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TEXTILE FINISHING AND COMPOSITION
THEREFOR

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This invention relates to the treating and finishing of textiles and textile-forming materials such as cotton, linen, wool, viscose, cellulose acetate, spun rayon, silk and the like. The invention includes processes for finishing textiles of the above and other types, textile finishing compositions for use in such processes, the preparation of such textile finishing compositions, and textiles of improved properties finished with the compositions and by the processes of the invention.

A wide range of softening agents, lubricants, creaseproofing agents and waterproofing agents have heretofore been applied to textile threads and fibers. The treatment of textiles for these and similar purposes such as shrinkproofing 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 improved water-resistance, a more desirable hand, and in many cases an improved resistance against shrinking. Other important objects will become apparent from the following detailed description of methods and compositions constituting preferred embodiments of the invention, and will be pointed out in the appended claims.

I have discovered that monoacyl guanidines in which the acyl group contains at least 7 carbon atoms are excellent finishing agents for textiles, particularly when applied in conjunction with acid-curing thermosetting aminoplast resins of the type hereinafter described. Some of the monoacyl guanidines are known chemical compounds and have been prepared by heating guanidine hydrochloride with acyl chlorides under pressure, but I have found that the higher acyl guanidines, in which the acyl group contains at least 7 carbon atoms and preferably 10-18 carbon atoms, possesses the important characteristic of imparting hydrophobic properties to textiles when applied thereto in relatively small amounts on the order of 0.25-3% by weight. The acyl guanidines are also softeners for textile fibers such as cotton and spun rayon cloth, particularly when used in amounts greater than 3%, and my invention in its broader aspects is therefore not limited to the use of any specific quantities of these compounds.

The acyl guanidines are not resin-forming materials, and it is therefore necessary to apply them to the textiles in admixture with a resinous ma-

terial which will act as a binder. I have discovered, as one of the most important features of my invention, that thermosetting nitrogen-containing resins of the type of urea-formaldehyde resins, thiourea-formaldehyde resins and melamine-formaldehyde resins will not only function as a physical binder but will actually combine with a higher acyl guanidine on the textile fibers during the curing thereof to form a chemically homogeneous finish of excellent water-proofing properties. Since there is a definite cooperation between the higher acyl guanidines and the acid-curing thermosetting nitrogen-containing resins of the above-described class that is not found in any other type of curable resin so far investigated, I prefer to employ the resins of this class in practicing my invention and the term "acid-curing thermosetting aminoplast resin" will be used in the present specification and claims to define the members of that group of acid-curing thermosetting resins consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins. All of the resins of this group are capable of being prepared in a curable state in which they are soluble or dispersible in water or organic solvents such as ethyl or propyl alcohol, but upon evaporation of the solvents and exposure of the resulting films to high temperatures they can be rapidly cured to an infusible, water-insoluble stage. Although it is possible to cure these resins simply by long heating at relatively high temperatures they are cured much more rapidly and effectively by heating in the presence of acids or acid-forming materials, known as curing accelerators, and such accelerators are preferably employed in the commercial practice of my invention.

The higher monoacyl guanidines in which the acyl group contains at least 7 carbon atoms are preferably prepared by the reaction of guanidine with an ester of a higher fatty acid containing 7 or more carbon atoms, and preferably containing 10-18 carbon atoms, such as the methyl or ethyl ester of caproic acid, caprylic acid, lauric acid, myristic acid, palmitic acid or stearic acid. It is preferable to employ saturated higher fatty acids in order to avoid the possibility of rancidity in the treated textiles upon storage, although in some cases higher monoacyl guanidines in which the acyl group is unsaturated may be employed. The acyl guanidines used in practicing the present invention are preferably prepared by mixing a slight excess of the ester of the higher fatty acid with a solution of free guanidine in any suitable solvent such as methanol, ethanol and the

like, and allowing the mixture to stand at room temperature until the corresponding acyl guanidine is formed.

Although any acid-curing thermosetting aminoplast resin may be employed in conjunction with any higher monoacyl guanidine in preparing textile finishing compositions in accordance with the present invention, I greatly prefer the melamine-formaldehyde resins and particularly the alkylated melamine-formaldehyde resins prepared by heating a monomeric melamine-formaldehyde condensation product with a primary alcohol in the presence of an acid condensing agent. Alkylated melamine-formaldehyde resins prepared by the use of lower aliphatic alcohols such as methanol, ethanol and butanol, and especially the methylated methylol melamines are the preferred resin-forming compounds for use in practicing my invention, for these materials are easily dispersed in water or aqueous solvents such as alcohol-water mixtures and produce an extremely high degree of water-repellency when applied to the textile fibers.

