United States Patent Office 3,293,178 Patented Dec. 20, 1966



3,293,178

# PRODUCT AND PROCESS FOR RENDERING PILE FABRICS SOIL RESISTANT

Frederick John MacDonald, Havertown, Pa., and Alton A. Cook, Bloomfield, N.J., assignors to Arkansas Company, Inc., Newark, N.J., a corporation of New Jersey

No Drawing. Filed Feb. 20, 1964, Ser. No. 346,128
14 Claims. (Cl. 252—8.8)

This invention relates to textile finishing and particularly to soil resistant fibers; to methods for rendering fibers soil resistant; and to compositions useful in such methods.

In recent years in the textile field, and particularly in the carpet industry, widespread use has been made of pastel colors. Also, the use of synthetic fibers and blends of fibers which tend to soil more readily than wool has greatly increased. These trends have intensified interest in durable textile finishes which retard soiling.

The small irregularities in the surface of most fibers act as voids to collect particles of dirt and dust. This retention of soil particles, in addition to the obvious effect of soiling, produces color changes in the fiber or fabric and other undesirable effects. The principle involved in the function of soil retardant compositions prior to the present invention, is to deposit water insoluble finely divided particles of inorganic substances such as colloidal silica, titanium hydrate, aluminum phosphate, etc., in the voids, therefore, preventing soil particles from lodging in these voids. In other words, the deposited particles of the soil retardant finish act as barriers to the accumulation of dust and dirt.

The soil retarding compositions heretofore available have a number of defects when applied to floor covering fabrics. The most serious defects are a tendency to dull the surface, discoloration with age, and development of a harsh or otherwise undesirable hand on the fabric.

In addition to the use of finely divided water insoluble inorganic compounds, non-film forming resin emulsion, synthetic rubber latices, etc., have been suggested in the prior art.

As contrasted with the prior art, the present invention makes use of an entirely new and different principle of developing soil resistance on fibers which are commonly used in the manufacture of floor coverings. Instead of filling the voids in the fibers with finely divided or colloidal particles of inorganic substances, the principle of the present invention is to coat the fiber with a strongly adhesive flexible film consisting of a cationic complex, namely, an imidazoline-urea derivative plus a water soluble, colorless, heavy metal salt. This is effected primarily by the cationic or substantive nature of the composition of this invention which is deposited on the fibers by exhaustion of a bath containing the composition under controlled conditions.

The primary object of this invention is to provide a composition which will render fibers and textile fabrics soil resistant.

Another object of the invention is to provide fibers and textiles which are resistant to soiling.

A further object is to provide finishing compositions which minimize color changes and similar undesirable effects.

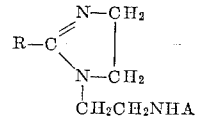
Additional objects of the invention are to provide such compositions which are durable to cleaning, do not yellow on aging, do not dull a fiber surface when applied in effective amounts, and do not cause a harsh or other undesirable hand on fabrics to which it is applied.

A still further object is the provision of a process by which fibers and fabrics can be easily treated to inhibit their becoming permanently soiled and discolored.



Other objects and the advantages of the invention will appear from the following detailed description.

In accordance with the invention, it has been discovered that the soiling tendency of fibers, fabrics and similar textiles can be substantially reduced by applying to their surfaces a strongly adhesive, flexible, film-forming, cationic composition comprising a mixture of a water-soluble, multivalent metal salt and a di-(2-R-imidazoline)-ethylene urea formed by combining urea with an imidazoline derivative having the formula:




wherein R is derived from a saturated fatty acid of the formula RCOOH containing from 16 to 22 carbon atoms, and A is hydrogen, ethylene amine or diethylene diamine. Optionally, the composition may contain about 1 to 3% of a dispersing agent of the nonionic, non-fatty type and/or about 1 to 3% of an organic acid; these percentages being based on the solids content of the composition by weight. Such additives are not to be regarded as necessary in the practice of this invention. The use of an organic acid is as an adjustment relating to the pH control of the treating bath. The use of a dispersing agent relates to the ease of obtaining a satisfactory solution or dispersion in water. The preferred organic acids are hydroxy-acetic and lactic. The preferred dispersing agents are condensates of ethylene oxide with a hydrophobic base formed by condensing propylene oxide with propylene glycol or an alkylaryl polyethoxy alcohol of high molecular weight (2000 to 3000).

