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## METHOD OF IMPARTING ANTI-STATIC PROPERTIES TO TEXTILE MATERIALS

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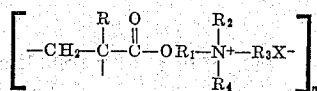
This invention relates to certain polymeric anti-static agents and to the treatment of textile materials therewith. More particularly it relates to certain polymeric alkyl sulfate quaternary ammonium salts of the acrylyloxyalkylamine type and methods for their application to textiles.

One disadvantage of textile fibers and fabrics prepared from synthetic hydrophobic materials and of all textile fibers treated with conventional resin finishes, is that they tend to develop a static electrical charge. This charge is objectionable during the manufacture of the textile and in the finished garment. During the manufacture, the static charge on the fibers or fabric interferes with their convenient handling during spinning, reeling, weaving and the like. Finished articles which are designed to drape like cotton and wool articles fail to do so properly due to their electrostatic charge and often cling uncomfortably to the wearer.

Many anti-static agents have been proposed to overcome this difficulty of synthetic textiles, however, none is entirely satisfactory either because of effectiveness or the difficulty of application to the textile material.

It is therefore an object of this invention to provide a novel and effective method for applying anti-static compositions which remain after repeated washings and dry cleanings. Another object is the provision of an anti-static agent which may be applied to synthetic textile fibers and fabrics by padding and exhausting procedures. Other objects will appear as the description of the invention proceeds.

These and other objects are accomplished by the application to textiles of a polyacrylyloxyalkyltrialkyl ammonium alkyl sulfate salt in which the alkyl radicals contain from 1 to 4 carbon atoms, together with up to an equivalent weight of water-soluble salt of a carboxylate or alkyl sulfate having from 8 to 20 carbon atoms. The invention also includes textile materials having anti-static properties produced by these methods. The textiles after treatment carry a small but effective amount of a composition, the active anti-static ingredient of which has the following formula:



where R is H, CH<sub>3</sub> or C<sub>2</sub>H<sub>5</sub>; R<sub>1</sub> is a divalent hydrocarbon radical of 1 to 4 carbon atoms; R<sub>2</sub>, R<sub>3</sub> and R<sub>4</sub> are aliphatic hydrocarbon groups of 1 to 3 carbon atoms; X is a hydrocarbon sulfate or carboxylate anion of 8 to 20 carbon atoms; and n is greater than 10.

The amount usually is about 0.05% to 2.0% or more in extreme cases of the methylsulfate salt or its equivalent based on the weight of the dry textile. The preferred amount is about 0.25% to about 1.0%, and about twice this amount for the long-chain alkyl sulfate or carboxylate salt.

While any of the quaternary compounds defined above impart satisfactory anti-static properties to the synthetic textiles, it has been found that beta-methacrylyloxyethyl-

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methyl-diethyl ammonium methylsulfate together with sodium hexadecenyl sulfate yields exceptionally satisfactory hand, color and freedom from odor.

The quaternary salts may be made by any known method. One such method consists essentially of quaternizing an appropriate acrylyloxyalkyldialkylamine in water with dimethyl sulfate followed by polymerization as described in Example II of copending application Serial No. 260,119, filed December 5, 1951.

The water-soluble salts of the alkyl sulfates having from 8 to 20 carbon atoms mentioned above are readily obtainable on the open market where they are sold as detergents. The more common ones are the sodium and amine salts of the alkyl sulfates bearing primary straight-chain alkyl groups. The common amines used are diethanolamine, triethanolamine, and N-diethylcyclohexylamine. The sodium salts of the long-chain alkyl sulfates are preferred in this invention on account of their ready availability and lower cost.

The anti-static composition is diluted to about 0.01% to 0.1% to form an aqueous solution of one of the above defined quaternary salts. The textile material is then placed in the bath, followed by slow addition of the sulfate or carboxylate salt which serves as an exhaustion aid. Alternatively, the salt can be added before the textile. For best operation, a non-ionic dispersing agent should also be present. The preferred amount of the exhaustion aid is equal to that of the anti-static agent on an equivalent weight basis.

The ratio of the bath to the textile may vary from 10 to 50 parts by weight of the bath for each part of the textile material. Usually more concentrated baths are not satisfactory, although there is no limit to the amount of dilution, except that baths which are more dilute than 100 to 1 may be uneconomical because of inconveniently long application times or limitations on the amount of textile treated per batch.

The temperature of the application bath can vary between room temperature and 190° F. Under some conditions the bath may be maintained at its boiling point, but this is usually undesirable for mill use.

This exhaustion procedure may be used on textiles bearing any of the ordinary crease-resistant resin finishes for textiles as will be apparent from certain of the following examples. Among the resins used for this purpose are the methylol ureas, methylol melamines, 1,3-bis(hydroxymethyl)-imidazo[1,2-a]pyridine and their ethers. These agents impart stiffness, crease-resistance, dimensional stability and other properties to textiles. It is preferable to conduct this process after resin treatment rather than before. Simultaneous application cannot be accomplished.

It has been found that ordinary loadings of these resins and finishes usually increase the static properties of textiles, and therefore the present invention is particularly applicable to textiles treated with these resins together with the compositions of the present invention.

The following examples are given by way of illustration, but it is to be understood that any of the quaternary ammonium compounds and water-soluble, long-chain sulfates falling within the above generic description of the invention may be substituted in like amount for any of those given in the examples with comparable results.

