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WRINKLE RESISTANT CELLULOSE TEXTILES AND PROCESSES FOR PRODUCING SAME

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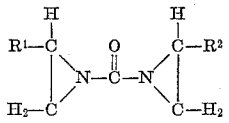
(Granted under Title 35, U.S. Code (1952), sec. 266)

A nonexclusive, 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 application is a continuation-in-part of our application Serial No. 761,660, filed September 17, 1958, now abandoned.

This invention relates to a process of treating a cotton textile with a carbonylbisaziridine compound to render the cotton textile rot-, crease-, and shrink-resistant.

According to the invention, the process involves impregnating a cotton textile, e.g., a fabric, cloth, etc., with an aqueous medium containing a monomeric carbonylbisaziridine compound of the formula



wherein each of R^1 and R^2 is a member selected from the group consisting of hydrogen, methyl, and ethyl, R^1 being equal to R^2, to afford a resin pickup of at least about 2% by weight, based on the dry weight of the impregnated cotton textile, and heating the impregnated cotton textile at a temperature of about from 100° to 170° C. for about from 10 minutes to 2 minutes, the longer time intervals being employed with the lower heating temperatures, to polymerize the carbonylbisaziridine compound on and within the fibers of the cotton textile and to crosslink the cellulose molecules of said fibers. The thus-modified cotton textile is characterized in that it is rot-, crease-, and shrink-resistant.

We have discovered that monomeric carbonylbisaziridine and its methyl and ethyl derivatives, as embraced within the above formula, can be readily applied to a cotton textile and caused to polymerize on or within the fibers thereof and also to crosslink the cellulose molecules of the fibers. The polymerization and crosslinking takes place by the opening of the reactive aziridine rings.

Carbonylbisaziridine and its methyl and ethyl derivatives can be prepared by substantially any of the known processes for producing aziridinyl compounds. In general they are prepared by reacting ethylenimine or alkyl substituted ethylenimines with carbonyl chloride, sometimes referred to as phosgene. The preparation and polymerization of carbonylbisaziridine $[(CH_2)_2N]_2CO$, has been described by Bestian and coworkers [Bestian et al., Ann. 566, 210–244 (1950)].

Simultaneous polymerization within and crosslinking of the cellulose molecules of the fibers provided by this invention can be produced in acidic, neutral, or alkaline conditions. Water and organic solvents, such as toluene, acetone, and dimethyl formamide, can be used for application to the fibers. Cellulose fibers treated in accordance with this invention are insoluble in cuprammonium hydroxide solutions.

The wrinkle resistance of cotton textiles can be greatly increased by impregnating the fibrous materials with an aqueous solution of the monomeric compounds and with water soluble partially polymerized monomers formed by reacting the monomeric compounds until partial polymerization occurs and curing the impregnated materials at the temperatures conventionally used for curing cotton textiles.

Surface active agents, water repellents, and other textile treating agents may be incorporated in the aqueous treating medium, which includes emulsions, to modify the treated textiles. For example, the wrinkle resistance, tear strength, and abrasion resistance can be improved by incorporating a softening agent or water repellent in the treating media.

When a cotton textile is being treated it is of advantage to pass it through the impregnating liquor and remove excess liquor by passing the textile through squeeze rolls. It is also advantageous to dry the textile at about 70° to 110° C. before it is cured at a temperature of from about 100° to 170° C. for 10 to 2 minutes using the longer time for the lower temperature. In some cases it is advantageous to cure the impregnated textile by pressing with a hot iron set at from 200° to 270° C. Permanent creases can be imparted to the textile by curing the creases in the impregnated textile by pressing with a hot iron.

In addition to good wrinkle resistance and crease retention, other desirable properties imparted to cotton textiles by this invention are: shrink resistance, rot resistance, and improved dyeability to acid dyes. The effects of the treatment are permanent, and resistant to laundering and drycleaning. The treated textiles have an excellent hand even with a resin add-on up to 10%. As little as 2% resin add-on imparts considerable improvement in wrinkle resistance. The tear strength is reduced but is acceptable, particularly when softening agents are used. The degree of crease resistance, rot resistance, etc., can be varied by varying the amount of compound put in the textile.

The following examples are illustrative of the invention. The textile testing data shown in the examples was obtained by the standard methods of the American Association of Textile Chemists and Colorists. All parts are by weight.

EXAMPLE 1

A solution was prepared by dissolving 37.5 parts of carbonylbisaziridine (crude) and 2.5 parts of the isooctylphenyl ether of polyethylene glycol (used as a wetting agent) in 210 parts of water. The pH of the solution was 9.3. This solution was applied to two samples of 80 x 80 cotton print cloth in a padder to give a wet pickup of about 76–77%. One sample was dried by hanging in an oven for 5 minutes at 85° C. and then cured for 10 minutes at 140° C. A second sample was dried for 5 minutes at 85° C. and cured for 20 minutes at 140° C. The sample had a good hand and the discoloration was hardly noticeable. The samples were washed well in hot water and dried. Half of each sample was softened by padding through a 1% aqueous emulsion of N,N-octadecyl ethyleneurea, drying for 15 minutes at 90° C., washing, and drying. The resin add-on and test results before and after softening are shown below in Table I.

