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TEXTILE MATERIAL AND PROCESS OF TREATING THE SAME

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The present invention relates to porous, non-dusting, foraminous textile materials having a soft hand and drape along with enhanced wrinkle- and crease-resistance and to methods of making the same. More specifically, the present invention relates to improved porous, non-dusting, foraminous textile materials and to improved methods of making the same, involving the application to the textile materials of textile-treating compositions containing wrinkle- and crease-resistance imparting resins in substantially monomeric form and in such amounts as to provide textile materials having a soft hand and drape along with enhanced wrinkle- and crease-resistance without lessening the porous nature of the foraminous textile material and without creating any dusting problems, even after said resins have been dried and cured in situ in the textile fabric.

Various synthetic plastics and resins are available in industry for textile-treating and textile-coating purposes. Among the more commonly used of such treating and coating materials are the urea-formaldehyde resins. The present invention will be further described in greater detail by comparison to such urea-formaldehyde resins but such has been done primarily only for illustrative and comparative purposes.

Urea-formaldehyde resins are normally prepared industrially in monomeric form as a paste to be sold as a standard commodity of commerce. Such a paste, however, is not completely chemically stable and continually undergoes gradual polymerization from the monomeric to the polymeric form of the resin. As a result, the monomeric nature and content of the paste is continuously decreasing whereas its polymeric nature and content is continuously increasing from the very moment it is originally made. As a consequence, when the commercially available urea-formaldehyde paste is applied as a treating or coating material to a textile fabric, the exact nature of the applied material will vary from one treatment to the next treatment depending upon the freshness or age of the resin.

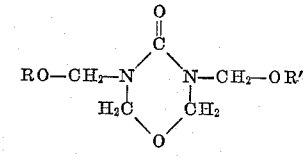
As a consequence, the resulting properties and characteristics of the treated fabrics will unpredictably vary from one lot to another and there will be no positive assurance of the precise properties and characteristics of a particular fabric. Additionally, if the resin has polymerized beyond a certain extent, impregnation of the fabric is not thorough; adherence to the fabric is not as good as desired; and many objectionable features, notably dusting and reduction of the porous nature of the foraminous textile material result.

It is the principal purpose of the present invention to provide a porous, non-dusting, resin-treated foraminous textile material having a soft hand and drape along with enhanced wrinkle- and crease-resistance and to provide for methods of making such textile materials.

This principal purpose may be accomplished by preparing a textile-treating composition by mixing a formaldehyde-binding material with an aqueous solution of an N,N'-dimethyloluron dialkyl ether and, before any substantial polymerization can take place in the textile-treating composition, applying the same to a textile material in an amount within a specified weight range, and then drying and curing the textile-treating composition in situ on the textile material whereby it is rendered substantially non-dusting and possesses a soft hand and drape along

with enhanced wrinkle- and crease-resistance without deleteriously affecting its porosity and foraminous nature.

The N,N'-dimethyloluron dialkyl ethers used in the application of the present invention have the following general structural formula:



wherein R and R' are alkyl groups containing from 1 to 4 carbon atoms.

These N,N'-dimethyloluron dialkyl ethers may be obtained by methods known to industry. Generally, such methods involve the reaction of 1 mol of urea with at least 4 mols of formaldehyde at elevated temperatures, in the presence of an alkaline material, whereby a tetramethylol urea is believed to be formed, and then converting the same by the action of an alcohol in the presence of an acidic material to an N,N'-dimethyloluron dialkyl ether.

The formaldehyde-binding material may be selected from a wide group of materials capable of reacting or combining with formaldehyde. For the purposes of illustration, urea will be used to describe the present invention in greater detail. It is to be appreciated that this is illustrative and not limitative and that other formaldehyde-binding materials such as dicyandiamide, biuret, thiourea, ammonia, melamine, guanidine, or mixtures, derivatives, or substitution products thereof, may be used.

