent solids of a crosslinking resin, dimethyloldihydroxyethyleneurea, 4 percent solids of a catalyst, magnesium chloride hexahydrate, 2 percent solids of a softener, polyethylene emulsion, and the remainder water. Three drops of a wetting agent, an alkyl aryl ethyleneoxide alcohol, were added to the pad bath per each 100 grams of solution. The solution was used at about pH 6, which was the pH of the solution without adjustment. The fabric was fed directly into the steam chamber of a flash ager at a rate calculated to allow the fabric to be steamed for 3 minutes at 138° C. On exit from the steam chamber, the fabric was moist. The fabric was air dried at ambient temperature and subsequently cured. The results are given under treatments 3 and 4 of Table I.

EXAMPLE 2

Exactly the same as Example 1, except the pH of the pad bath was adjusted to pH 4 with hydrochloric acid before use. The results are given under treatments 5, 6, and 7 of Table I.

EXAMPLE 3

Exactly the same as Example 1, except the pad bath solution was prepared using 5 percent of a methylated methylolmelamine, 5 percent dimethyloldihydroxyethyleneurea, 4 percent catalyst, 2 percent softener, and the remainder water. The results are given under treatment 1 of Table I.

EXAMPLE 4

Exactly the same as Example 3, except the pH of the pad baths was adjusted to pH 4.5 using hydrochloric acid. The results are given under treatment 2 of Table I.

EXAMPLE 5

Exactly the same as Example 1, except the pad bath solution was prepared using 10 percent of a methylated methylolmelamine, 5 percent dimethyloldihydroxyethyleneurea, 4 percent catalyst, 2 percent softener, and the remainder water. The results are given under treatment 8 of Table I.

EXAMPLE 6

Exactly the same as Example 5, except the pH of the pad bath was adjusted to pH 4.5 using hydrochloric acid. The results are given under treatments 9, 10, and 11 of Table I.

EXAMPLE 7

Wrinkle and abrasion resistant cellulosic fabric is produced in a process whereby Style #413 cotton twill (Testfabrics, Inc.) was padded at 20 p.s.i. pressure in a solution of the following composition: 7 percent solids of a polymer builder of a melamine type resin methylated methylolmelamine, 3 percent solids of a crosslinking resin, dimethyloldihydroxyethyleneurea, 1 percent of a mixed catalyst consisting of a 1:1 molar ratio of citric acid to magnesium chloride hexahydrate, 2 percent solids of a softener, polyethylene emulsion, and the remainder water. Three drops of a wetting agent, an alkyl aryl ethyleneoxide alcohol, were added to the pad bath per each 100 grams of solution. The solution was used at about pH 3.5 which was the pH of the solution without alteration. The fabric was fed directly into the steam chamber of a flash ager at a rate calculated to allow the fabric to be

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steamed for 3 minutes at 138° C. On exit from the steam chamber the fabric was moist. The fabric was air dried at ambient temperature and subsequently cured. The results are given under treatments 15, 16, and 17 of Table II.

EXAMPLE 8

Exactly the same as Example 7, except the pad bath solution was prepared using 5 percent of a methylated methylolmelamine, 5 percent dimethyloldihydroxyethyleneurea, 1 percent mixed catalyst, 2 percent softener, and the remainder water. The results are given under treatments 12, 13, and 14 of Table II.

EXAMPLE 9

Exactly the same as Example 8, except the fabric used was Style 1.42 cotton twill (Fairforest Finishing Division) and the time of exposure in the steam chamber was 1 minute. The results of textile tests for this fabric cured at 130° C. for 15 minutes were as follows: conditioned wrinkle recovery angle was 283° (warp plus filling), wet wrinkle recovery angle was 265° (warp plus filling), Scott warp breaking strength was 73 percent of the original washed fabric, Scott filling breaking strength was 64 percent of the original washed fabric, Elmendorf warp tearing strength was 96 percent of the original washed fabric, and the warp flex abrasion resistance was 99 percent of the original washed fabric.

EXAMPLE 10

Wrinkle and abrasion resistant cellulosic fabric is produced in a process whereby Style #413 cotton twill (Testfabrics, Inc.) was padded at 20 p.s.i. pressure in a solution of the following composition: 5 percent solids of a polymer builder of a melamine type resin, methylated methylolmelamine, 5 percent solids of a crosslinking resin, dimethyloldihydroxyethyleneurea, 4 percent solids of a catalyst, magnesium chloride hexahydrate, 2 percent solids of a softener, a polyethylene emulsion, and the remainder water. Three drops of a wetting agent, an alkyl aryl ethyleneoxide alcohol, were added to the pad bath per each 100 grams of solution. The solution was used at about pH 6, which was the pH of the solution without alteration. The fabric was steamed at 107° C. for 2 minutes in a gas and steam heated forced draft oven. On removal from the steam oven the fabric was moist. The fabric was air dried at ambient temperature and subsequently cured for 3 minutes at 160° C. The average results of textile tests were as follows: conditioned wrinkle recovery angle was 286° (warp plus filling), wet wrinkle recovery angle was 272° (warp plus filling), Scott warp breaking strength was 61 percent of the original washed fabric, Elmendorf warp tearing strength was 78 percent of the original washed fabric and the warp flex abrasion resistance was 65 percent of the original washed fabric.

