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[54] **TEXTILE FINISHING PROCESS USING A CHLORINATED HYDROCARBON AS THE REACTANT SOLVENT**
13 Claims, No Drawings

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ABSTRACT: Crease-resistance and easy-care properties of textile materials containing at least 20 percent by weight of cellulosic material are improved by impregnating the textile material with a solution or dispersion in a halogenated hydrocarbon solvent of a resin precondensate and an acid catalyst therefor, then subjecting the impregnated material to the action of hot water or steam and finally heating.

TEXTILE FINISHING PROCESS USING A CHLORINATED HYDROCARBON AS THE REACTANT SOLVENT

This invention relates to a process for the finishing of textile materials, and more particularly to a process for applying resin precondensates from a halogenated hydrocarbon solvent to textile materials consisting wholly or in part of cellulosic material, whereby the crease-resistance and easy-care properties of the textile materials are improved.

According to the present invention there is provided a process for the treatment of textile materials containing at least 20 percent by weight of cellulosic material which comprises impregnating the said textile material with a solution or dispersion in a halogenated hydrocarbon solvent of a resin precondensate as hereinafter defined which reacts under acid conditions, together with a catalyst which is an acid or an acid-generating substance, immediately thereafter subjecting the textile material so impregnated to the action of hot water or steam and thereafter heating the textile material.

By a resin precondensate is meant an initial stage condensation product of formaldehyde with a nitrogen-containing compound, which is capable of reacting with itself under acid conditions to give a fully cured resin and/or is also capable of reacting with the hydroxyl groups present in the cellulosic textile material. Resin precondensates suitable for use in the process of the invention include the initial stage condensation products of formaldehyde with urea, thiourea, dicyandiamide, amides, carbamates, aminotriazines, urons, ureins, ureides, imidazolidinones, pyrimidinones and triazinones, especially the methylol derivatives of such compounds and their lower alkyl ethers. Preferred resin precondensates are the alkyl ethers of the aforesaid methylol compounds in which the alkyl groups contain from one to four carbon atoms. Particular examples of such preferred resin precondensates include methoxymethylurea, polymethoxymethylmelamine, 1,3-dimethoxymethyl-4,5-dihydroxy-imidazolidin-2-one and 1,3-dimethoxymethyl-hexahydropyrimidin-2-one.

Halogenated hydrocarbon solvents which may be employed in the process of the invention include any of the commercially available halogenated hydrocarbons; it is preferable that the solvent should be sufficiently volatile to be capable of being rapidly removed from the textile material on treatment with hot water or steam. Particularly suitable solvents are chlorinated and chlorofluorinated hydrocarbon solvents, for example trichloroethylene, perchloroethylene and 1:1:1-trichloroethane.

As catalysts there are used in the process of the invention substances which are themselves acid or which generate acid under the conditions of the process, the said substances being soluble in the halogenated hydrocarbon solvent. Suitable solvent-soluble acids include, for example, monochloracetic acid, dodecylbenzene sulphonic acid and 2,2-bis(hydroxymethyl) butylphosphonic acid; suitable solvent-soluble acid-generating compounds include, for example, esters such as methyl p-toluene-sulphonate and quaternary ammonium salts such as cetyl pyridinium bromide and stearamidomethyl-pyridinium chloride.

The reactive components which are to be applied to the cellulosic textile material according to the process of the invention, that is to say, the resin precondensate and the acid catalyst, are initially dissolved or dispersed in the halogenated hydrocarbon solvent, and the resulting solution or dispersion is applied to the textile material by any of the known methods, such as impregnating, coating or spraying. The textile material may then be squeezed, if desired, to remove any excess of the solution. The textile material so treated is then immediately conducted either into a vessel of hot water or into a chamber containing an atmosphere of steam. In the case where hot water is employed, the water is conveniently maintained at a temperature of from 80° C. to 100° C. and the textile material is allowed to remain in contact with the hot water for a period of from 5 secs. to 60 secs. It is, however, preferred to subject the textile material to which the solution has been applied to the action of a steam atmosphere. The steam is preferably at atmospheric pressure, so that the textile material attains a temperature of from 80° C. to 100° C., but superheated steam under pressure may be employed if higher temperatures are desired. The textile material is preferably maintained in contact with the steam atmosphere for a period of from 5 secs. to 60 secs. The steam treatment may conveniently be carried out in a steam chamber such as that described in British Pat. specification No. 916,338 or that described in our copending application No. 42620/66.

After the treatment with hot water or steam, the textile material is heated in order to bring about the necessary curing of the resin precondensate under the influence of the acid catalyst. The heating operation may be carried out within a wide range of temperatures, but it is preferred to heat the textile material at a temperature within the range 120° C. to 200° C. The duration of the heating may vary between 10 seconds and 30 minutes but is preferably between 1 and 5 minutes; normally the higher the temperature which is employed, the shorter is the heating time necessary. The heating operation may if desired be preceded by a drying step in which the textile material is heated at a lower temperature, for example in the range 50° C. to 120° C. Alternatively, the heating operation may optionally be deferred until after the textile material has been made up into a garment. The resin-treated textile material may optionally also be subjected to a mild washing treatment to remove residual formaldehyde and/or acid.

