

[54] COMPOSITION AND METHOD OF IMPARTING PREMANENT PRESS AND SOIL RELEASE PROPERTIES TO FABRICS

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[51] Int. Cl. .... **C08g 37/32**

[58] Field of Search... **260/851, 856; 117/138.8, 139.4; 8/115.5, 115.6, 116.3**

[56]

References Cited

UNITED STATES PATENTS

3,405,003	10/1968	De Paolo.....	260/851
3,377,249	4/1968	Marco .....	260/851
3,540,835	11/1970	Marco .....	117/139.4
3,437,626	4/1969	Glabisch.....	260/29.6
3,495,930	2/1970	Machell.....	8/115.5
3,236,685	2/1966	Caldwell et al.....	117/138.8 F
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[57]

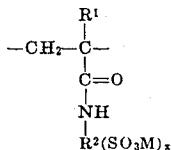
ABSTRACT

Textile materials are given permanent press and soil release properties by treating them with a textile resin and a polymer of an N-sulfohydrocarbon-substituted acrylamide, preferably 2-acrylamido-2-methylpropanesulfonic acid, and usually also with a textile resin catalyst. The textile resin is normally cured on the fabric by heating the same to 130°-200° C.

20 Claims, No Drawings

**COMPOSITION AND METHOD OF IMPARTING  
PREMANENT PRESS AND SOIL RELEASE  
PROPERTIES TO FABRICS**

This invention relates to compositions and methods 5 for improving permanent press and soil release properties of fabrics. More particularly, it relates to a composition comprising a textile resin and at least one polymer containing units of the formula



wherein  $\text{R}^1$  is hydrogen or a lower alkyl or substituted lower alkyl radical;  $\text{R}^2$  is a divalent or trivalent hydrocarbon or substituted hydrocarbon radical;  $\text{M}$  is hydrogen or one equivalent of a cation; and  $x$  is 1 or 2.

The recent development of synthetic or partially synthetic special purpose fabrics, including "permanent press" fabrics, is described in detail in U.S. Pat. Nos. 3,377,249 and 3,535,141, the disclosures of which are incorporated by reference herein. As taught in those patents, permanent press qualities are imparted to fabrics by applying thereto a textile resin. Many fabrics, especially synthetics, have a strong tendency to accept and retain grime, dirt and oily deposits. This tendency is frequently increased by the application of a textile resin thereto. It is of interest, therefore, to provide a method for fabric treatment which will decrease the tendency of the fabric to retain soil, or better yet, will cause it to repel soil.

Accordingly, the principal object of the present invention is to provide improved compositions and methods for fabric treatment.

A further object is to provide compositions which may be applied to a fabric to impart permanent press 40 and soil release properties.

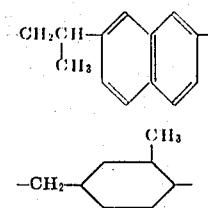
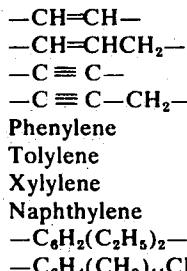
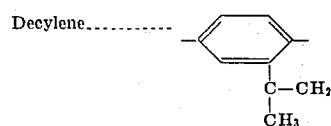
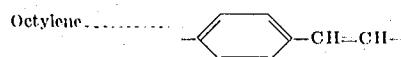
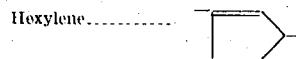
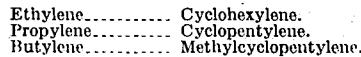
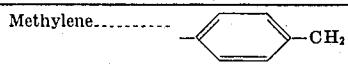
Other objects will in part be obvious and will in part appear hereinafter.