The proportions by weight of the N,N'-dimethyloluron dialkyl ether (hereinafter called "UFL Base") and the urea which are mixed to prepare the resin constituent of the textile-treating composition may be varied within relatively wide limits, depending upon the purpose and use of the resulting textile-treating composition. Inasmuch as the UFL Base is normally prepared and is available in approximately 66% solids solution, the term "UFL Base" hereinafter shall mean such a solution. A weight ratio of UFL Base solution:urea of from about 1½:1 to about 8:1 has been found satisfactory for the purposes of the present invention. Within the more commercial aspects of the present invention, however, ratios of from about 2:1 to about 4:1 have been found most desirable. These ratios, when couched in terms of N,N'-dimethyloluron dialkyl ether:urea, are 1:1 to about 5⅓:1 and 1⅓:1 to about 2⅔:1, respectively.

The concentration of resin constituent (UFL Base and urea) in the textile-treating composition may be varied within relatively wide ranges depending, for example, upon the apparatus and method of application to the textile fabric. Within the broader aspects of the present invention, a concentration of from about 5% to about 40% by weight of combined UFL Base and urea has been found acceptable, with from about 10% to about 30% by weight being preferred.

If desired, other additives may be included in the textile-treating composition during its compounding. For example, curing catalysts such as zinc nitrate, monoethanolamine hydrochloride, 2-amino-2-methyl-1-propanol hydrochloride; surface resins (high polymeric type) such as dimethylol urea syrups, or methylol melamine or its methyl ether derivatives, or dimethylol urea dimethyl ether; softening agents such as anionic-type sulfonated vegetable oils, or nonionic-type ethylene oxide-fatty alcohol condensation products, or cationic dioctadecyl dimethyl ammonium chloride; may be used. Other additives which can be included, as desired or required, are binders, dyes, pigments, flame-retardant agents, moth- or mildew-proof-

ing materials, wet strength agents, insecticides, germicides, disinfectants, etc.

The textile materials to which the textile-treating materials may be applied are preferably woven fabrics but knitted, felted or braided fabrics are also applicable, as well as nonwoven fabrics, lace, etc. These textile materials may be made basically of any type of fiber including natural vegetable fibers such as cotton and flax; natural animal fibers such as wool; silk; synthetic or man-made fibers including the cellulosics such as regenerated cellulose, cellulose acetate; the polyamide fibers such as nylon 66 and nylon 6; polyester fibers such as "Dacron"; acrylic fibers such as "Dynel" and "Acrilan"; vinyl fibers such as "Vinyon"; etc. or mixtures and blendes of such fibers.

The textile treating composition must be applied to the textile fabric as soon after formulation as possible. This is necessarily so because, although the UFL Base solution and the urea, by themselves, are stable over long periods of time, once they are mixed together and the added urea reacts with the free formaldehyde in the UFL Base, polymerization will commence.

If there is too long a delay between the formulation of the textile-treating composition and its application to a textile material, the amount of monomer present will undesirably decrease whereas the polymer content will correspondingly undesirably increase in the textile-treating composition. As a consequence, if the percentage of polymer in the textile-treating composition has risen to a substantial figure, satisfactory application and penetration of the textile-treating composition is rendered extremely difficult and perhaps impossible.

This undesirable state of affairs is notably so during summer months when the textile-treating composition is necessarily prepared and stored at higher room temperatures than prevail in the wintertime. For example, textile-treating compositions of the present invention may be safely stored up to several days under lowered temperature conditions. On the other hand, it has been found that storage for less than a day or so under elevated temperatures may be sufficient to create sufficient polymerization as to render the composition unacceptable.

The important determination is therefore not only the length of time which has elapsed between preparation and application to the textile fabric but also the degree of polymerization which has taken place. Degrees of polymerization of greater than about 30%–40% have been found to be unacceptable for the purposes of the present invention.

The textile-treating composition may be applied to the textile materials by various methods known in industry. For example, they may be applied either by spraying, immersion, or by contact, either with or without the assistance of padding, nip or squeeze rolls or other types of extracting equipment. Inasmuch as a wide choice may be made in the relative proportions of the constituents of the textile-treating composition, it is possible to use all types of apparatus in applying the compositions to textile materials.