The treatment of the textile material with hot water or steam immediately following the application of the solution of the resin precondensate and the acid catalyst is an essential part of the process of the invention. Not only does this treatment serve to remove the halogenated hydrocarbon solvent by violatilisation, but it also brings about an unexpected enhancement of the desirable properties of the textile material which are conferred by the process. If the halogenated hydrocarbon solvent is removed from the textile material by the application of dry heat, the beneficial effects of the treatment are very much diminished.

The concentration of the resin precondensate in the solution which is applied to the textile material may be varied within wide limits, but in general a concentration of from 3 percent to 20 percent by weight of the solution is preferred. The amount of the acid catalyst present in the solution is normally from 5 percent to 20 percent of the weight of the resin precondensate.

The amount of the resin precondensate which is applied to the textile material by the process of the invention may be varied according to the magnitude of the treatment effect which it is desired to achieve, but in general an amount of from 2.5 percent to 15 percent of the dry weight of the textile material is preferred.

The process of the invention may with advantage be carried out in such a way that the halogenated hydrocarbon solvent is recovered for reuse after its removal from the textile material by hot water or steam. In the case where hot water is employed for removal of the solvent, a solvent recovery system such as that described in British Pat. specification No. 812,894 may be used. When the solvent is removed by the preferred method of treating with steam, the solvent may conveniently be recovered in conventional manner, for example by condensation of the vapors by means of a condenser positioned inside or outside the flash-off vessel, the condensate being passed to a water separator to remove water from the liquid solvent, as described, for example, in our copending application No. 42620/66. In this way loss of solvent vapor to the atmosphere can be obviated, and the resulting liquid solvent can be reused in the treatment of further textile material.

Cellulosic textile materials which may be treated according to the process of the invention include fibers, yarns and fabrics containing at least 20 percent by weight of cellulosic material. It is preferred to treat such textile materials which are composed wholly of cellulose, for example, cotton, viscose rayon (including rayon made from polynosic fibers) and linen. The process may, however, be applied to textile materials which are blends of cellulosic material with a noncellulosic material, for example a synthetic polymeric material such as a polyester, such blends containing at least 20 percent by weight, and desirably 50 percent by weight, of cellulosic material. Treatment of such textile materials by the process of the invention results in a substantial improvement in the crease-resistance and easy-care properties of the materials.

There may be added to the solution in the halogenated hydrocarbon solvent of the resin precondensate and the acid catalyst as hereinbefore defined other textile treatment agents of known type which are soluble in the said solvent. Such other treatment agents include lubricants and softeners, for example low molecular weight polyethylene waxes, water repellent agents, for example silicones, methylolstearamide and stearamidomethyl pyridinium chloride, and stiffening agents, for example polyvinyl acetate.

The invention is illustrated but not limited by the following Examples, in which parts and percentages are by weight:

EXAMPLE 1

A mercerized cotton poplin fabric is impregnated in a solution of 6.2 parts of polymethoxymethylmelamine, prepared as described below, and 0.6 part of monochloracetic acid in 93 parts of trichloroethylene. The fabric is then passed through squeeze rollers at such a pressure that the amount of trichlorethylene solution retained by the fabric is 65 percent of its dry weight. It is attached to a frame and immersed in a steam chamber at 95° C. for 30 seconds in order to remove the solvent. The fabric is then heated at 150°–155° C. for 3 minutes.

The resulting fabric is found to possess good crease resistance (when measured by British Standard Method of Test B.S. 3086:1959) and superior smooth-drying performance to the untreated cotton. This improvement is illustrated by the following crease recovery figures:

	Untreated fabric	Treated fabric
Crease recovery angle (warp + weft)	174°	268°

If the process is carried out by removing the trichloroethylene from the fabric simply by heating, i.e. without contact with water or steam, the treated cotton possesses only slightly superior crease resistance to the untreated fabric. A piece of fabric treated in this way is found to possess a crease recovery angle (warp + weft) of 183°.

After washing the treated and untreated cotton for 1 hour at 95° C. in a solution of 0.1 percent soap and 0.1 percent soda ash, the crease recovery figures obtained are as follows:

	Untreated fabric	Treated fabric
Crease recovery angle (warp + weft)	171°	265°

The finish is therefore durable to the above washing treatment.

The solvent-soluble polymethoxymethylmelamine used in the above treatment is prepared as follows:

126 Parts of melamine are added to 412 parts of a 51 percent *w/w* methanolic formaldehyde solution which have been basified by the addition of 7.2 parts of 47 percent *w/w* aqueous caustic soda solution. The mixture is stirred and heated to reflux temperature, the mixture then being refluxed for one-half hour. The mixture is cooled to room temperature, 191 parts of methanol and 52 parts of concentrated hydrochloric acid are added, and the mixture is stirred at 25–30° C. during 1½ hours. The solution is basified to pH 7.8 by the addition of 40 parts of anhydrous sodium carbonate and the mixture is filtered from precipitated salts. The clear filtrate is evaporated under reduced pressure, the final conditions being 1 hour at 90° C. and 15 mm. mercury pressure. The product is filtered hot from a further small amount of precipitated salts, yielding a clear viscous liquid product which is readily soluble in halogenated hydrocarbon solvents.

