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PROCESS FOR PRODUCING WRINKLE RESISTANT CELLULOSIC TEXTILE MATERIALS

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This invention relates to the treatment of cellulosic textiles in order to make them resistant to creasing and wrinkling.

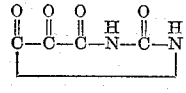
It is well known that textiles which resist the formation of wrinkles and creases during use have long been preferred by consumers. Items, of which crease, muss, and wrinkle resistance are desirable characteristics include clothing, bed spreads, sheets, pillow cases, drapery and upholstery material, slipcovers, and others. Of these, in clothing, which usually requires frequent ironings to maintain a smooth appearance, wrinkle resistance is most important.

Various compounds have been applied to textiles to impart crease and wrinkle resistance, the most widely used ones being cyclic ethyleneurea-formaldehyde resins, urea-formaldehyde resins, melamine-formaldehyde resins, epoxy ethers, and formaldehyde. These compounds impart varying degrees of wrinkle resistance to the textiles, depending on the amount and type of wrinkle resistance agent used, the catalyst used, and the method of curing employed. Such textiles are usually called "minimum care" or "wash-and-wear fabrics."

It is also important that the wrinkle resistant textiles be resistant to fiber damage and discoloration due to retention of chlorine upon hypochlorite bleaching. Of equal importance is the durability of wrinkle-resistance finishes to both home and the more vigorous commercial launderings. With many of the above creaseproofing agents there is a compromise between maximum crease-resistance to give a durable finish, and the susceptibility of the finish to degradation by retention of chlorine. In the case of formaldehyde, one of the greatest drawbacks is the precise control of conditions required for curing.

It is the purpose of this invention to produce a wrinkle resistance finish which will impart to textiles maximum crease resistance but negligible chlorine retention and damage due to such retention. Another object of this invention is to produce a crease resistance finish stable under acidic or alkaline conditions, durable to laundering and bisulfite or perborate bleaching. The crease resistance finish can be applied to textiles using a wide range of treating conditions. This is commercially advantageous.

The crease resistance finish which is the subject of this invention involves the reaction product of formaldehyde (including sources such as paraformaldehydes) and the ureide of mesooxalic acid (alloxan). The latter compound is represented by the formula:



The finishing agent can be prepared by reacting from 1 to 4 or more moles of formaldehyde with one mole of the ureide under acidic conditions (pH 2–6) in sufficient

water to give a 33⅓ percent solution of the reaction product. At least two moles formaldehyde is required to prepare the dimethylol compound, however less can be used to prepare a partially methylolated compound. The solution thus prepared is diluted to the padding concentration and padded onto a cellulosic textile immediately or up to times in excess of three months.

In accordance with the invention an aqueous solution prepared as described above, and containing from 1 to 20 percent of the reaction product containing 2 or more moles of formaldehyde in combination with 1 mole of the ureide, is padded onto the cloth by passing through efficient squeeze rolls, for example, using two dips and two nips to give dry add-ons of 0.01 percent to 5 or more percent based on the weight of the dry cloth. About 0.5 percent to 3 percent by weight, of a catalyst, based on the total weight of the padding bath, is added to the padding bath to facilitate curing, and to produce a more durable finish. Catalysts which can be used to promote curing include mineral acids, various salts of strong acids, such as ammonium salts, alkanolamine salts, metallic salts of strong acids and weak bases, such as zinc nitrate and magnesium chloride.

The curing step, the reaction of the formaldehyde-ureide with the textile is carried out by the usual procedure. The padded textile is dried at an elevated temperature, for example, 60°–70° C. for 7 minutes then further heated to effect the curing. Improved properties are obtained at 120°–160° C. from 0.5 to 5 minutes. Curing temperatures above 160° C. are not generally employed, as they have a degradative effect on the fabric. The curing time is temperature dependent, and can vary over a wide range. Thus, improved properties can be obtained at 160° C. for 30 to 60 seconds. The curing step is best followed, by an after wash with a non-ionic detergent, and 1 to 2 percent sodium perborate to remove any excess unreacted crease resistance agent and catalyst, to remove any discoloration brought on by curing, and to improve the hand of the textile.

The process to which this invention relates can be used to treat any hydrophilic fibrous cellulosic textiles such as cotton, linen, ramie, jute, regenerated cellulose, and others.