As previously indicated, the compositions of this invention contain two essential components, the first of 45 which is a textile resin. Textile resins are defined in the aforementioned U.S. patents and generally include epoxy, acetal, aminoplast and similar resins, with the aminoplast resins being preferred. As used herein, the term "aminoplast resin" means a condensation product, usually of an amine or amide with an aldehyde, which is transformed to the thermoset state at temperatures of about 130°-200°C. This product may be monomeric or polymeric. Exemplary of the aminoplast resins suitable for use in the present invention are the urea formaldehydes, e.g., propylene urea formaldehyde or dimethylol urea formaldehyde; melamine formaldehydes, e.g., tetramethylolmelamine, pentamethylolmelamine or hexamethylolmelamine; ethylene ureas, e.g., dimethylol ethylene urea, dihydroxy dimethylol ethylene urea, ethylene urea formaldehyde or hydroxyethylene urea formaldehyde; carbamates, e.g., alkyl carbamate formaldehydes; formaldehyde-acrolein condensation products; formaldehyde-acetone condensation products; alkylolamides, e.g., N-methyloformamide, N-methyloacetamide, N-methyloacrylamide, N-methyolmethacrylamide, N-

methylol-N-methyacrylamide, N-methyolmethacrylamide (acrylamide) or methylenebis(N-methyolacrylamide); haloethyleneacrylamide; diureas, e.g., trimethylol acetylene diurea, tetramethylol acetylene diurea; triazones, e.g., dimethylol-N-ethyl trizone, N,N'-ethylene-bis(dimethylol)triazone and halo triazones; haloacetamides, e.g., N-methyol-N-methylchloroacetamide; urons, e.g., dimethylol uron or dihydroxy dimethylol uron; and the like. Also useful are the analogous thioureas, thioamides and the like.

The second essential component of the compositions of this invention is at least one (usually only one) polymer of an N-sulfohydrocarbon-substituted acrylamide. These polymers are represented by the above formula in which  $\text{R}^1$  is hydrogen or a lower (as defined hereinafter) alkyl radical and  $\text{R}^2$  is a divalent or trivalent hydrocarbon radical. As used herein, the term "hydrocarbon radical" includes aliphatic, cycloaliphatic and aromatic (including aliphatic- and cycloaliphatic-substituted aromatic and aromatic-substituted aliphatic and cycloaliphatic) radicals. It also includes cyclic radicals wherein the ring is completed through another portion of the molecule; that is, any two indicated substituents may together form a cyclic hydrocarbon radical.

The following are illustrative of divalent hydrocarbon radicals within the scope of this invention. Where a named radical has several isomeric forms (e.g., butylene), all such forms are included.



Trivalent radicals are similar to the above but have an additional hydrogen atom abstracted.

Many obvious variations of these radicals will be apparent to those skilled in the art and are included within the scope of the invention.

Substituted hydrocarbon, alkyl, aryl, alkylene, arylene, etc., radicals are considered fully equivalent to the hydrocarbon, alkyl, aryl, alkylene, arylene, etc., radicals and to be part of this invention. By "substituted" is meant radicals containing substituents which do not alter significantly the character or reactivity of the radical. Examples are:

Halide (fluoride, chloride, bromide, iodide)

Hydroxy

Ether (especially lower alkoxy)

Keto

Carboxy

Ester (especially lower carbalkoxy)

Aminoacyl (amide)

Amino

Nitro

Cyano

Thioether

Sulfoxyl

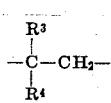
Sulfone

Sulfonic acid ester, amide, etc.

In general, no more than about three such substituent groups will be present for each 10 carbon atoms in the radical.

Preferably, the hydrocarbon or substituted hydrocarbon radicals in the N-sulfohydrocarbon-substituted acrylamides are free from ethylenic and acetylenic unsaturation and have no more than about 30 carbon atoms, desirably no more than about 12 carbon atoms. A particular preference is expressed for lower hydrocarbon radicals, the word "lower" denoting radicals containing up to seven carbon atoms. Still more preferably, they are lower alkylene or arylene radicals, most often alkylene.

In the formula, M is hydrogen or one equivalent of a cation and is usually hydrogen or alkali metal. R<sup>1</sup> is 40 hydrogen or lower alkyl but is preferably hydrogen or methyl, usually hydrogen. R<sup>2</sup> may be any divalent or trivalent hydrocarbon radical, preferably lower alkylene or arylene and usually lower alkylene. In a preferred embodiment of this invention, R<sup>2</sup> is 45

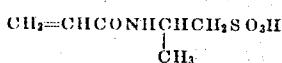


wherein R<sup>3</sup> is hydrogen or a lower alkyl radical, R<sup>4</sup> is a lower alkyl radical and the sulfonic acid group is attached to the unsubstituted methylene carbon. These 55 polymers may be obtained by the polymerization, either alone or in combination with other polymerizable vinyl monomers, of the corresponding monomeric N-sulfohydrocarbon-substituted acrylamides of which the following acids, and their salts, are examples.