The percent wet pick-up referred to herein is a measure of the amount of liquid treating composition, by weight, which is picked up and retained by the textile materials. For example, 100% wet pick-up means that the textile materials have picked up an amount by weight of the textile-treating composition equal to the weight of the textile materials. The percent dry add-on referred to herein is a measure of the amount of dry resin solids which are picked up and retained on the textile materials after the contained water and other volatile materials have been driven off during heating or drying and curing.

The amount of dry add-on is critical and it has been found that, if dusting is to be avoided, and proper adherence of the textile resin to the textile fabric is desired, amounts less than 20% by weight be used. If the amount of dry add-on is too high, such will have an undesirable effect upon the porosity and foraminous nature of the textile fabric. Within the broader aspects of the present invention from about ½% to about 20% has been found desirable. Within the more commercial aspects of the present invention, from about 1% to about 15% by weight has been found preferable.

The invention will be further illustrated in greater detail by the following specific examples. It should be understood, however, that although these examples may describe in particular detail some of the more specific features of the invention, they are given primarily for purposes of illustration and the invention in its broader aspects is not to be construed as limited thereto. The percentages indicated are by weight, unless specifically stated otherwise.

Example I

N,N'-dimethyloluron dimethyl ether is prepared as follows: 120 pounds of urea is dissolved with stirring in 3330 pounds of U.F. Concentrate-85 (59% formaldehyde; 26% urea; formaldehyde:urea mol ratio—4.6:1; 15% water). Approximately 5 pounds of caustic soda (50° Baumé; specific gravity 1.53) is added with stirring to adjust the pH to about 10. The temperature of the reaction mixture is raised to about 210° F. with stirring and maintained at that temperature for abou 15 minutes. A reaction takes place and N,N'-polymethylol urea (presumably N,N'-tetramethylol urea) is formed. The reaction mixture is allowed to cool to about 90° F. During the cooling period about 1430 pounds of methanol is added. About 93 pounds of technical hydrochloric acid (20° Baumé; specific gravity 1.16; 31.45% HCl) is added; the pH drops to about 1; and an exothermic reaction takes place wherein it is believed the uron ring is formed to yield N,N'-dimethyloluron dimethyl ether.

The reaction mixture is neutralized to a pH of about 7 with about 35 pounds of caustic soda (50° Baumé; specific gravity 1.53). The neutralized solution is clear, water-white and stable, containing about 66% solids and about 12% free formaldehyde. This solution is referred to herein as "UFL Base."

A textile-treating solution having the following composition:

15 parts UFL Base (66% solids)
5 parts urea (crystals)
1 part catalyst RP (amine hydrochloride)
79 parts water

is applied to a 100% viscose rayon 80 x 80 woven fabric. The application is made within three hours of the mixing and substantially no polymerization of the UFL Base and urea solution takes place during that time. The application is made by passing the fabric against an immersion-type coating roller. The wet pick-up is about 70% of the weight of the fabric. The treated fabric is dried on a tenter frame to a moisture content of less than 5% and then cured at a temperature of about 300° F. for a period of about 5 minutes. The dry add-on is about 14% of the weight of the fabric.

The resin-treated fabric is porous and foraminous and is substantially non-dusting and has a relatively soft hand and drape. It possesses excellent wrinkle- and crease-resistance along with good light fastness and resistance to light fading. Dimensional stability is good.

Example II

N,N'-dimethyloluron dimethyl ether is prepared as follows: 90 pounds of urea is dissolved with stirring in 2500 pounds of U.F. Concentrate-85. Sodium hydroxide solution (50% strength) is then added to bring the pH of the solution to about 9½. With good agitation, the solution is heated to about 215° F. and maintained at that temperature for about 10 minutes. The solution is then allowed to cool to about 115° F. and about 1074 pounds of methanol is added. The solution is then further cooled to about 75° F. and about 85 pounds of hydrochloric acid (20° Baumé) is added. The reaction which takes

place is exothermic and the temperature rises to about 115° F. About 5 minutes after the maximum temperature is reached, the reaction is stopped by the addition of sufficient sodium hydroxide solution to bring the pH to about 7½. The final resin solution contains about 15% free formaldehyde. The percent active ingedients in this solution is about 62%.