The following examples are given as illustrations and are not by any means intended to limit the scope of this invention. All percentages given in the examples are percentages by weight of the total solution. The fabrics were tested by the following methods: crease recovery angle, Monsanto method, American Society for Testing Materials (ASTM), Standards for Textile Materials D1295–53T, tearing strength, Elmendorf Method ASTM designation D1424–56T; damage caused by retained chlorine (scorch test), American Association of Textile Chemists and Colorists tentative test method 69–1952; wash-and-wear (appearance), AATCC tentative test method 88–1958. Two types of laundering were used to test the durability of the finish: ten home-type launderings were performed in an agitator-type home automatic washer with a household detergent, 0.02% available chlorine in hot water, and followed by drying in a tumble dryer. The other laundering procedure was the more vigorous one described in AATCC test method 14–53. To test acid durability the samples were allowed to stand for 30 minutes at 80° C. in a stripping solution (50 times the sample weight) containing 5% urea and 1.5% phosphoric acid, rinsed in water, and dried in a tumble dryer.

EXAMPLE 1

A 33⅓% water solution of alloxan was prepared in the following manner: alloxan was dissolved in 2.8 times its weight in water and sufficient 36.3% formaldehyde solution was added to afford a mole ratio of formaldehyde to

alloxan of 2:1. This solution, pH 3, of 33⅓% solids concentration was allowed to stand overnight at room temperature. After this time the 33⅓% solution was diluted with sufficient water to give a 7% solids concentration, and ⅓% by weight of hydrated magnesium chloride ($MgCl_2 \cdot 6H_2O$) was added as catalyst. The treating solution was padded onto a sample of 80 x 80 cotton print cloth to give a 70–80% wet pickup. The wet fabric was dried at original dimensions for seven minutes at 60° C., and then cured at original dimensions for 3 minutes at 160° C. The curing step was followed by an after wash in warm water with a nonionic detergent, and 0.2% sodium perborate added, and then tumble drying. The finished fabric possessed a crease recovery angle of 275° (warp+fill) while an untreated, after-washed sample had a crease recovery angle of only 187° (warp+fill).

EXAMPLE 2

A sample treated as described in Example 1 was subjected to the urea-phosphoric acid stripping procedure. The crease recovery angle of the fabric after stripping was 133° (warp), when compared with that of the original treated fabric 139° (warp) shows that severe acidic conditions do not remove the finish from the fabric. The crease recovery angle of the untreated fabric is 95° (warp).

EXAMPLE 3

For comparison purposes the following percent solutions of alloxan $\cdot 2CH_2O$ were prepared and applied to 80 x 80 cotton print cloth with 1.5% $MgCl_2 \cdot 6H_2O$ as catalyst, and processed as described in Example 1:

A. A 33⅓% solution of the finishing agent prepared as in Example 1, was diluted with sufficient water to give a 1% solids concentration.

B. A 33⅓% solution of the finishing agent, prepared as described in Example 1, was diluted with sufficient water to give a 5% solids concentration.

C. A 33⅓% solution of the finishing agent, prepared as described in Example 1, was diluted with sufficient water to give a 7.5% solids concentration.

D. A 33⅓% solution of the finishing agent, prepared as described in Example 1, was diluted with sufficient water to give a 10% solids concentration.

E. A 33⅓% solution of the finishing agent, prepared as described in Example 1, was diluted with sufficient water to give a 15% solids concentrations.

F. A 33⅓% solution of the finishing agent, prepared as described in Example 1, was diluted with sufficient water to give a 20% solids concentration.

Properties of the treated fabrics are given in Table I.

Table I

Percent alloxan, $\cdot 2CH_2O$ in pad bath	Percent add-on	Physical properties						
		Wash-wear rating	Crease recovery angle (deg.)			Tearing strength (ams.)		
			W.	F.	W. and F.	W.	F.	W. and F.
Control		1	95	92	187	1,127	780	1,907
(A) 1	0.01	3	106	105	211	333	227	560
(B) 5	0.05	4	129	132	261	407	207	614
(C) 7.5	0.7	5	128	137	265	327	180	507
(D) 10	1.2	5	137	137	274	327	180	507
(E) 15	1.5	5	135	138	273	353	167	520
(F) 20	4.6	5	143	139	282	220	120	340

EXAMPLE 4

To determine the effect of curing conditions on the physical properties of the finished fabric, a series of samples, treated with a solution containing 7%

alloxan $\cdot 2CH_2O$

and 1.5% $MgCl_2 \cdot 6H_2O$, prepared as described in Example 1, were dried for 7 minutes at 60° C., and cured as listed in Table II. After curing the samples were given an after wash and tumble-dried as described in Example 1.

