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TEXTILE FINISHING COMPOSITION COMPRISING A METHOXYMETHYL MELAMINE AND AN ALIPHATIC ALCOHOL HAVING AT LEAST EIGHT CARBON ATOMS

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This invention relates to the treating and finishing of textiles and textile-forming materials. The invention includes processes for finishing textiles, textile finishing compositions, the preparation of such textile finishing compositions, and textiles of improved properties finished with the compositions and by the processes of the invention.

Textile fabrics, yarns, threads and fibers have been heretofore treated with a wide variety of creaseproofing agents, waterproofing agents, materials to prevent shrinking, felting, fulling, etc., lubricants, softening agents, sizes, binding agents and other materials to improve the appearance and "feel" or hand of the goods. The treatment of textiles for these and similar purposes is known as textile finishing, and the agents employed for this purpose are known as textile finishing agents. It is a principal object of the present invention to provide textile finishing compositions and methods for their preparation and application to textiles, which compositions and methods will produce finished textiles having a greatly improved water resistance, a more desirable hand, crease-resistance, a very much reduced tendency for shrinking and other desirable improvements which will be apparent from the description which follows. Other important objects will appear hereinafter.

The various textile materials which may be treated in accordance with my invention include fibers, thread, yarns, knit and woven cloth, and other fabricated materials of cotton, linen, hemp, jute, regenerated cellulose, cellulose esters, cellulose ethers, wool, synthetic wool, silk, synthetic silk and other fibrous materials whether natural or synthesized. Hereinafter and in the claims for purposes of brevity all of these various textile-forming materials and structures containing the same will be referred to as textile fabrics.

Although textile fabrics having a greatly reduced tendency to shrink as well as crease-resistance and an improved hand may be obtained by treatment with aqueous dispersions of alkylated methylol melamine followed by drying and curing the resin in the fabric, fabrics so treated have but a fair resistance to the penetration of water and therefore leave much to be desired insofar as their water-repellent characteristics are concerned. By treatment of textile fabrics with compositions such as will be presently described I am enabled to retain all of the advantages of shrink-resistance, crease-resistance, improved hand, etc. which are obtainable by treatment of the fabric with lower alkylated methylol melamines and in addition impart to such fabrics a very high degree of water-repellency.

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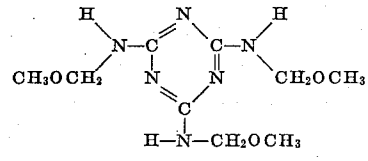
My improved textile finishing process includes the steps of treating textile fabrics such as mentioned above with dispersions of alkylated methylol melamine and a higher aliphatic alcohol followed by drying and curing these resin-forming constituents on the fabric. For example, I may treat the textile fabric with an aqueous dispersion containing approximately 10 parts of methylated methylol melamine and 1.5 parts of octadecyl alcohol followed by an appropriate heat treatment. The improved results which I obtain are due, I believe, to certain chemical reactions which take place during the drying and curing treatment between the alkylated methylol melamine and the higher alcohol.

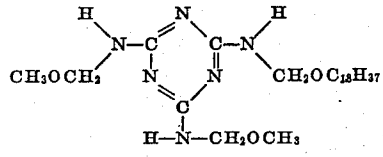
When a textile fabric treated with a methylated methylol melamine having a structure such as



is subjected to a heat treatment the methylated methylol melamine polymerizes, or cures, in the fabric. Curing of the resin takes place by splitting off one or more molecules of methyl alcohol followed by a molecular rearrangement which favors polymerization. While, as stated before, fabrics so treated have a very marked resistance to shrinking, a good hand, crease-resistance and other important characteristics the fabrics are not particularly water-repellent. This lack of water-resistance is due in part at least to the fact that the polymerized resin molecule has no effective hydrophobic groups available to repel water. In other words the cured resin is fairly easily wetted and water is allowed to penetrate into the interstices of the fabric. By adding to the alkylated methylol melamine textile treating composition a higher aliphatic alcohol and then impregnating, drying and curing the fabric under conditions presently to be described I believe that a chemical reaction takes place between the alkylated methylol melamine and higher alcohol which modifies the nature of the cured resin yet does not materially influence the polymerization mechanism necessary to obtain a permanent water-insoluble, cured resin in the fabric.