Table I

Curing Time (min.)	Before Softening: Percent Resin	Tensile Strength Warp (lbs.)	Elmendorf tear warp (lbs.)	Wrinkle recovery warp (angle)	After Softening: Tensile strength warp (lbs.)	Elmendorf tear warp (lbs.)	Wrinkle recovery warp (angle)
10	8.2	33.9	1.1	120	29.9	1.4	135
20	8.6	32.2	1.1	127	29.6	1.5	141
Control		43.8	2.3	78			113

The first sample in Table I was given five laundry cycles according to Federal Spec. CCC–T–191*b*. The wrinkle recovery angle of the unsoftened sample after laundering was 123° and of the softened sample, 125°. The second sample in Table I was given a chlorine bleach and scorch. The unsoftened sample lost only 6% of its tensile strength, and the softened sample only 14%.

EXAMPLE 2

A solution was prepared by dissolving 16.3 parts of carbonylbisaziridine (purified by distillation) and 0.8 part of the isooctylphenyl ether of polyethylene glycol in 64.4 parts of water. The solution was padded on to 80 x 80 cotton print cloth in the same manner as in Example 1. One sample was dried for 15 minutes at 85° C. and cured for 10 minutes at 120° C. It had a resin add-on of 8.2% and a wrinkle recovery angle of 281° (warp plus filling). A second sample was dried for 15 minutes at 85° C. and cured for 10 minutes at 150° C. It had a resin add-on of 8.9% and a wrinkle recovery angle of 141° (warp). The latter sample after softening with N,N-octadecyl ethyleneurea as in Example 1 had a wrinkle recovery angle of 149° (warp). The softener may be incorporated in the treating solution instead of being applied as a separate treatment.

EXAMPLE 3

A part of the solution prepared in Example 2 was allowed to stand for a period of 24 hours at room temperature. Then it was padded on to a sample of 80 x 80 cotton print cloth, dried for 15 minutes at 85° C. and cured for 10 minutes at 120° C., washed in hot water, and dried. It had a resin add-on of 7.9%, a wrinkle recovery angle of 132° (warp), a tensile strength of 42.3 lbs., and an Elmendorf tear strength of 1.4 lbs. These results compare favorably with samples treated with the fresh solution in Example 2. This shows that the stability of the solution after ageing for 24 hours is still good. Fabric treated with solutions aged for longer periods give stiffer finishes due to partial polymerization during the ageing process.

EXAMPLE 4

An aqueous solution was prepared containing 10% crude carbonylbisaziridine. The solution was padded on to 80 x 80 cotton print cloth, dried for 5 minutes at 85° C., cured for 10 minutes at 140° C., washed in hot water, and dried. The sample contained 4.5% resin. This same sample was padded through the same solution a second time and dried and cured as before. The doubly treated sample contained 8.9% resin and had a wrinkle recovery angle of 133° (warp), a tensile strength of 34.3 lbs. and an Elmendorf tear strength of 1.1 lbs. After softening with N,N-octadecyl ethyleneurea (as in Example 1) the sample had a wrinkle recovery angle of 145° (warp) and an Elmendorf tear strength of 1.3 lbs. The softened sample lost only 6% of its tensile strength after a chlorine bleach and scorch, and had a wrinkle recovery angle of 148° (warp) after five laundry cycles according to Federal Specifications CCC–T–191*b*.

EXAMPLE 5

An aqueous solution was prepared containing 100 parts of crude carbonylbisaziridine, 5 parts of the isooctylphenyl ether of polyethylene glycol, and 395 parts of water. The pH of the solution was 8.6. The solution was padded on to 80 x 80 print cloth to a wet pickup of 77%, dried for 5 minutes at 85° C., cured for 5 minutes at 150° C., washed in hot water, and dried. The sample contained 7% resin and a wrinkle recovery angle of 253°, a tensile strength of 39.7 lbs., an Elmendorf tear strength of 1.2 lbs., and a flex abrasion of 141 cycles. After softening with 1% N,N-octadecyl ethyleneurea (as in Example 1) the sample had a wrinkle recovery angle of 278°, a tensile strength of 34 lbs., an Elmendorf tear strength of 1.5 lbs., and a flex abrasion of 517 cycles.

A second sample was treated in the same manner as above except the drying step was omitted. Comparable results were obtained.

EXAMPLE 6

Cotton fabrics containing 6 to 7.5% resin were prepared by padding with aqueous solutions of carbonylbisaziridine and curing by pressing the wet fabrics with a hot hand iron. Creases pressed in the fabric by this method were sharp and could not be removed even after pressing flat from the wet state with a hot iron.