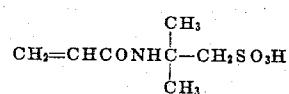
2-Acrylamidoethanesulfonic acid



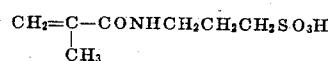
2-Acrylamidopropanesulfonic acid



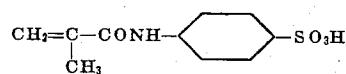
2-Acrylamido-2-methylpropanesulfonic acid



3-Methacrylamidopropanesulfonic acid



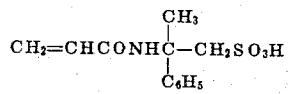
4-Methacrylamidocyclohexanesulfonic acid



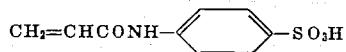
2-Acrylamido-2-phenylethanesulfonic acid



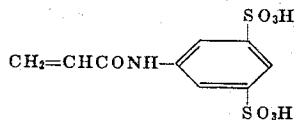
2-Acrylamido-2-phenylpropanesulfonic acid



4-Acrylamidobenzenesulfonic acid



5-Acrylamidobenzene-1,3-disulfonic acid



60 From the standpoint of economy, ease of preparation and polymerization, and effectiveness, the most desirable polymers are those of 2-acrylamido-2-methylpropanesulfonic acid or its salts. The term "N-sulfohydrocarbon-substituted acrylamide" when used 65 hereinafter will refer to this class of compounds generally, with the understanding that the above-named compound is especially preferred.

The N-sulfohydrocarbon-substituted acrylamide polymers used in the compositions of this invention may be homopolymers or copolymers, the latter containing at least about 5 percent by weight, and preferably at least about 50 percent, of N-sulfohydrocarbon-substituted acrylamide units. The identity of the other monomer or monomers is not critical except that the polymer must be water-soluble or capable of forming a stable aqueous emulsion. The most useful polymers are homopolymers and copolymers with 5-95 percent, preferably 5-50 percent and most desirably 5-30 percent, of an unsaturated acid (e.g., maleic acid) or a derivative thereof, especially an acrylic monomer such as acrylic or methacrylic acid or a salt or amide thereof, notably acrylamide, methacrylamide, N-methylacrylamide, diacetone acrylamide and the like.

The polymer may be prepared in bulk, solution, suspension or emulsion. Since the polymers are water soluble, and it is frequently convenient to prepare them in aqueous solution. Another method is to prepare an aqueous solution of the monomer or monomers and suspend the same, prior to polymerization, in a water-immiscible solvent such as an aliphatic or aromatic hydrocarbon or halogenated hydrocarbon, removing the water after polymerization. Generally, the sulfonic acid monomer is converted to its metal salt prior to polymerization by means of a suitable alkaline reagent; however, it is also within the scope of this invention to prepare and use a polymer of the free acid. When polymerization is effected in suspension, ordinary suspending agents known to those skilled in the art are used.

The polymerization may be promoted by typical initiators used in aqueous systems, especially peroxides, persulfates, persulfate-bisulfite and the like. It has been found that the alkali metal salts, especially the sodium salt, of 2-acrylamido-2-methylpropanesulfonic acid may frequently be polymerized in the absence of polymerization initiator.

It is sometimes advantageous to carry out the polymerization in the presence of a small amount of chain transfer agent, which tends to cause formation of a polymer with more uniformity in molecular weight than is otherwise produced. Suitable chain transfer agents are known to those skilled in the art.

The compositions of this invention may contain additional ingredients such as textile resin curing catalysts, emulsifying agents, wetting agents, softeners and the like. While the presence of curing catalysts has previously been considered mandatory, it has been found that the N-sulfohydrocarbon-substituted acrylamide polymers used in the compositions of this invention frequently serve themselves as acidic curing catalysts and that an additional catalyst is therefore frequently unnecessary. However, it is usually advantageous to incorporate an acidic or basic catalyst depending on the curing conditions of the textile resin, in the composition. The most commonly used acidic catalysts are metal salts such as magnesium chloride, zinc nitrate and zinc fluoborate, and amine salts such as monoethanolamine hydrochloride and 2-amino-2-methylpropanol nitrate. As basic catalysts, it is desirable to use compounds which do not react under the conditions of acid catalysis but which can be activated

by heat or the use of another chemical compound. Illustrative of heat-activated basic catalysts are sodium carbonate, potassium carbonate, potassium bicarbonate, sodium silicate, sodium or potassium phosphates, barium carbonate and quaternary ammonium hydroxides and carbonates. Typical of the chemically activated type is an alkali metal sulfite, which can be decomposed into the corresponding hydroxide by contacting the same with formaldehyde incorporated (for example) in the steam used for curing the textile resin.

