

UNITED STATES PATENT OFFICE

2,371,892

PERMANENT FINISH FOR TEXTILES

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No Drawing. Application August 30, 1941.
Serial No. 408,991

8 Claims. (Cl. 260—72)

This invention relates to textile finishing compositions and more particularly to a permanent water-repellent finish resistant to laundering. A principal object of the invention resides in the provision of a permanent finish of this type that is stable on storage, easy to apply, and which will produce a satisfactory softness, fullness of hand, and water-repellent characteristics on textile threads and fabrics when applied thereto. A further object is the provision of a finish of this type that is resistant to soaping and laundering and which will therefore have a long effective life after its application to the fabric.

I have discovered that the above and other desirable characteristics are exhibited by compositions comprising essentially mixtures of "alkylated" or alcohol-reacted melamine-aldehyde condensation products and salts of alkoxypropylamines. Textile treating compositions containing these two essential classes of ingredients are readily applied to textile threads and fibers in the form of aqueous emulsions and when properly cured they are both water-repellent and durable in character.

The "alkylated" or alcohol-reacted melamine-aldehyde condensation products are resinous in character, and it has previously been proposed to apply them to textile fabrics for creaseproofing purposes. I have now discovered that the resins of this class, when used in the form of solutions in organic solvents, can be emulsified or dispersed in water by the addition of relatively small quantities of alkoxypropylamine salts, and that the resulting aqueous emulsions or dispersions can be employed as textile finishing baths. When textile fibers or fabrics such as cotton, wool, viscose, vinyl polymer, cellulose acetate, spun rayon and the like are impregnated in such baths, dried and cured for a few minutes at reasonably high temperatures they become hydrophobic in character and are resistant to both water and to stains carried by water and alcohol.

Although a wide range of alkoxypropylamine salts may be used as emulsifying agents for alcohol-reacted melamine-aldehyde resins such as butylated melamine-formaldehyde resin, I have found that the molecular weight of the amine has an important bearing on the ultimate water-repellent characteristics of the finished composition. Thus, for example, a compound of relatively low molecular weight such as $C_5H_{11}O.C_3H_7NH_2$, when used in the form of its acetate or other salt, produces a finish of relatively low water-repellency as compared with higher homologs such as the octyloxypropyla-

mine, decyloxypropylamine and dodecyloxypropylamine acetates or other salts. Still better water-repellency is frequently obtained with salts of secondary or tertiary amines in which at least one of the two additional substituent chains of the amine is also of relatively high molecular weight, and the best waterproofing properties are exhibited by secondary or tertiary alkoxypropylamine salts in which the amine contains at least two alkoxypropyl groups of substantial molecular weight. From what has been said, it is evident that the water-resistant properties of various compositions falling within the scope of my invention will improve gradually with the number of carbon atoms in the amine salts and it is therefore rather difficult to assign any definite lower limit of operativeness. On the basis of numerous tests, however, it can be stated that the alkoxypropylamine salt should contain at least eleven carbon atoms, and in most cases it is preferable to have at least eight carbon atoms in the alkyl radical forming the alkoxypropyl group of the amine. For better results it is usually advisable to employ an alkoxypropylamine salt in which the alkyl radical contains from 12-18 carbon atoms, and the best results have been obtained with bis-octadecoxypropylamine salts.

Representative compounds that have given the most satisfactory results are bis(3-N-octadecoxypropyl)amine acetate, hydrochloride, sulfate, hydroxyisobutyrate, etc., and similar salts of 3-N-octadecoxypropylamine, bis(3-N-dodecoxypropyl)amine, N-butyl-bis(3-N-octadecoxypropyl)amine, methyl-bis(3-N-octadecoxypropyl)amine, and bis(3-hexadecoxypropyl)amine.

The "alkylated" melamine-aldehyde resins with which the above compounds are employed are known, and need not be discussed in great detail. It is known that melamine can be condensed with formaldehyde, acetaldehyde, propionaldehyde, butyraldehyde, benzaldehyde and the like in molar ratios of 1:2 up to and including 1:6 to form resinous complexes. As is described in greater detail in U. S. Patent No. 2,197,357 these complexes can be transformed into organic solvent-soluble "alkylated" compounds by further reaction with alcohols such as methyl, ethyl, propyl, butyl, amyl, hexyl, octyl or benzyl alcohols, preferably in the presence of a small quantity of an acid condensation catalyst such as phosphoric acid. Preferably an excess of the alcohol is used and the mixture is heated until substantially all the water has been removed, whereupon there is obtained a solution of the resinous material in the excess solvent. The solution may then be fur-

ther diluted with other alcohols or with limited amounts of hydrocarbon solvents such as toluene, xylene and the like.