The textile finishing compositions of my invention are preferably prepared by dissolving the higher acyl guanidine and a solution of the resin-forming material such as methylated methylol melamine in an organic solvent such as ethanol or preferably isopropanol, after which the solution is emulsified or otherwise dispersed in sufficient water to form a dispersion of the desired concentration for use. In many cases it is unnecessary to employ an emulsifying or dispersing agent, but it is sometimes desirable to dissolve butyl or isopropyl naphthalene sodium sulfonate or sodium dioctyl sulfosuccinate in the water to a concentration of about 0.5-3% by weight. A suitable curing accelerator for the resin, such as a mixture of 4 parts hexamethylene tetramine and 40 parts diammonium phosphate may also be added to the water or aqueous solution in which the higher monoacyl guanidine and resin solution are dispersed.

The finishing compositions of my invention are preferably applied to textile fibers by immersing or otherwise impregnating the cloth with the aqueous dispersion prepared as described above, after which the cloth is preferably passed through squeeze rollers to adjust the quantity of finishing agent to be absorbed. The cloth is then preferably dried in a steam heated drier and the finishing composition is cured by heating for 5-15 minutes at temperatures of 160-200° F. The cloth is then preferably washed with an 0.5% aqueous soap solution to remove any uncured finishing composition, and again dried. When applied in this manner the finishing composition does not change the surface appearance of the cloth and does not affect the color or brightness of printed or dyed patterns, but the cloth becomes hydrophobic in character and is resistant to absorption of water.

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

Example 1

100 parts by weight of methyl stearate, 20 parts of guanidine and 51 parts of ethanol were mixed and allowed to stand at room temperature and

after a few hours standing 158 parts of acetone were added to maintain fluidity. After 24 hours standing the crystals were filtered off and recrystallized from acetone. Analysis showed the product to be monostearoyl guanidine.

Methylated methylol melamine was prepared by heating 126 parts by weight of melamine and 486 parts of 37% aqueous formaldehyde solution at 60-70° C. to dissolve the melamine, adding NaOH solution to raise the pH to 7.5, concentrating under a partial vacuum to 75% solids, adding 864 parts of methanol containing sufficient phosphoric acid to neutralize the NaOH, and distilling off methanol and water to obtain a resin solution containing 62% methylated methylol melamine and 38% methanol.

A solution of a water-dispersible urea-formaldehyde resin was prepared by adding 60 parts by weight of urea to 105.3 parts of neutral 37% aqueous formaldehyde solution and agitating at room temperature until the urea was dissolved.

Textile finishing compositions were prepared by dissolving 5 parts by weight of stearyl guanidine and sufficient resin solution to make 20 parts by weight of resin solids in 10 parts of isopropyl alcohol followed by the addition of 175 parts of water and 25 parts of a curing accelerator consisting of a 10% solution of a mixture of 4 parts hexamethylene tetramine and 30 parts diammonium phosphate. Two compositions were prepared in this manner, one containing the methylated methylol melamine solution and the other the urea-formaldehyde resin solution, and each composition was applied to 80x80 cotton cloth which was then heated in an oven at 290° F. for 22 minutes. The cloth was then washed in an 0.5% soap solution at 140° F. for 30 minutes, dried and tested for water repellency. In both cases the cloth was found to possess excellent water resistance.

Example 2

Spun rayon challis and 80 x 80 cotton cloth were impregnated with 10% of methylated methylol melamine, 1.5% of stearyl guanidine and 1% of accelerator, based on the dry weight of the cloth, by passing the cloth through a dispersion prepared as in Example 1 and then through squeeze rolls. After drying and curing for 7 minutes at 290° F. the cloth was subjected to a standard spray test, which consisted in determining the percent of water absorbed after a one-minute spray under a six foot head. The results were as follows:

	Original un-soaped	Soaped at 160° F.				Dry cleaned 30 min. ¹
		15 min.	30 min.	60 min.	2½ hours	
Spun rayon.....	50.5	43.5	42.9	38.1	40.4	45.8
80x80 cotton.....	35.2	31.0	33.6	34.7	32.2	40.1

¹ Dry cleaned after soaping 30 minutes.