Table II

Curing time (min.)	Curing temperature (deg. C.)	Add-on, percent	Physical properties							
			Wash-wear rating	Crease recovery angle (deg.)			Tearing strength (gms.)			
				W.	F.	W. and F.	W.	F.	W. and F.	
Control			1	95	92	187	1,127	780	1,907	
3	120	0.01	2+	110	111	221	733	473	1,206	
5	120	0.01	2+	107	104	211	673	453	1,126	
3	135	0.01	3	114	118	232	640	400	1,040	
5	135	0.01	3+	115	117	232	547	347	894	
3	150	0.01	4	108	118	226	480	307	787	
5	150	0.01	4	117	125	242	440	273	713	
0.5	160	0.01	3	104	109	213	680	460	1,140	
1	160	0.01	3	110	114	224	607	373	980	
2	160	0.01	4	120	126	246	473	287	760	
3	160	0.70	5	128	137	265	327	180	507	
5	160	0.70	5	119	128	247	367	213	580	

EXAMPLE 5

Solutions, 33⅓% of the finishing agent, prepared as described in Example 1 were adjusted with dilute NaOH to pH 4, pH 5, pH 6 before diluting to 7% padding concentration and addition of 1.5% $MgCl_2 \cdot 6H_2O$ and padding onto 80 x 80 cotton print cloth. The physical properties of the finishes obtained from these solutions were compared with those obtained from a alloxan $\cdot 2CH_2O$ solution diluted without adjusting the pH.

Table III

pH 33⅓% soln.	Add-on, percent	Physical properties						
		Wash-wear rating	Crease recovery angle (deg.)			Tearing strength (gms.)		
			W.	F.	W. and F.	W.	F.	W. and F.
Control		1	95	92	187	1,127	780	1,907
3	0.7	5	139	136	275	327	187	514
4	0.01	5	133	137	270			
5	0.01	5	140	140	280			
6	0.01	4	123	125	248			

As can be seen from Table III, an acceptable finish may be obtained when the pH of the finishing solution is high as 6.

EXAMPLE 6

A sample of 80 x 80 cotton print cloth was treated with a 7% solution of alloxan $\cdot 2CH_2O$, and 1.5% $MgCl_2$ as catalyst, as described in Example 1. This fabric, a sample finished with 7½% dimethylol ethyleneurea, catalyzed by 0.5% $Zn(NO_3)_2 \cdot 6H_2O$, and an untreated washed control were given 5 AATCC 14–53 washes and 10 home launderings with hypochlorite bleach. The effect of these washes on the physical properties of the finish are listed in Tables IV and V. (The original properties of the fabrics listed in Table V are the same as those included in Table IV.)

Table IV

Finishing agent used	Original			After 5 AATCC 14-53 Washes		
	Crease rec. angle (deg.) W. and F.	Tearing strength (gms.) W. and F.	Brk. str. ret. after AATCC 69-1952 scorch test, percent orig.	Crease rec. angle (deg.) W. and F.	Tearing streng h (gms.) W. and F.	Brk. str. ret. after AATCC 69-1952 scorch test, percent orig.
Untreated fabric	187	1,907	91.3	172	1,707	106.4
7% DMA	275	514	82.0	265	507	102.8
7½% DMEU	300	787	94.8	277	867	46.4

Table V

Finishing agent used	After 10 home launderings with 0.02% available chlorine		
	Crease rec. angle (deg.) W. and F.	Tearing strength (gms.) W. and F.	Brk. str. ret. after AATCC 69-1952 scorch test, percent orig.
Untreated fabric	172	1,233	102.0
7% DMA	250	453	100.1
7½% DMEU	288		101.0

Tables IV and V show that the alloxan·$2CH_2O$ finish is durable through both the home launderings and the more vigorous AATCC 14–53 washes. The

alloxan·$2CH_2O$

finish is far superior in resistance to damage by retained chlorine after 5 AATCC 14–53 washes than is the DMEU finish. After 10 home launderings the alloxan·$2CH_2O$ finish is equal to the DMEU finish in resistance to damage by retained chlorine.

EXAMPLE 7

The alloxan·$2CH_2O$ in 33⅓% solution, prepared as described in Example 1 has a shelf life of more than one month. Fabric treated with a 7% solution of the stored finishing agent had a crease recovery angle of 133° (warp). Fabric treated with a 7% solution, prepared from a 33⅓% solution allowed to stand overnight before padding had a crease recovery angle of 139° (warp).

I claim:

A process for finishing textiles comprising wetting hydrophilic fibrous cellulosic textile to about 80% by weight wet pick-up with an aqueous solution containing from 1 to 20% by weight of the essentially monomeric reaction product of 1 mole of the ureide of oxalic acid with about from 1–4 moles of formaldehyde and from 0.5 to 3.0% by weight of an acidic polymerization catalyst and heating the wetted textile at a temperature of from 120° to 160° C. for a period of from 0.5 to 5 minutes, the longer time intervals being employed with the lower temperatures.

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