This reaction, which is in the nature of an ether interchange probably takes place between the

methylated methylol melamine to produce intermediate compounds having the probable structure:



in which at least one of the short chain CH_3— groups is replaced with a long chain alkyl group. It will also be noted that the above compound also contains at least one methoxymethyl group which at the temperature of the curing process can split off methanol allowing a molecular rearrangement to take place which favors polymerization with other alkylated methylol melamine molecules. The long chain alkyl residue of the higher alcohol is not split off at the temperature of the curing process. The polymerized compound is of course water-insoluble and the long alkyl chain attached thereto renders the resin hydrophobic and water-repellent. It is to be clearly understood that the above discussion is merely a suggested mechanism of what takes place in my finishing process and I do not intend to be bound thereby since regardless of whatever theory may be advanced the superior results obtained by my process remain as a distinct advance in the art.

From the above considerations it will be seen that I may employ any alkylated methylol melamine having at least two alkylated methylol groups, one for reaction with the higher aliphatic alcohol and the other providing a point at which polymerization may proceed. I may, of course, employ alkylated methylol melamines having more than two alkylated methylol groups and ordinarily do so.

It should also be understood that my invention in its broader aspects is not limited to the use of methylated methylol melamines as illustrated above. Alkylated methylol melamines having short chain alkyl groups of not more than 4 carbon atoms such as ethylated, isopropylated and butylated methylol melamines may also be employed but require greater care in the curing steps. Ethyl alcohol and butyl alcohol split off during the curing steps with greater difficulty and a proper cure is more difficult to obtain without employing temperatures which might damage the fabric.

The alkylated methylol melamines which I employ are prepared by known methods. Methylated methylol melamine may, for example, be prepared by reacting 2 to 6 moles of formaldehyde with 1 mole of melamine to form a condensation product believed to be mostly methylol melamine. This product is then reacted with 2 to 6 moles of methanol whereby methylated methylol melamine is formed. By the term methylated methylol melamine I intend to include all of these various reaction products containing from 2 to 6 methylated methylol groups per molecule. Excessive polymerization should be avoided during the preparation since the resin is preferably employed by me in the form of an aqueous dispersion, or solution, and products polymerized excessively tend to be difficultly soluble in water. I have also found that excessive polymerization of the product tends to give the fabrics a stiff hand.

The higher alcohols which I add to my new textile finishing composition are those primary,

secondary and tertiary aliphatic alcohols having at least 8 carbon atoms per molecule in order that the cured resin in the fabric will possess hydrophobic characteristics sufficient to impart to the textile fabric a high degree of water resistance. I have for example employed alcohols such as ethyl hexanol, dodecanol, tetradecanol, hexadecanol, octadecanol and the like. A particularly advantageous higher alcohol suitable for use in my textile finishing composition because of its low cost is a commercially obtainable mixture of higher alcohols known to the trade as "Lorol" which consists predominantly of lauryl alcohol with smaller percentages of both higher and lower aliphatic alcohols.

Although the structural formula given above indicates that one mole, or even more, of the higher alcohol may be employed with each mole of alkylated methylol melamine I have found that it is not necessary to use such a high proportion of higher alcohol to obtain satisfactory waterproofing of textile fabrics. In general, I have found that the water-resistant characteristics of the treated fabrics do not improve appreciably when more than 5 parts of higher alcohol is used with each 10 parts by weight of the alkylated methylol melamine. Ordinarily I employ about 1.5 parts by weight of the higher alcohol for each 10 parts by weight of the methylated melamine in my finishing composition. Smaller proportions of the higher alcohol may be used with, however, a decrease in the water-resistance of the treated fabric. One-half part by weight of the higher alcohol for each 10 parts by weight of the alkylated methylol melamine will form a finishing composition capable of rendering most textile fabrics fairly water-resistant. All the advantages of shrink-resistance, crease-resistance, improved hand, etc. obtained by treatment with methylated methylol melamine alone are of course imparted to the fabric as a result of my treatment whether the proportion of higher alcohol be either high or low in the finishing composition.