In general, the compositions of this invention will contain about 5-25 percent by weight, preferably about 5-15 percent, of the textile resin; about 1-15 percent, preferably about 1-10 percent, of the N-sulfohydrocarbon-substituted acrylamide polymer; and if a catalyst is used, about 0.5-15 percent thereof. The preferred percentage range for acid catalysts is about 0.5-5 percent, and for basic catalysts about 2-16 percent.

The following are illustrative of N-sulfohydrocarbon-substituted acrylamide polymers which may be used in the compositions of this invention. Inherent viscosity figures are given for a solution of the polymer in 3 percent aqueous sodium chloride solution at 30°C. All parts and percentages are by weight.

1. A homopolymer of 2-acrylamido-2-methylpropane-sulfonic acid, prepared from a suspension in benzene of an aqueous solution of the monomer, using a hydrogen peroxide-ferrous sulfate polymerization catalyst. The polymer has an inherent viscosity (0.5 percent solution) of 1.11.

2. A homopolymer of sodium 2-acrylamido-2-methylpropanesulfonate, prepared in benzene suspension like polymer 1 using an ammonium persulfate-sodium bisulfite catalyst. Its inherent viscosity (0.5 percent solution) is 1.90.

3. A 50:50 (by weight) copolymer of sodium 2-acrylamido-2-methylpropanesulfonate and acrylic acid, prepared in aqueous solution using an ammonium persulfate-sodium bisulfite catalyst and having an inherent viscosity (0.25 percent solution) of 2.33.

4. An 85:15 (by weight) copolymer of sodium 2-acrylamido-2-methylpropanesulfonate and maleic acid, prepared in benzene suspension as in Example 1 using a sodium lauryl sulfate suspending agent and an ammonium persulfate-sodium bisulfite catalyst. Its inherent viscosity (0.25 percent solution) is 1.11.

5. An 85:15 (by weight) copolymer of sodium 2-acrylamido-2-methylpropanesulfonate and diacetone acrylamide, prepared in benzene suspension and using the suspending agent and catalysts described under polymer 4. Its inherent viscosity (0.5 percent solution) is 1.87.

6. A 50:50 (by weight) copolymer of sodium 2-acrylamido-2-methylpropanesulfonate and diacetone acrylamide, prepared in aqueous solution using an ammonium persulfate-sodium bisulfite catalyst and having an inherent viscosity (0.5 percent solution) of 1.80.

In the following table are listed several compositions of this invention. The balance of each composition, other than the ingredients listed, is water.

	Percent by weight						
	A	B	C	D	E	F	G
Polymer 1	3	6	9				
Polymer 2						6	
Polymer 3						3	
Polymer 4							6

Polymer 5	6
Dihydroxydimethylethylene- urea	10 10 10 10 9
N-Methylolacrylamide	12 12
Zn(NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O	1 1 1 1 1
MgCl <sub>2</sub> · 6H <sub>2</sub> O	3 3 5
Polyethoxynonylphenol surfactant	0.3 0.3 0.3 0.3 0.3 0.3 0.3

The textile materials which may be treated with the compositions of this invention include, without limitation, natural and synthetic textiles whether knitted, woven or non-woven, but woven materials are preferred. Illustrative of natural materials are cotton, wool, jute, flax, viscose rayon, regenerated cellulose and the like. Typical synthetic materials are polyesters such as poly(ethylene terephthalate), polyamides such as Nylon-6 and Nylon-66, acrylic fibers such as polyacrylonitrile, and the like. The invention is particularly useful with mixtures of linear polyester and cellulosic (e.g., cotton) fibers.

The compositions of this invention may be applied to the textile material by any of several methods such as dipping, spraying, padding (which is preferred) or the like. The conditions of application are adjusted to provide a predetermined "wet pickup" of the composition, typically about 30-100 percent. That is, the weight of liquid remaining on the fabric is 30-100 percent of the weight of the fabric.