The imidazoline derivative can be prepared by reacting one mol of fatty acid with one mol of an alkylene amine with the removal of about two mols of water. This reaction is conventionally carried out at about 150° to 160° C. This is a well-known method and is described, for example, in U.S. Patents 2,267,965, issued to Wilson, and 2,214,152, issued to Wilkes. As indicated above, the fatty acid is a saturated acid containing from 16 to 22 carbon atoms, such as stearic, palmitic, arachidic and behenic acids. The alkylene amine is diethylene triamine, triethylene tetramine or tetraethylene pentamine.

The imidazoline derivative is condensed with urea to produce the di-(2-R-imidazoline)-ethylene urea. This condensation reaction is carried out by heating the reactants to about 150° to 160° C. and preferably with continuous agitation. Two mols of the imidazoline derivative is combined with each mol of urea and approximately two mols of ammonia are removed from the reaction mixture. The resulting condensate is the corresponding di(2-alkylimidazoline)-ethylene urea.

The multivalent colorless water soluble salt is a salt of zirconium, tin, zinc, magnesium, calcium or barium. The salt is preferably the chloride or acetate of these metals, although other soluble salts may be employed, if desired. Mixtures of multivalent metal salts can also be utilized. Types of suitable salts are zirconium acetate, zirconium oxychloride, stannous chloride, zinc acetate, zinc chloride, magnesium chloride, magnesium acetate, barium acetate, barium chloride, calcium acetate and calcium chloride. The term water-soluble as used in the specification and claims means that the salt must be soluble in water to at least about 20 percent at room temperature.

The process of forming the compounds of this invention is described as follows: The di-(2-alkylimidazole)-ethylene urea condensate is melted and added to previously heated water at 140° to 160° F. and thoroughly

dispersed. Optionally, an organic acid, and/or a dispersing agent may be added to the water phase previous to the addition of the melted condensate. Subsequently, the metallic salt previously dissolved in water is added with thorough stirring and the mixture is then cooled to room temperature to form a smooth off-white soft-to firm paste.

The composition is cationic in nature and acid in character and preferably has a pH in the range of about 2 to 5 in a 1% solution. The amount of the anhydrous multivalent salt in the composition can vary over a range of about 30 to about 50% based on the weight of the di-(2-alkylimidazoline)-ethylene urea.

The finishing treatment can be carried out on the fibers in the form of yarn, staple, thread or when manufactured into textile fabric such as woven or tufted carpet. The fiber can be composed of one or more different fibrous materials which can be natural or synthetic in origin. The invention finds its greatest utility in rendering woven and tufted floor coverings of wool, nylon and other synthetic fibers, soil resistant.

The soil retarding composition of the invention can be readily dispersed in cold or warm water for application to a fiber, fabric or carpet.

The product of this invention is preferably applied by an exhaust method in a dye beck immediately following the dyeing operation. 2% to 8% of the product, based on the weight of the material, is weighed out and mixed thoroughly with an equal amount of warm water. This premix is then added to the application bath at 110° F. with thorough mixing to provide a concentration of about 0.1% to 0.5% of the composition in the bath. The material is added to the bath and the temperature of the bath is gradually raised to 150°–160° F. The material is run for 20 to 30 minutes at this temperature to permit exhaustion of the soil retardant compound onto the goods. In most cases an actual dry add-on of about 1 to 3% and preferably about 1.5% is adequate to impart maximum soil resistant properties although less than 1% may be used with reduced effect and more than 3% may be used with loss of economy. Following the application the processed material is extracted in the usual manner and finally dried. Drying temperatures over 240° F. should be avoided.

As an alternative to the exhaustion method the product of this invention may be applied by spraying. This method has the advantage that a relatively small quantity of the composition is effective due to the fact that it is deposited on or near the surface of the pile where resistance to the soil is most needed. For this type of application a 1% to 5% solution of the soil retardant compound is prepared in warm water and this solution is sprayed directly on the fabric in such a way as to obtain uniform application. Other methods such as application in a padder or quetsch are usually not satisfactory for obtaining maximum soil resistance properties.