The alkylated methylol melamine-higher alcohol composition which I employ in my process is ordinarily, and preferably, applied to the cellulose fabrics in the form of an aqueous dispersion containing from about 2–15% or more of the above materials. These dispersions may be prepared by simply stirring a suitable amount of the higher alcohol dissolved in a solvent such as ethyl alcohol, isopropyl alcohol, etc. into an aqueous disperson of the alkylated methylol melamine, both solutions being preferably at temperatures of 80° F. to 140° F. Dispersing agents may be used such as sodium isopropyl naphthalene sulfonate, dioctyl sodium sulfosuccinate, ammonium caseinate, gelatine, glue, gum arabic, etc. The preparation of these dispersions is illustrated in greater detail in the specific examples. The dispersions may be applied to the fabrics in various ways known to those in the art; thus, for example, the dried fabrics to be treated may be immersed in the resin dispersion and then passed through suitable rolls as in a padder or mangle to secure uniform impregnation and to remove excessive resin. The fabric, however, may be impregnated by other methods such as by spraying or with suitable boxes located on the mangle. My invention is not limited to any particular method of impregnating the fabrics and other methods will occur to those skilled in the art.

The amount of my alkylated methylol melamine-higher aliphatic alcohol finishing com-

position which may be applied to fabrics may vary considerably depending upon the nature of the fabric treated and the particular finish desired. Fabrics of wool, silk, cotton, etc. may be rendered water-repellent and substantially resistant to shrinkage by the application of from about 2 to 8% by weight of the resin-forming constituents based on the dry weight of the fabric. Smaller amounts down to 0.5% may be used with less effective results. Larger amounts as for example 8–14% by weight of the fabric will increase the water repellency of the fabric and also impart thereto a high degree of crease-resistance. With particular types of fabrics and to secure particular results it may be desirable to use larger amounts of my finishing composition up to 30% by weight of the alkylated methylol melamine-higher aliphatic alcohol mixture as based on its weight in the finishing composition. After the fabric has been impregnated it is dried and the fabric heated at elevated temperatures to polymerize the resin.

In order to speed up the curing of the resin and decrease the heating time a suitable catalyst may be added to the aqueous finishing composition. Oxalic acid, diammonium hydrogen phosphate and methyl acid pyrophosphate are particularly good for this purpose. Other catalysts such as triethanolamine phthalate, zinc chloride, acetic acid, mineral acids such as hydrochloric acid and others may also be used. Other catalysts for the curing of alkylated methylol melamine resins are know to chemists in the resin art and may be employed.

The curing temperatures are in general quite low anad may vary considerably from about 200° F. to about 300° F. with a corresponding reduction in time of cure with increase of temperature. The drying and curing operation is flexible and may be varied to suit the equipment available to the processes. High temperatures of the order of 280° F. to 300° F. will cure the resin to a suitably water-insoluble state in three or four minutes. Where facilities are not available for curing the resin at moderate temperatures within the range of about 240–280° F. the fabric after being impregnated with the desired amount of my finishing composition may be framed to width on a pin tenter, dried, batched up on a shell and allowed to stand hot to obtain a total drying and heating time sufficient to dry the cloth and insolubilizing the resin in the fabric. With some fabrics a drying and curing time of two or three hours may be required at 200° F. Drying and curing times will also depend to some extent upon the effectiveness of the particular accelerator employed and upon the nature of the fabric.