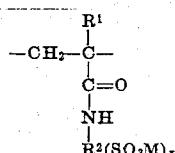
It is also within the scope of the present invention to apply the N-sulfohydrocarbon-substituted acrylamide polymer separately from the textile resin, either before or after. It is generally found, however, that soil-release effectiveness is maximized if the two are applied from the same solution.

Following the application of the composition of this invention, the treated fabric is cured at a temperature of about 130°-200°C. to transform the textile resin to the thermoset state. If the textile resin contains vinyl groups or other unsaturated centers, it may also be subjected to conditions promoting vinyl polymerization, typically irradiation.

The effectiveness of the compositions of this invention is illustrated by a series of tests in which a 50:50 polyester-cotton twill trouser material is treated with said compositions under conditions so as to provide 100 percent wet pickup, mounted on tenter frames, dried 20 minutes at 80°C. and cured 10 minutes at 160°C. The cured samples are rinsed and tumble dried. They are then soiled with mineral oil, salad oil and dirty motor oil, subjected to a typical laundering cycle and examined for residual oil stains. Comparison is made with a control which contains 10 percent dihydroxydimethylethyleneurea, 1 percent zinc nitrate hexahydrate and 0.3 percent polyethoxynonylphenol surfactant, but no soil release agent. It is found that the fabric samples coated with compositions A, B, C and E retain substantially less soil than the control.

What is claimed is:

1. A composition comprising an aminoplast textile resin and at least one polymer containing at least about 5 percent by weight of units of the formula



wherein R<sup>1</sup> is hydrogen or a lower alkyl or substituted lower alkyl radical; R<sup>2</sup> is a divalent or trivalent hydrocarbon or substituted hydrocarbon radical; M is hydrogen or one equivalent of a cation; and x is 1 or 2.

2. A composition according to claim 1 wherein R<sup>1</sup> is hydrogen or methyl, R<sup>2</sup> is a lower alkylene radical and x is 1.

3. A composition according to claim 2 which also contains a textile resin catalyst.

4. A composition according to claim 2 wherein the polymer contains units derived from 2-acrylamido-2-methylpropanesulfonic acid or a salt thereof.

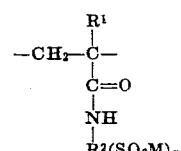
5. A composition according to claim 4 wherein the textile resin is dihydroxydimethylethyleneurea.

6. A composition according to claim 5 wherein the polymer is a homopolymer.

7. A composition according to claim 6 which also contains a textile resin catalyst.

8. A composition according to claim 7 wherein the catalyst is zinc nitrate or magnesium chloride.

9. A method for imparting soil release and durable press characteristics to a textile material which comprises applying thereto an aminoplast textile resin and a polymer containing at least about 5 percent by weight of units of the formula



wherein R<sup>1</sup> is hydrogen or a lower alkyl or substituted lower alkyl radical; R<sup>2</sup> is a divalent or trivalent hydrocarbon or substituted hydrocarbon radical; M is hydrogen or one equivalent of a cation; and x is 1 or 2.

10. A method according to claim 9 wherein the textile material comprises linear polyester fibers.

11. A method according to claim 10 wherein the textile material is a cellulosic containing material.

12. A method according to claim 11 wherein R<sup>1</sup> is hydrogen or methyl, R<sup>2</sup> is a lower alkylene radical and x is 1.

13. A method according to claim 12 wherein a textile resin catalyst is also applied to said textile material.

14. A method according to claim 13 wherein the polymer is a homopolymer of 2-acrylamido-2-methylpropanesulfonic acid or a salt thereof.

15. A method according to claim 12 wherein the textile material is heated at a temperature of about 130°-200°C., subsequent to applying said textile resin and polymer thereto, for a time sufficient to cure said textile resin.

16. A method according to claim 15 wherein the polymer contains units derived from 2-acrylamido-2-methylpropanesulfonic acid.

17. A method according to claim 16 wherein the textile resin is dihydroxydimethylethyleneurea.

18. A method according to claim 17 wherein the polymer is a homopolymer.

19. A method according to claim 18 wherein a textile resin catalyst is also applied to said textile material.

20. A method according to claim 19 wherein the catalyst is zinc nitrate or magnesium chloride.

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