In addition to imparting soil resistance to the treated fibers and fabrics, the treating composition of the invention will not dull or cause other deleterious effects. Carpets and rugs produced in accordance with the invention when subjected to standard soiling tests and to actual foot traffic over a period of several weeks, possess excellent soil resistance and are more readily cleanable than the untreated material.

The following examples are given for purposes of illustration:

*Example 1*

About 284 grams (1 mol) of stearic acid are heated at about 160° C. with about 130 grams (1 mol) of diethylene-triamine. The reaction is complete when about 36 grams (2 mols) of water have been removed. The product of this reaction is principally stearyl imidazoline. The temperature is maintained at about 160° C. and 30 grams (0.5 mol) of urea crystals are added. The urea condenses with the imidazoline with the accompanying loss of about two mols of ammonia. The final reaction product which consists chiefly of di-(2-stearyl imidazoline)-ethylene urea, is a waxy solid having a melting point of about 50° C.

*Example 2*

Twenty parts of the di-(2-stearyl imidazoline)-ethylene urea produced in accordance with Example 1 is melted and is then added slowly to 35 parts of water containing 3 parts of 65% hydroxyacetic acid and 2 parts of a nonionic, non-fatty dispersing agent, heated to about 60° C. Forty parts of aqueous zirconyl acetate (22% $ZrO_2$) is then added slowly to the aqueous dispersion, with continued stirring while maintaining the temperature at 60° C. The resulting product which is a smooth, off-white paste, is cationic in character and has a pH of about 4 in a 1% solution. It is dispersible in cold and warm water with adequate stirring and gives a pearly cloudy solution. Application of this product to textile materials particularly floor coverings composed of wool, nylon or other synthetic fibers, using 2% to 8% in the bath based on the weight of the fiber, produces an excellent degree of soil resistance.

*Example 3*

22 parts of the product of Example 1 is melted and dispersed in 2 parts of water heated to 60° C. The water contained 2 parts of hydroxyacetic acid and 1 part of a non-fatty, nonionic dispersing agent. Approximately 33 parts of a 20% solution of stannous chloride is then slowly added with continuous stirring. The product is a smooth, off-white paste which is cationic in character. The product substantially increased the soil resistance of fibers when applied in their surfaces.

*Example 4*

Following the procedure of Example 3 utilizing 33 parts of a 25% aqueous solution of magnesium chloride and 2 parts of lactic acid there was produced a smooth, off-white paste which was readily dispersible in water and when applied to the surface of a fiber substantially increased its soil resistance.

*Example 5*

Following the procedure of Example 3 utilizing 20 parts of a 20% solution of stannous chloride and 20 parts of a 20% solution of zinc chloride there was produced a water dispersible white paste which is an excellent soil resistant finish for textiles.

*Example 6*

25 parts of the product of Example 1 are dispersed in 48 parts of water containing 2 parts of nonionic, non-fatty dispersing agent at about 60° C. 12.5 parts of zirconium oxychloride, previously dissolved in 12.5 parts of water at room temperature, are added to the above mix with continuous stirring. The resulting product is a smooth off-white paste which is strongly cationic in character, and a 1% solution of this product has a pH range of 2.0 to 2.5. When properly applied to the textile materials such as floor coverings containing wool, nylon or other synthetic fibers, it produces a high degree of soil resistance.

*Example 7*

A typical example of the application of the products of this invention is as follows: The product of Example 2 was applied to a tufted loop pile 100% nylon carpet material on a dye beck. The nylon carpet material was first scoured in accordance with mill practice followed by dyeing to a taupe shade. Subsequent to the dyeing, the dye bath was dropped and fresh water added. 6% of the product of Example 2 on the weight of the fabric, was thoroughly dispersed in a small amount of cold water and added to the water in the dye bath at a temperature of