Example 3

Lauroyl guanidine was prepared by the method described in Example 1, using 75 parts of methyl laurate, 20 parts of guanidine and 40 parts of ethanol, and used in admixture with methylated methylol melamine for finishing heavy cotton broadcloth. The cloth was impregnated with 10% of its weight of methylated methylol melamine, 2% of lauroyl guanidine and 1% of accelerator, dried and cured and subjected to the spray test as described in the preceding example, first after washing 15 minutes at 160° F. in a

.5% soap solution and again after washing for an additional one-hour period at the same temperature and soap concentration. Water retention values of 28.6 and 29.9, respectively, were obtained.

What I claim is:

1. A method of finishing textiles which comprises the steps of applying thereto an aqueous finishing composition containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms and also containing substantial amounts of a curable acid-curing thermosetting aminoplast resin selected from the group consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins and then curing the aminoplast resin on the textile fibers.

2. A method of finishing textiles which comprises the steps of applying thereto an aqueous finishing composition containing 0.25-3 parts by weight of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms together with 5 to 20 parts by weight of a curable acid-curing thermosetting aminoplast resin selected from the group consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins and then curing the aminoplast resin on the textile fibers.

3. A method of finishing textiles which comprises the steps of applying thereto an aqueous finishing composition containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms and also containing substantial amounts of a curable acid-curing thermosetting aminoplast resin selected from the group consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins together with a curing accelerator therefor and then curing the aminoplast resin on the textile fibers.

4. A method of finishing textiles which comprises the steps of applying thereto an aqueous finishing composition containing 0.25-3 parts by weight of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms and also containing 5 to 20 parts by weight of a curable acid-curing thermosetting aminoplast resin selected from the group consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins together with a curing accelerator therefor and then curing the aminoplast resin on the textile fibers.

5. A method of finishing textiles which comprises the steps of applying thereto an aqueous finishing composition containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms and also containing substantial amounts of a curable melamine-formaldehyde resin and then curing the melamine-formaldehyde resin on the textile fibers.

6. A method of finishing textiles which comprises the steps of applying thereto an aqueous finishing composition containing 0.25-3 parts by weight of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms and also containing 5 to 20 parts by weight of a curable melamine-formaldehyde resin and then curing the melamine-formaldehyde resin on the textile fibers.

7. A method of finishing textiles which comprises the steps of applying thereto an aqueous

finishing composition containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms and also containing substantial amounts of a curable methylated methylol melamine and then curing the methylated methylol melamine on the textile fibers.

8. A method of finishing textiles which comprises the steps of applying thereto an aqueous finishing composition containing 0.25-3 parts by weight of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms and also containing 5 to 20 parts by weight of a curable methylated methylol melamine and then curing the methylated methylol melamine on the textile fibers.

9. A textile finishing composition comprising an aqueous dispersion containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms together with substantial amounts of a curable acid-curing thermosetting aminoplast resin selected from the group consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins.

10. A textile finishing composition comprising an aqueous dispersion containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms together with substantial amounts of a curable acid-curing thermosetting aminoplast resin selected from the group consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins and a curing accelerator.

11. A textile finishing composition comprising an aqueous dispersion containing substantial amounts of stearoyl guanidine together with substantial amounts of a curable acid-curing thermosetting aminoplast resin selected from the group consisting of melamine-aldehyde resins and urea and thiourea-aldehyde resins.

12. A textile finishing composition comprising an aqueous dispersion containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms together with substantial amounts of a curable melamine-formaldehyde resin.

13. A textile finishing composition comprising an aqueous dispersion containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms together with substantial amounts of a curable methylated methylol melamine.

14. A textile finishing composition comprising an aqueous dispersion containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms together with substantial amounts of a curable melamine-formaldehyde resin and a curing accelerator.

15. A textile finishing composition comprising an aqueous dispersion containing substantial amounts of a monoacyl guanidine in which the acyl group is a fatty acid radical containing at least seven carbon atoms together with substantial amounts of a curable methylated methylol melamine and a curing accelerator.

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