EXAMPLE 11

Exactly the same as Example 10, except the pH of the pad bath was adjusted to pH 4 with hydrochloric acid before use. The average results of textile tests were as follows: conditioned wrinkle recovery angle was 297° (warp plus filling), wet wrinkle recovery angle was 275° (warp plus filling), Scott warp breaking strength was 64 percent of the original washed fabric, Elmendorf warp tearing strength was 74 percent of the original washed fabric and the warp flex abrasion resistance was 46 percent of the original washed fabric.

EXAMPLE 12

Exactly the same as Example 10, except the time of exposure to the steam atmosphere was increased to 5 minutes. The average results of textile tests were as follows: conditioned wrinkle recovery angle was 286° (warp plus filling), wet wrinkle recovery angle was 273° (warp plus filling), Scott warp breaking strength was 61 percent of the original washed fabric, Elmendorf warp tearing strength was 75 percent of the original washed fabric

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and the warp flex abrasion resistance was 58 percent of the original washed fabric.

EXAMPLE 13

Wrinkle and abrasion resistant cellulosic fabric is produced in a process whereby Style 1.42 cotton twill (Fairforest Finishing Division) was padded at 20 p.s.i. pressure in a solution of the following composition: 5 percent solids of a polymer builder of a melamine type resin, methylated methylolmelamine, 5 percent solids of a crosslinking resin, dimethyloldihydroxyethyleneurea, 1 percent of a mixed catalyst consisting of a 1:1 molar ratio of citric acid to magnesium chloride hexahydrate, 2 percent solids of a softener, polyethylene emulsion and the remainder water. Three drops of a wetting agent, an alkyl aryl ethyleneoxide alcohol, were added to the pad bath per each 100 grams of solution. The solution was used at about pH 3.5 which was the pH of the solution without alteration. The fabric was fed directly into the steam chamber of a flash ager at a rate calculated to allow the fabric to be steamed for 3 minutes at 138° C. On exit from the steam chamber the fabric was moist. The fabric was air dried at ambient temperature, machine washed to remove any unfixed material and tumble dried. The fabric was then padded in a solution of the following composition: 2.5 percent solids of a polymer builder of a melamine type resin, methylated methylolmelamine, 2.5 percent solids of a crosslinking resin, dimethyloldihydroxyethyleneurea, 1 percent of a mixed catalyst consisting of a 1:1 molar ratio of citric acid to magnesium chloride hexahydrate, 2 percent solids of a softener, polyethylene emulsion and the remainder water. Three drops of a wetting agent, an alkyl aryl ethyleneoxide alcohol, were added to the pad bath per each 100 grams of solution. The solution was used at about pH 3.5 which was the pH of the solution without alteration. The fabric was pin framed, dried 4 minutes at 60° C., cured 5 minutes at 160° C., machine washed and tumble dried. The results of the textile tests were as follows: conditioned wrinkle recovery angle was 285° (warp plus filling), wet wrinkle recovery angle was 253° (warp plus filling), Scott warp breaking strength was 76 percent of the original washed fabric, Scott filling breaking strength was 68 percent of the original washed fabric, Elmendorf warp tearing strength was 99 percent of the original washed fabric and the warp flex abrasion resistance was 100+ percent of the original washed fabric.

EXAMPLE 14

In order to substantiate the assertions concerning efficiency of use of resins made in this invention, some of the fabrics prepared as described in the above examples were subjected to nitrogen analyses. The nitrogen analyses were performed on:

(A) Dry fabric samples which were washed immediately after the fixation.

(B) Dry fabric samples which were fixed but were not cured or washed.

(C) Dry fabric samples which were fixed, cured, and then washed.

For comparative purposes, a 1 percent nitrogen content may be considered to be equivalent to about 3-5 percent add-on when Aerotex 23S is used as the polymer builder. These data (see Table IV) demonstrate that the process of this invention is a highly efficient one—not only in that the resins applied are retained essentially 100 percent in many cases but that in many cases very low resin contents (1.1-1.2 percent nitrogen) on the finished fabrics are required to produce a fabric with outstanding properties. The higher nitrogen contents in columns A, B, and C in Table IV when Aerotex M-3 is the polymer builder generally reflect the higher nitrogen content of Aerotex M-3 as compared to Aerotex 23S. However, the data also suggest that when Aerotex M-3 is used as the polymer builder, the percent of nitrogen in

the resin actually fixed (column A) is greater than when Aerotex 23S is used as the polymer builder.

Thus, during the fixation step all the resins present is not fixed in the sense that it cannot be removed by washing. Part of the resin is bound to the cellulosic fabric in some manner; however, the remainder is present in such a form that it emits little or no formaldehyde odor and can be used in the subsequent cure.