110°–120° F. The ratio of carpet to water in the bath was 1:25–100 lbs. of carpet to 2500 lbs. of water—but may vary from about 1:20 to 1:50. The temperature of the bath was then gradually raised to 150°–160° F. and the processing of the carpet material continued for approximately 20 minutes. At this temperature the soil resistant finish exhausts rapidly onto the goods and forms a strongly adhesive flexible film on the fiber. The material was then subsequently subjected to centrifuging to about 50% moisture content, and then dried at a temperature of about 220° F. The carpet thus treated was subjected to service tests on the floor for approximately ten days during which it was exposed to normal foot traffic. An untreated carpet fabric of the same type was placed beside the treated fabric and subjected to the same foot traffic. At the end of the test the treated fabric was relatively unsoiled, as compared to the untreated material which was markedly soiled. Furthermore, the treated carpet was found to be readily cleanable by vacuuming and brushing, whereas on the untreated fabric the soil from the foot traffic showed marked adherence to the pile of the fabric. The treatment of the dyed nylon carpet showed little if any change of shade due to the application of the soil resistant compound, and also, did not effect the light fastness adversely. In this respect, exposure in the fadeometer for 40 hours showed no more fading on the treated sample than on the corresponding untreated sample. Furthermore, the treatment does not effect adversely the hand or appearance of the carpet fabric.

Any departure from the above description which conforms to the present invention is intended to be included within the scope of the claims.

What is claimed is:

**1.** A composition useful for rendering fibers soil resistant, consisting essentially of a di-(2-alkyl imidazoline)-ethylene urea condensate selected from the group consisting of di-(2-alkyl imidazoline)-ethylene urea, di-(2-alkyl imidazoline)-ethyleneaminoethylene urea and di-(2-alkyl imidazoline) - (diethyleneamino)-ethylene urea wherein said alkyl group contains 15 to 21 carbon atoms and a multivalent metal salt selected from the group consisting of water soluble salts of zirconium, tin, zinc, magnesium, calcium and barium, said salt being present in an amount equivalent to about 30 to 50% by weight of said condensate in said composition.

**2.** A composition of claim **1**, wherein said multivalent metal salt is zirconyl acetate.

**3.** A composition of claim **1**, wherein said multivalent metal salt is stannous chloride.

**4.** A composition of claim **1**, wherein said multivalent metal salt is zinc chloride.

**5.** A composition of claim **1**, wherein said multivalent metal salt is magnesium chloride.

**6.** A composition of claim **1**, wherein said multivalent metal salt is zirconium oxychloride.

**7.** A composition for application to fibers to increase their soil resistance, consisting essentially of an aqueous dispersion containing about 10 to 30% by weight of a di-(2-alkyl imidazoline)-ethylene urea condensate selected from the group consisting of di-(2-alkyl imidazoline)-ethylene urea, di-(2-alkyl imidazoline)-ethyleneaminoethylene urea and di-(2-alkyl imidazoline)-(diethyleneamino)-ethylene urea wherein said alkyl group contains 15 to 21 carbon atoms, and about 3 to 15% by weight of a multivalent metal salt selected from the group consisting of water soluble salts of zirconium, tin, zinc, magnesium, calcium and barium, the remainder of said composition being substantially water.

**8.** A composition for application of fibers to increase their soil resistance consisting essentially of an aqueous dispersion containing about 10 to 30% by weight of a di-(2-alkyl imidazoline)-ethylene urea condensate selected from the group consisting of di-(2-alkyl imidazoline)-ethylene urea, di-(2-alkyl imidazoline) - ethyleneaminoethylene urea and di-(2-alkyl imidazoline)-(diethyleneamino)-ethylene urea wherein said alkyl group contains 15 to 21 carbon atoms, about 3 to 15% by weight of a multivalent metal salt selected from the group consisting of water soluble salts of zirconium, tin, zinc, magnesium, calcium and barium, about 1 to 3% of an organic acid selected from the group consisting of hydroxyacetic acid and lactic acid, and about 1 to 3% of a non-fatty, nonionic dispersing agent the remainder of said composition being substantially water.