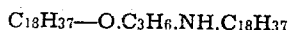
In preparing the textile finishing compositions of the present invention, I find that the best results are obtained with melamine-formaldehyde condensation products containing from 2-5 and preferably about 4 moles of formaldehyde for each mole of melamine. These condensation products are preferably reacted with lower aliphatic alcohols such methyl alcohol or butyl alcohol to the solvent-soluble stage, after which suitable amounts of other organic solvents may be added. A suitable alkoxypyrrolamine salt is then preferably dissolved at room temperature in the resin solution whereupon water is slowly added with rapid, efficient stirring until an emulsion of the water-in-oil type having a particle size of one micron or less is obtained. For application to the fabrics this emulsion is further diluted with about 5 to 10 times its weight of water at 70-120° F. and the cloth is passed through the bath under conditions such that the amount of finish will range from 3% to 12%. The cloth is then preferably dried for about 5 minutes at 240-250° F. after which the resin is cured for 4 minutes at 285-310° F. or higher.

Cloth finished by the above process has a soft, full hand and good water repellency, which are characteristics that have been much desired in the trade. Tests carried out at a number of commercial laundries have proven the finish to have good resistance both to laundering and to dry cleaning. The finish is compatible with corn starch, gelatin and locust bean gum, and the latter actually improves the hand of the treated fabric when incorporated into the textile treating bath. The finish is completely stable upon storage and undesirable odors are not generated even upon storage in temperate climates.

The invention will be illustrated in greater detail by the following specific examples. It should be understood, however, that while these examples may describe in detail some of the more specific features of the invention, they are given primarily for illustrative purposes and the invention in its broader aspects is not limited thereto.

Example 1

A quantity of 3-N-octadecoxypyrrolnitrile, obtained by the reaction of acrylonitrile with octadecyl alcohol in the presence of a small quantity of sodium ethylate, was hydrogenated at 110° C. with hydrogen under 800-1200 lbs. per sq. in. pressure in the presence of Raney nickel catalyst. The resulting product was largely converted into the corresponding secondary amine by the application of heat immediately after the hydrogenation was completed. Upon separation from the catalyst it was found to contain about 82% of bis-(3-N-octadecoxypyrrol) amine and 18% of 3-N-octadecoxypyrrolamine. A small amount of mixed amine of the formula



was also present.

21.2 parts by weight of the above product was melted and stirred at 83° C. and 2.42 parts of glacial acetic acid were added. The stirring was continued until the salt formation was complete, after which the batch was cooled.

12 parts by weight of the above mixture of amine salts were dissolved at room temperature in 48 parts of a solution containing 50% by weight

of the butyl alcohol reaction product of tetramethylol melamine dissolved in a mixture of about equal quantities of butyl alcohol and xylene. 40 parts by weight of water at 70° F. were then slowly added with rapid agitation. The batch thickened at first, but upon continued agitation and water addition it was converted into a fluid emulsion of the oil-in-water type. At 80-90° F. this emulsion remained fluid, while at 67° F. it became a soft paste that could be easily stirred. The product is stable at both low and high temperatures. It has been frozen at the extremely low temperature of solid carbon dioxide without permanent damage. Samples stored at 122° F. for 500 hours and at room temperatures for several months have been satisfactory for immediate use. The pH of the emulsion as prepared may vary from 3.5 to 4.5, but can be adjusted over a much wider range without affecting the stability of the emulsion.

A portion of the batch was diluted by stirring into 6 times its weight of water warmed to 70-120° F. and cloth was passed through the bath and then dried for 5 minutes at 240-250° F. The cloth was then heated on tenter frames for 4 minutes at 285° F. to cure the finish. 80 x 80 bleached cotton percale was finished in this manner and sent to a commercial laundry for a regular laundering including high temperature (140-160° F.) soaping, chlorine bleach, and rinsing and also a regular colored goods wash consisting of low temperature (120° F.) soaping and rinsing. Samples of 68 x 60 spun rayon, 4.59 yards per pound in weight, were also finished with 3% and 4.5% of the finish and sent to the same laundry for the colored goods wash only. The fabrics were then tested for water-repellency by subjecting a weighed six-inch square to a fine spray of water at 70° F. for one minute and determine the increase in weight. The results were as follows:

	Percent water absorbed		
	3% on cotton	3% on rayon	4.5% on rayon
Original	37.8	62.2	64.5
Regular laundering	39.9	58.5	51.0
Colored goods wash	35.6		

Example 2

A finish similar to that described in Example 1 was prepared, but an octyl alcohol-reacted pentamethylol melamine was substituted for the butylated compound. The emulsion was diluted with water for application and bleached 80 x 80 cotton percale was impregnated and cured for six minutes at 290° F. After soaping for 1 hour and 2 hours in 0.5% soap and 0.2% soda ash at 160° F. the cloth retained 92% and 81%, respectively, of the finish. Spray tests similar to those described in Example 1 showed a percentage water absorption of 50.3 for the original treated fabric, 54.0 for the fabric after one hour soaping and 60.4 after 2 hours.

Example 3

Finishes similar to the composition of Example 1 were prepared in which 7, 8, 9, 10 and 11 parts of the amine salt were incorporated instead of the 12 parts previously described. It was found that the heat stability of the emulsion was lowered by this reduction.

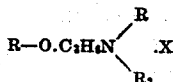
Example 4

12 parts by weight of N-butyl bis(3-N-octadec-oxypropyl)amine acetate were dissolved in 48 parts of a 50% solution of butylated melamine-formaldehyde resin and the mixture was emulsified in water with efficient high-speed stirring. The emulsion was diluted, applied to cloth, and dried and cured by heating in the usual manner. The treated cloth was found to be equal in water-repellent properties and as resistant to laundering as the product of Example 1, and possessed the further advantage that there was no chlorine absorption during bleaching in the laundry.

What I claim is:

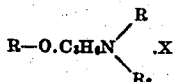
1. A composition for finishing textiles which comprises essentially a mixture of an alcohol-reacted melamine-aldehyde condensation product and a salt of an alkoxypropylamine containing at least eleven carbon atoms.

2. A composition for finishing textiles which comprises essentially a mixture of an alcohol-reacted melamine-aldehyde condensation product and a salt of an alkoxypropylamine corresponding to the formula



in which R is an alkyl radical, R₁ is a member of the group consisting of hydrogen and alkyl radicals, R₂ is a member of the group consisting of hydrogen and alkoxy radicals and X is a salt-forming anion and in which a total of at least eight carbon atoms is contained in the groups R, R₁ and R₂.

3. A composition for finishing textiles which comprises essentially a mixture of an alcohol-reacted melamine-formaldehyde condensation product and a salt of an alkoxypropylamine corresponding to the formula



in which R is an alkyl radical, R₁ is a member of the group consisting of hydrogen and alkyl radicals, R₂ is a member of the group consisting of hydrogen and alkoxy radicals and X is a salt-forming anion and in which a total of at least

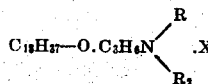
eight carbon atoms is contained in the groups R, R₁ and R₂.

4. A composition for finishing textiles which comprises essentially a mixture of an alcohol-reacted melamine-formaldehyde condensation product and a salt of an alkoxypropylamine corresponding to the formula



10 in which R is an alkyl radical containing at least eight carbon atoms, R₁ is a member of the group consisting of hydrogen and alkyl radicals and X is a salt-forming anion.

15 5. A composition for finishing textiles which comprises essentially a mixture of an alcohol-reacted melamine-formaldehyde condensation product and a salt of an octadecoxypropylamine corresponding to the formula



20 in which R₁ is a member of the group consisting of hydrogen and alkyl radicals, R₂ is a member of the group consisting of hydrogen and alkoxy radicals and X is a salt-forming anion.

25 6. A composition for finishing textiles which comprises essentially a mixture of an alcohol-reacted melamine-formaldehyde condensation product and a salt of an octadecoxypropylamine corresponding to the formula



30 in which R₁ is a member of the group consisting of hydrogen and alkyl radicals and X is a salt-forming anion.

35 7. A composition for finishing textiles which comprises essentially a mixture of a butyl alcohol-reacted melamine-formaldehyde condensation product and a salt of bis(3-N-octadecoxypropyl)amine.

40 8. A composition for finishing textiles which comprises essentially the butyl alcohol reaction product of a melamine-formaldehyde condensation product containing about four molecules of formaldehyde for each molecule of melamine together with a salt of bis(3-N-octadecoxypropyl)-amine.

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