After the fabric has been treated as described it should, particularly in the case of woolen goods, be given a short mild soaping which renders it soft and pliable. The fabric may then be given other usual finishing treatments such as decatizing, brushing, sheering, pressing, etc. My process may be employed with both colored and uncolored goods without appreciably affecting the color or shade and without damage to the material.

My invention will now be illustrated in detail by the following specific examples which show representative compositions included herein and the water-repellent effects obtained thereby. It should be understood, however, that although these examples may describe some of the more specific details of the invention they are given primarily for illustrative purposes and the invention in its broader aspects is not limited thereto.

Example 1

960 parts by weight of formalin (37% by weight CH_2O) and 252 parts by weight of melamine were charged into a reaction vessel fitted with an agitator, thermometer, and reflux condenser and arranged so that it could be placed under a vacuum and heated. The contents of the reaction vessel were heated in one-half hour to a temperature of 62° C. and the pH adjusted to 7.5 with 2 normal NaOH. After heating for another one-half hour, a vacuum of 28 inches of mercury was applied and the mixture concentrated by removal of water. After 40 minutes heating at approximately 54° C. the heat and vacuum were removed and 1000 parts by weight of methyl alcohol containing enough 2 normal H_3PO_4 to neutralize the 2 normal NaOH added previously. The mixture at a pH of 6.4 was then heated to distill off an azeotropic mixture of methanol and water, anhydrous methanol being continuously added to replace the distillate. After heating in this way for about 6 to 7 hours, one part of the distillate would tolerate 100 parts of toluol and the resin was considered dried. It was then concentrated under a vacuum of 28 inches of mercury to a solids content of approximately 80%.

25 parts of the above-described 80% methylated methylol melamine, 20 parts of a 5% solution of sodium isopropyl naphthalene sulfonate and 135 parts of water were mixed and heated to approximately 100° F. 3 parts by weight of octadecyl alcohol dissolved in 48 parts by weight of isopropyl alcohol and having a temperature of 80–100° F. was poured into the methylated methylol melamine solution while stirring. A stable dispersion of fine particle size was obtained. To this dispersion was then added diammonium hydrogen phosphate in the amount of 4% based on the total resin solids.

Samples of 80 x 80 cotton percale were then impregnated with this dispersion by the dip and nip process, regulating the take-up so that the fabric contained approximately 11.5% by weight of the methylated methylol melamine-octadecyl alcohol constituents. The impregnated fabric was then dried and cured for seven minutes at 290° F. The treated cloth samples were then given a short soaping for ½ hour at 160° F. in a ½% soap solution, rinsed and dried. Other samples of the treated fabric were soaped at 160° F. in a .5% soap solution for four hours and 6 hours in order to establish the permanency of the water-repellent finish. Still other samples were cleaned in Stoddard solvent for two hours and four hours at room temperatures to determine the stability of the finish to dry-cleaning. The samples were then subjected to a standardized water-repellency test.

The water repellency test was conducted as follows: A 6 inch square of the cloth treated with the finishing composition was suspended 10 inches from a horizontal spray nozzle supplied with water at 80° F. under a hydraulic head of exactly 6 feet. The cloth was sprayed for one minute during which time 1.5 gallons of water were discharged through the nozzle. The cloth samples were then dried for 10 seconds, rolled between pieces of absorbent paper to remove surface water, and then weighed. The increase of weight was expressed as percent water absorbed, based on the original dry weight of the cloth. An un-

treated sample of the cloth absorbed from 90–100% of its weight of water. The results of these spray tests are as follows:

	Moisture Absorption in Per cent				
	Soaped at 160° F. in ½% Soap			Dry Cleaned in Stoddard Solvent (Varsol) at room temperature	
	30 min.	4 hr.	6 hr.	2 hr.	4 hr.
Sample #1	27	30	26	29	33
Sample #2	31	30	30	31	32

The results of these spray tests show the very effective water-proofing of the cloth samples as a result of my treatment and further show the high degree of permanency of the finish to both ordinary washing and dry-cleaning processes. It was also found that the cloth showed a very low degree of shrinking as a result of the washing.