**9.** Textile fiber having a high resistance to soiling, having on its surface a thin coating of a composition consisting essentially of a di-(2-alkyl imidazoline)-ethylene urea condensate selected from the group consisting of a di-(2-alkyl imidazoline)-ethylene urea, di-(2-alkyl imidazoline)-ethyleneaminoethylene urea and di-(2-alkyl imidazoline)-(diethyleneamino)-ethylene urea wherein said alkyl group contains 15 to 21 carbon atoms and a multivalent metal salt selected from the group consisting of water soluble salts of zicronium, tin, zinc, magnesium, calcium and barium, said salt being present in an amount equivalent to about 30 to 50% by weight of said ethylene urea condensate, the total weight of said salt and condensate on said fiber being about 1 to 4% of the weight of said fiber.

**10.** Textile fiber having applied thereto a composition of claim **8**, wherein the total weight of said salt and condensate on said fiber is within the range from about 1% to about 4%, by weight, of the fiber.

**11.** A textile fabric having a high resistance to soiling, to which has been applied a composition consisting essentially of a di-(2-alkyl imidazoline)-ethylene urea condensate selected from the group consisting of a di-(2-alkyl imidazoline)-ethylene urea, di-(alkyl imidazoline)-ethyleneaminoethylene urea and di-(2-alkyl imidazoline)-(diethyleneamino)-ethylene urea wherein said alkyl group contains 15 to 21 carbon atoms and a multivalent metal salt selected from the group consisting of water soluble salts of zirconium, tin, zinc, magnesium, calcium and barium, said salt being present in an amount equivalent to about 30 to 50% by weight of said ethylene urea condensate, the total weight of said salt and condensate on said fabric being about 1 to 4% of the weight of said fabric.

**12.** A tufted carpet having a high resistance to soiling, having applied to its pile a composition consisting essentially of a di-(2-alkyl imidazoline)-ethylene urea condensate selected from the group consisting of di-(2-alkyl imidazoline) - ethylene urea, di-(2-alkyl imidazoline)-ethyleneaminoethylene urea and di-(2-alkyl imidazoline)-(diethyleneamino)-ethylene urea wherein said alkyl group contains 15 to 21 carbon atoms and a multivalent metal salt selected from the group consisting of water soluble salts of zirconium, tin, zinc, magnesium, calcium and barium, said salt being present in an amount equivalent to about 30 to 50% salt by weight of said ethylene urea condensate, the total weight of said salt and condensate on said carpet being about 1 to 4% of the weight of said carpet.

**13.** A process for rendering a fibrous material soil resistant, which comprises applying to said fibrous material a composition consisting essentially of an aqueous dispersion containing about 10 to about 30% by weight of a di-(2-alkyl imidazoline)-ethylene urea condensate selected from the group consisting of di-(2-alkyl imidazoline)-ethylene urea, di-(2-alkyl imidazoline)-ethyleneaminoethylene urea and di-(2-alkyl imidazoline)-(diethyleneamino)-ethylene urea wherein said alkyl group contains 15 to 21 carbon atoms and about 3 to 15% by weight of a multivalent metal salt selected from the group consisting of water soluble salts of zirconium, tin, zinc, magnesium, calcium and barium.

**14.** A process for producing a composition readily dispersible in water for use as a soil-retarding composition for textiles and the like, which comprises dispersing a di-(2-alkyl imidazoline)-ethylene urea condensate in about 1 to 2 times its weight of hot water containing an organic

fatty acid selected from the group consisting of hydroxyacetic acid and lactic acid, and a non-fatty, nonionic dispersing agent, each of said acid and said dispersing agent being present in an amount equivalent to about 1 to 3% by weight of the total composition, cooling the dispersion, and admixing the cooled dispersion with an aqueous solution of a multivalent metal salt selected from the group consisting of water soluble salts of zirconium, tin, zinc, magnesium, calcium, and barium, said alkyl group containing from 15 to 21 carbon atoms and said salt being employed in said process in an amount equivalent to about 30 to 50% by weight of said condensate in the resulting composition.



**References Cited by the Examiner**

UNITED STATES PATENTS

| | | | |
|---|---|---|---|
| 2,875,210 | 2/1959 | Bollenback et al. | 252—8.8 |
| 2,876,140 | 3/1959 | Sheehan | 252—8.8 |

SAMUEL H. BLECH, *Primary Examiner.*

JULIUS GREENWALD, *Examiner.*

J. T. FEDIGAN, *Assistant Examiner.*