EXAMPLE 2

A mercerized cotton poplin fabric is impregnated with a solution containing 6.2 parts of methoxymethylurea, prepared as described below, and 0.77 part of stearamidomethyl pyridinium chloride in 93 parts of trichloroethylene. The fabric is squeezed through a pad mangle so that the amount of solution it retains is 65 percent of its own weight. The solvent is then removed from the fabric by attaching the fabric to a frame and immersing the frame in a steam chamber at 95° C. for 30 seconds. The fabric is finally heated at 150° C. for 3 minutes.

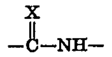
The treated cotton is found to possess a crease recovery angle (warp+weft) of 223°, while that of the untreated is 136°.

The solvent-soluble methoxymethylurea used in the above treatment is prepared as follows:

180 Parts of urea are added to 794 parts of 51 percent *w/w* methanolic formaldehyde solution which have been basified by the addition of 10.8 parts of 47 percent *w/w* aqueous caustic soda solution. The solution is stirred at 60–65° C. during 3 hours and cooled to room temperature. The solution is acidified to pH 2 by the addition of about 10 parts of concentrated hydrochloric acid, the solution then being stirred at 25–30° C. during 2 hours. The solution is basified to pH 9.5 by the addition of about nine parts of 47 percent aqueous caustic soda solution and is then evaporated under reduced pressure, the final conditions being 1 hour at 90° C. and 15 mm. mercury pressure. The product is filtered from a small amount of precipitated salts, yielding a clear liquid product which is readily soluble in halogenated hydrocarbon solvents.

What we claim is:

1. A process for the treatment of textile materials containing at least 20 percent by weight of cellulosic material which comprises the steps of (a) impregnating the said textile material with a liquor comprising (i) a halogenated aliphatic hydrocarbon solvent, (ii) a resin precondensate which is an initial stage condensation product of formaldehyde with a compound containing the grouping



$$-\overset{\overset{\displaystyle X}{\|}}{C}-NH-$$

where X is oxygen, sulphur or an imino group and which is capable of reacting with itself under acid conditions to give a fully cured resin and/or is capable of reacting with the hydroxyl group present in the said cellulosic material, and (iii) a catalyst for the said resin precondensate selected from acids and acid-generating substances, (b) immediately after the said impregnation treating the textile material with an aqueous agent selected from hot water and steam, and (c) thereafter heating the textile material at 120° to 200° C. for a period of from 10 seconds to 30 minutes.

2. A process as claimed in claim 1, wherein the resin precondensate is an alkyl ether of a methylol derivative selected from the methylol derivatives of urea, thiourea, dicyandiamide, an amide, a carbamate, an aminotriazine, a uron, a urein, a ureide, an imidazolidimone, a pyrimidinone and a triazinone the alkyl group of the alkyl ether containing from one to four carbon atoms.

3. A process as claimed in claim 2, wherein the resin precondensate is selected from methoxymethylurea, polymethoxy-methylmelamine, 1,3-dimethoxymethyl-4,5-dihydroxy-imidazolidin-2-one and 1,3-dimethoxymethyl-hexahydropyrimidin-2-one.

4. A process as claimed in claim 1, wherein the halogenated hydrocarbon solvent is selected from trichloroethylene, perchloroethylene and 1;1;1-trichloroethane.

5. A process as claimed in claim 1, wherein the catalyst is an acid selected from monochloroacetic acid, dodecylbenzenesulphonic acid and 2,2-(bishydroxmethyl)-butylphosphonic acid.

6. A process as claimed in claim 1, wherein the catalyst is an acid-generating substance selected from methyl p-toluene sulphonate, cetyl pyridinium bromide and stearamidomethylpyridinium chloride.

7. A process as claimed in claim 1, wherein the textile material is treated, after said impregnation, with water at a temperature of from 80° to 100° C. for a period of from 5 secs. to 60 secs.

8. A process as claimed in claim 1, wherein the textile material is treated, after impregnation, with steam at atmospheric pressure, so that the textile material attains a temperature of from 80° to 100 ° C. for a period of from 5 secs. to 60 secs.

9. A process as claimed in claim 1, wherein the textile material is heated for a period of from 1 to 5 minutes.

10. A process as claimed in claim 1, wherein the concentration of the resin precondensate in the treatment liquor which is applied to the textile material is from 3 percent to 20 percent by weight of the liquor.

11. A process as claimed in claim 1, wherein the amount of catalyst present in the treatment liquor is from 5 percent to 20 percent of the weight of the resin precondensate.

12. A process as claimed in claim 1, wherein the amount of the resin precondensate which is applied to the textile material is from 2.5 percent to 15 percent of the dry weight of the textile material.

13. A textile material treated by a process as claimed in claim 1.

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