TABLE IV.—NITROGEN ANALYSES OF VARIOUS FABRICS¹

Treatment, as in—	Resin content	Nitrogen (percent)		
		A	B	C
Example:				
3-----	5% 23S/5% 183-----	0.05	1.14	1.04
3-----	5% M-3/5% 183-----	0.29	1.60	1.34
1-----	7% 23S/3% 183-----	0.10	1.25	1.22
1-----	7% M-3/3% 183-----	0.45	1.83	1.84
2-----	7% 23S/3% 183-----	0.67	1.20	1.19
4-----	5% 23S/5% 183-----	0.20	1.14	1.05
4-----	5% M-3/5% 183-----	1.02	1.56	1.50
8-----	5% 23S/5% 183-----	0.50	1.18	1.15

¹ Cured fabrics were heated at 160° C. for 3 minutes. Columns A, B, and C are defined in Example 14. In the "Resin Content" column, 23S stands for Aerotex 23S, M-3 stands for Aerotex M-3, and 183 stands for Permafresh 183. All percentages are solids content, by weight.

We claim:

1. A process for accomplishing the wet-fixation of mixed resins in a cellulosic fabric preparatory to heat-curing of the mixed resins, said process comprising the following steps:

- (a) padding the cellulosic fabric to a wet pickup of about from 60 to 80 weight percent with an aqueous solution containing about 5 weight percent of a methylated methylolmelamine, about 5 weight percent of dimethyloldihydroxyethyleneurea, about 4 weight percent of magnesium chloride hexahydrate and about 2 weight percent of emulsified polyethylene, the pH of which aqueous solution has been adjusted to within the range pH 3 to pH 6,
- (b) subjecting the mixed resin padded cellulosic fabric to live steam at a temperature within the range 100° C. to 140° C. for a period of time from 1 to 10 minutes, the longer time intervals being employed with the lower temperatures, to produce a cellulosic fabric, the nitrogen content of which is about 1 percent by weight.

2. A process for accomplishing the wet fixation of mixed resins in a cellulosic fabric preparatory to heat-curing of the mixed resins, said process comprising the following steps:

- (a) padding the cellulosic to a wet pickup of about from 60 to 80 weight percent with an aqueous solution containing about 5 weight percent of a methylated methylolmelamine, about 5 weight percent of dimethyloldihydroxyethyleneurea, about 1 weight percent of a mixed catalyst consisting of a 1:1 molar ratio of citric acid and magnesium chloride hexahydrate, and about 2 weight percent of emulsified polyethylene, the pH of which aqueous solution has been adjusted to within the range pH 3 to pH 4.

- (b) subjecting the mixed resin padded cellulosic fabric to live steam at a temperature within the range 100° C. to 140° C. for a period of time of from 1 to 10 minutes, the longer time intervals being employed with the lower temperatures, to produce a cellulosic fabric the nitrogen content of which is about 1 percent by weight.

3. A process for producing a wrinkle resistant mixed resin finish on a cellulosic fabric which process includes wet-fixation of the mixed resins in the cellulosic fabric preparatory to final heat-curing of the mixed resins, said process comprising the following steps:

- (a) padding the cellulosic fabric to a wet pickup of about from 60 to 80 weight percent with an aqueous solution containing about 5 weight percent of a melamine type resin, about 5 weight percent of a crosslinking resin, about 1 weight percent of a mixed catalyst consisting of a 1:1 molar ratio of citric acid and magnesium chloride hexahydrate,
- (b) subjecting the mixed resin padded cellulosic fabric to live steam at a temperature of about 135° C. for a period of about 3 minutes,
- (c) drying the steamed cellulosic fabric from step (b),
- (d) washing the dried cellulosic fabric from step (c) free of unfixed resin and drying the washed cellulosic fabric,
- (e) padding the cellulosic fabric from step (d) to a wet pickup of about 80 percent by weight with an aqueous solution containing about 2.5 weight percent of a melamine resin, about 2.5 weight percent of a crosslinking resin, about 1 weight percent of a mixed catalyst consisting of a 1:1 molar ratio of citric acid and magnesium chloride hexahydrate,
- (f) drying the padded cellulosic fabric from step (e) at 60° C. and
- (g) curing the dried cellulosic fabric from step (f) at a temperature of about 160° C. for a time interval of about 5 minutes.

References Cited

UNITED STATES PATENTS

2,763,574	9/1956	Ruperti	117—138.5
2,804,402	8/1957	Williams	117—139.4
3,052,570	9/1962	Polansky et al.	117—139.4
3,138,802	6/1964	Getchell	117—143X
3,177,093	4/1965	Van Loo et al.	117—139.4
3,245,831	4/1966	Shippee	117—139.4X
3,341,955	9/1967	Pyke et al.	117—139.4X
3,374,107	3/1968	Cotton	117—139.4

WILLIAM D. MARTIN, Primary Examiner

T. G. DAVIS, Assistant Examiner

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