A textile-treating solution is prepared having the following composition:

16 parts N,N'-dimethyloluron dimethyl ether solution
4 parts urea
1 part 2-amino-2 methyl-1 propanol hydrochloride (33% solution)
79 parts water

This textile-treating solution is applied to a 100% viscose challis on a padder with a wet pick-up of about 60% within 24 hours of its preparation. The degree of polymerization of the resin is less than 30%. The impregnated fabric is then frame-dried and cured in an oven at a temperature of about 320° F. for about 3 minutes. The resin-treated fabric is porous and foraminous and possesses a smooth and silky hand, along with good wrinkle-resistance and stabilization against shrinkage. Substantially no dusting of the resin-treated fabric is noted.

Example III

An N,N'-dimethyloluron dimethyl ether solution is prepared substantially as set forth in Example II.

A textile-treating solution having the following composition is prepared:

18 parts N,N'-dimethyloluron dimethyl ether solution
6 parts urea
2 parts dimethylol urea dimethyl ether
1 part dioctadecyl dimethyl ammonium chloride (75% active)
1 part dioctadecyl dimethyl ammonium chloride (75% solution)
72 parts water

The above textile-treating solution is applied to a 50% viscose-50% cellulose acetate gabardine on a padder so as to provide about 70% wet pick-up. The application of the textile-treating solution is made within 36 hours of its preparation. The degree of polymerization of the resin is less than 30%. The impregnated fabric is frame-dried and is cured at a temperature of about 300° F. for about 5 minutes. The treated fabric shows excellent wrinkle-resistance and stabilization against shrinkage. Substantially no dusting of the resin-treated fabric is noted.

Examples IV–IX

The procedures of Example II are followed substantially as set forth therein except that the following fabrics are treated: (*a*) a 100% cotton 80 x 80 woven fabric; (*b*) a 100% linen fabric; (*c*) a 400 grain per square yard nonwoven fabric containing 50% cotton fibers and 50% viscose rayon fibers, 1½ denier, 1½ inches staple length; (*d*) a polyamide (nylon 66) woven fabric; (*e*) an acrylic ("Acrilan") woven fabric; and (*f*) a cellulose acetate ("Arnel") woven fabric.

The results are comparable to those obtained in Example II except that it is noted that the shrinkage control and dimensional stability obtained in the case of the cellulosic fabrics is superior to that obtained for the non-cellulosic synthetic-fiber fabrics.

Examples X–XII

The procedures set forth in Example II are followed substantially as set forth therein except that the amounts of resin add-on, determined after drying and curing, are varied as follows: (*a*) 1%; (*b*) 10%; (*c*) 20%; and (*d*) 40% of the weight of the fabric.

The first three examples yield fabrics which are basically comparable to the fabric obtained for Example II. The last example (40%), however, is not satisfactory in that the treated fabric is embrittled and there is excessive dusting. All in all, the fabric is not commercially acceptable for many purposes.

Examples XIII–XIV

The procedures of Example II are carried out substantially as set forth therein except that after the urea is added to the UFL Base, different waiting periods are observed before application to the textile fabric. During this waiting period, the textile-treating solution is kept at room temperature (summer temperature—average about 77° F.) and normal atmospheric pressure.

The waiting periods are: (*a*) 1 day; (*b*) 2 days; and (*c*) 10 days. The first two examples have degrees of polymerization of less than 30% and provide commercially acceptable resin-treacted fabrics, with the fabric of the first example (*a*) being superior to the fabric of the second example (*b*). The third example (*c*), however, has a degree of polymerization greater than 30% and is commercially unacceptable. Study of the resin and the fabric reveals that sufficient polymerization has taken place prior to fabric treatment to prevent a satisfactory application of the resin thereto.