Example 2

3 parts by weight of cetyl alcohol was dissolved in 7 parts by weight of ethyl alcohol by heating and the solution then cooled. This alcoholic solution was then poured with stirring into a dispersion of methylated methylol melamine containing 25 parts by weight of an 80% dispersion of methylated methylol melamine, 20 parts by weight of a 5% solution of isopropyl naphthalene sulfonic acid and 135 parts by weight of water. 10 parts of a 10% solution of diammonium hydrogen phosphate was then added to the dispersion. 80 x 80 cotton percale was impregnated with 11.5% of the dispersion (solids basis) by the dip and nip process. The fabric was then air-dried and cured four minutes at 290° F. After soaping for .5 hours at 160° F. in a .5% soap solution the fabric was found to absorb only 29.2% water when subjected to the spray test described in the previous example.

What I claim is:

1. A method of finishing textile fabrics which comprises impregnating the textile fabric with an aqueous dispersion containing 2 to 15% by weight of a mixture of a substantially unpolymerized, water-soluble methoxymethyl melamine containing at least two methoxymethyl groups and an aliphatic alcohol having at least 8 carbon atoms, said aliphatic alcohol being present in the dispersion in amounts corresponding to about 0.5 part to 5.0 parts by weight for each 10 parts by weight of methoxymethyl melamine, and thereafter heating the treated textile to insolubilize the resin-forming constituents therein.

2. A method of finishing textile fabrics which comprises impregnating the textile fabric with an aqueous dispersion containing 2 to 15% by weight of a mixture of a substantially unpolymerized, water-soluble methoxymethyl melamine containing at least two methoxymethyl groups and octadecyl alcohol, said octadecyl alcohol being present in the dispersion in amounts corresponding to about 0.5 part to 5.0 parts by weight for each 10 parts by weight of methoxymethyl melamine and thereafter heating the textile fabrics to insolubilize the resin-forming constituents therein.

3. A method of finishing textiles which comprises impregnating the textile fabric with an aqueous dispersion containing 2 to 15% by weight of a mixture of a substantially unpolymerized, water-soluble methoxymethyl melamine containing at least two methoxymethyl groups and an aliphatic alcohol having at least 8 carbon atoms, said aliphatic alcohol being present in the dispersion in amounts corresponding to about 0.5 to 5.0 parts by weight for each 10 parts by weight of methoxymethyl melamine and curing the resin-forming constituents within the range 200° F. to 300° F. to a water-insoluble state.

4. A textile finishing composition comprising an aqueous dispersion of a substantially unpolymerized, water-soluble methoxymethyl melamine containing at least two methoxymethyl groups and dodecyl alcohol, said dodecyl alcohol being present in the dispersion in amounts corresponding to about 0.5 part to 5.0 parts by weight for each 10 parts by weight of methoxymethyl melamine.

5. A textile finishing composition comprising an aqueous solution of a water-soluble methoxymethyl melamine containing at least two methoxymethyl groups, said solution having dispersed therein an aliphatic alcohol having at least 8 carbon atoms.

6. A textile finishing composition comprising an aqueous solution of a water-soluble methoxymethyl melamine containing at least two methoxymethyl groups, said solution having dispersed therein an aliphatic alcohol having at least 8 carbon atoms in amounts of about 0.5–5 parts by weight for each 10 parts of the methoxymethyl melamine.

7. A textile finishing composition comprising an aqueous solution of a water-soluble methoxymethyl melamine containing at least two methoxymethyl groups, said solution having dispersed therein about 0.5–5 parts by weight of octadecyl alcohol for each 10 parts of the methoxymethyl melamine.

8. A method of finishing textile fabrics which comprises impregnating the fabric with a composition comprising a dispersion of an aliphatic alcohol of at least 8 carbon atoms in an aqueous solution of a water-soluble methoxymethyl melamine containing at least two methoxymethyl groups and thereafter heating the impregnated fabric to insolubilize the resin-forming constituents therein.

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