EXAMPLE 7

Crude liquid carbonylbisaziridine was cooled in a refrigerator until crystallization occurred. The crystals were filtered from the liquid portion. An aqueous solution was prepared by dissolving 30 parts of the white crystals and 2 parts of the isooctylphenyl ether of polyethylene glycol in 168 parts of water. The solution was padded on to 80 x 80 print cloth, dried for 5 minutes at 85° C., cured for 5 minutes at 150° C., washed, and dried. The sample had a resin add-on of 4.5% and had a wrinkle recovery angle of about 133° (warp) after softening with N,N-octadecyl ethyleneurea (as in Example 1).

EXAMPLE 8

An aqueous solution was prepared by dissolving 30 parts of crystalline carbonylbisaziridine and 2 parts of the isooctylphenyl ether of polyethylene glycol in 168 parts of water. The solution was padded on to 80 x 80 cotton print cloth, dried for 5 minutes at 85° C., cured for 5 minutes at 150° C., washed and dried. The fabric sample had a resin add-on of 4.5% and a wrinkle recovery angle of 281°. After softening with 1% aqueous N,N-octadecyl ethyleneurea the wrinkle recovery angle was 304°. After 15 laundering cycles the wrinkle recovery angle of the unsoftened sample was 267°, and that of the softened sample, 273°.

EXAMPLE 9

An aqueous solution was prepared by dissolving 20 parts of carbonylbisaziridine and 1.3 parts of the isooctylphenyl ether of polyethylene glycol in 112 parts of water. The solution was padded on to 80 x 80 cotton print cloth, dried for 15 minutes at 85° C. and cured for 10 minutes at 120° C., washed and dried. The fabric sample had a resin add-on of 2.2% and a wrinkle recovery angle of 206°, considerable improvement over the untreated fabric.

EXAMPLE 10

An aqueous solution was prepared by dissolving 46.5 parts of carbonylbisaziridine, 7.0 parts of dimethylformamide and 2.3 parts of the isooctylphenyl ether of polyethylene glycol in 182.6 parts of water. The solution was padded on to 80 x 80 print cloth, dried for 5 minutes at 85° C. and cured for 5 minutes at 140° C., washed, and dried. The resin add-on was 3.3%, and the wrinkle recovery angle was 278°.

EXAMPLE 11

An aqueous solution was prepared in the same manner as in Example 5 except that 1% zinc fluoborate, based on the weight of carbonylbisaziridine, was added as a catalyst. The solution was applied to 80 x 80 cotton print cloth in the same manner as described in Example 5. The sample contained 7.7% resin, and had good wrinkle resistance.

EXAMPLE 12

A series of solutions was prepared in the same manner as in Example 11 except that the concentration of zinc fluoborate was varied from 2% to 10%. Resin add-ons varying from 7% with the 2% catalyst to 14.6% with the 10% catalyst were obtained. Efficiencies based on resin add-on varied from 47% using 2% catalyst to 96% using 10% catalyst. The solution pH varied from 7.05 to 6.4. The fabric sample containing as high as 14.6% resin had a surprisingly good hand and appearance. Good wrinkle recovery was obtained at all catalyst concentrations.

EXAMPLE 13

A solution was prepared by dissolving 15 parts of carbonylbisaziridine, 1.5 parts of 40% aqueous zinc fluoborate, and 0.8 parts of the isooctylphenyl ether of polyethylene glycol in 57.7 parts of water. The solution was padded on to 80 x 80 cotton print cloth and cured for 3 minutes at 175° C., without going through a predrying stage. The fabric sample had a resin add-on of 9.6% and had good wrinkle recovery.

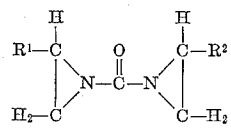
EXAMPLE 14

An aqueous solution was prepared by dissolving 15

parts of carbonylbis(2-methylaziridine) and 1 part of the isooctylphenyl ether of polyethylene glycol in 89 parts of water. The solution was padded on to 80 x 80 print cloth, cured for 5 minutes at 170° C. without predrying, washed, and dried. The fabric sample had a resin add-on of 2.3% and a wrinkle recovery angle of 224°.

We claim:

1. A process comprising impregnating a cotton textile with an aqueous medium containing a monomeric carbonylbisaziridine compound of the formula



wherein R^1 and R^2 are members selected from the group consisting of hydrogen, methyl, and ethyl, R^1 being equal to R^2, to afford a resin pickup of at least about 2% by weight, based on the dry weight of the impregnated cotton textile, and heating the impregnated cotton textile at a temperature of about from 100° to 170° C. for about from 10 minutes to 2 minutes, the longer time intervals being employed with the lower heating temperatures, to polymerize the carbonylbisaziridine compound on and within the cellulose fibers of the cotton textile and to crosslink the cellulose molecules of said fibers, thereby to produce a modified cotton textile characterized in that it is rot-, crease-, and shrink-resistant.

2. The process of claim 1 wherein R^1 and R^2 are both hydrogen.

3. The process of claim 1 wherein R^1 and R^2 are both methyl.

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