Although several specific examples of the inventive concept have been described, the same should not be construed as limited thereby nor to the specific features mentioned therein but to include various other equivalent features as set forth in the claims appended hereto. It is understood that any suitable changes, modifications and variations may be made without departing from the spirit and scope of the invention.

What is claimed is:

1. A method of improving the properties of a porous, foraminous textile material which comprises preparing a textile-treating composition by mixing a hydrophilic, water-soluble formaldehyde-binding material with an aqueous alkaline solution of a N,N'-dimethyloluron dialkyl ether in proportions, respectively, of from about 1:1 to about 5⅓:1, said aqueous alkaline solution of N,N'-dimethyloluron dialkyl ether containing at least about 12% free uncombined formaldehyde to react with said hydrophilic, water soluble formaldehyde-binding material and, before any substantial polymerization takes place in said textile-treating composition, applying the same to a textile material in an amount sufficient to provide from about 0.5% by weight to about 20% by weight dry add-on after heating and curing, and heating and curing said textile-treating composition in situ on said textile material whereby the same is rendered substantially non-dusting and possesses a soft hand and drape along with enhanced wrinkle- and crease-resistance.

2. A non-dusting, foraminous textile material having a soft hand and drape along with enhanced wrinkle- and crease-resistance made by the method of claim 1.

3. A method of improving the properties of a porous, foraminous textile material which comprises preparing a textile-treating composition by mixing a hydrophilic, water-soluble formaldehyde-binding material with an aqueous alkaline solution of a N,N'-dimethyloluron dimethyl ether in proportions, respectively, of from about 1:1 to about 5⅓:1, said aqueous alkaline solution of N,N'-dimethyloluron dimethyl ether containing at least about 12% free uncombined formaldehyde to react with said hydrophilic, water soluble formaldehyde-binding material, and, before any substantial polymerization takes place in said textile-treating composition, applying the same to a textile material in an amount sufficient to provide from about 0.5% by weight to about 20% by weight dry add-on after heating and curing, and heating and curing said textile-treating composition in situ on said textile material whereby the same is substantially non-dusting and possesses a soft hand and drape along with enhanced wrinkle- and crease-resistance.

4. A method of improving the properties of a porous, foraminous textile material which comprises preparing a textile-treating composition by mixing a hydrophilic, water soluble formaldehyde-binding material with an aqueous alkaline solution of a N,N'-dimethyloluron dialkyl ether in proportions, respectively, of from about 1:1 to about 5⅓:1, said aqueous alkaline solution of N,N'-dimethyloluron dialkyl ether containing from at least about 12% to about 15% free uncombined formaldehyde to react with said hydrophilic, water soluble formaldehyde-binding material, and, before any substantial polymerization takes place in said textile-treating composition, applying the same to a textile material in an amount sufficient to provide from about 0.5% by weight to about 20% by weight dry add-on after heating and curing, and heating and curing said textile-treating composition in situ on said textile material whereby the same is rendered substantially non-dusting and possesses a soft hand and drape along with enhanced wrinkle- and crease-resistance.

5. A method of improving the properties of a porous, foraminous textile material which comprises preparing a textile-treating composition by mixing a hydrophilic, water soluble formaldehyde-binding material with an aqueous alkaline solution of a N,N'-dimethyloluron dimethyl ether in proportions, respectively, of from about 1:1 to about 5⅓:1, said aqueous alkaline solution of N,N'-dimethyloluron dimethyl ether containing from at least about 12% to about 15% free uncombined formaldehyde to react with said hydrophilic, water soluble formaldehyde-binding material, and, before any substantial polymerization takes place in said textile-treating composition, applying the same to a textile material in an amount sufficient to provide from about 0.5% by weight to about 20% by weight dry add-on after heating and curing, and heating and curing said textile-treating composition in situ on said textile material whereby the same is substantially non-dusting and possesses a soft hand and drape along with enhanced wrinkle- and crease-resistance.

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