The treated textiles are tested as described in our copending application Serial No. 260,119.

### Example I

A. Polyethylene terephthalate stockings are placed in 20 parts by weight of an aqueous solution containing 0.5 weight percent (based on fabric weight) of poly-beta-methacrylyloxyethyl-diethylmethyl ammonium methylsulfate in water at 150° to 170° F. Then about 100 parts

of an aqueous solution containing 0.5 weight percent (on fabric weight) of sodium hexadecyl sulfate and 1.5 weight percent (on fabric weight) of the isoctylphenol condensate with about 12 mols of ethylene oxide is added to the bath over 0.5 hour. The stockings are removed and dried, and found to have excellent anti-static properties.

B. Good anti-static properties are also obtained when the isoctylphenol ethylene oxide condensate is omitted from this procedure.

#### Example II

Example I is repeated using sodium stearate in place of sodium hexadecyl sulfate. The stockings are found to have a very good anti-static rating. Likewise, very good results are obtained by adding all of the sodium stearate to the bath before the stockings, followed by working the stockings in the bath for 0.5 hour.

#### Example III

When Example I is repeated using 0.2, 0.4, 0.6 and 1.0 weight percent of the anti-static agent (based on the weight of hosiery), similar good results are obtained.

#### Example IV

When Example I is repeated with bath to fabricate ratios of 10:1 and 50:1, the anti-static properties of the stockings are greatly improved over untreated stockings.

Similar results are obtained when these applications are made at 75° to 100° F. and at 120° to 140° F.

#### Example V

Sweaters made of nylon, of cellulose acetate and of polyethylene terephthalate are treated by the procedure of Example I. The resulting anti-static ratings of the sweaters are greatly improved over those of untreated sweaters.

#### Example VI

Textiles made of nylon, polyacrylonitrile, polyethylene terephthalate and of cellulose and cellulose acetate are padded at 100% wet pick-up with an aqueous pad bath containing 15 weight percent (based on the bath) of a commercial dimethylolurea (an aqueous solution containing about 20% monomeric and 4% partially condensed dimethylolurea) and 0.5 weight percent of a commercial ammonium phosphate catalyst. The fabric is air-dried, baked 3 minutes at 350° F., soaped in a 0.25% tallow soap solution at 120° F. for 15 minutes, rinsed and dried. The resulting fabrics have very poor anti-static properties, which are greatly improved by treatment according to Example I.

The above padding operation was repeated with a methylated methylol melamine and a commercial  $(\text{NH}_4)_2\text{HPO}_4$  catalyst, followed by the exhaustion step of Example I. In this case, anti-static ratings were very good and fair for the one bearing the anti-static agent, and poor and nil for the piece of fabric bearing only the resin.

This padding operation was repeated again with 15 weight percent of 1,3-bis (hydroxymethyl)imidazolidone, 0.5 weight percent tartaric acid and 1.5 weight percent of the same polymeric quaternary ammonium anti-static agent, the latter by the exhaustion step of Example I. This treated textile had anti-static ratings of very good and good at 45% and 25% relative humidities. Without the anti-static agent the corresponding values varied from fair to nil. In every case, both with and without the anti-static agent, the pieces of textile have a pleasing hand and satisfactory degrees of crease-resistance and dimensional stability.

The present invention is particularly effective in reduc-

ing the anti-static properties of textile materials including fibers, yarns, threads and fabrics prepared from synthetic materials having high electrical resistance, such as those prepared from cellulose acetate, nylon, polyacrylonitrile, and polyethylene terephthalate. Another advantage of the present invention is that the anti-static agents may be applied with resinous, crease-resistant materials which normally increase the static effects of the synthetic polymeric textiles so treated, but when these resins are applied with the compositions of the present invention the static effects of the resin are obviated.

It will be apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof, and therefore it is not intended to be limited except as indicated in the appended claims.

We claim:

1. In the method of imparting durable, anti-static properties to textile material which normally accumulate an electrostatic charge the improvement which comprises immersing the textile material in an aqueous solution containing as an anti-static agent (1) from 0.01% to 0.1% by weight of a polyacryloyloxyalkyltrialkyl ammonium alkyl sulfate salt in which the alkyl radicals contain from 1 to 4 carbon atoms; and (2) about a molar equivalent thereof of a water-soluble salt from the group consisting of alkyl carboxylates and alkyl sulfates, the alkyl group of which contains from 8 to 20 carbon atoms.

2. The method of claim 1 in which the ratio of the application bath to the textile is between 10 to 50 parts of the former for each part by weight of textile.

3. The method of claim 1 in which the temperature of the bath is maintained at from 140° F. to its boiling point.

4. The method of treating textile material which comprises imparting anti-static properties thereto by immersing the textile material in an aqueous solution containing about 0.5% by weight, based on the textile material, of a poly-beta-methacryloyloxyethylmethyl ammonium methylsulfate, maintaining the temperature of the bath at about 150° to 170° F., adding about 100 parts of an aqueous solution containing about 0.5% by weight, based on the textile material, of a sodium hexadecyl sulfate and 1.5% by weight, based on the textile material, of an isoctylphenol condensate with about 12 mols of ethylene oxide over a period of about one half hour, and thereafter drying the said textile material.

5. The method of treating textile material which comprises imparting anti-static properties thereto by treating the textile material first with an aqueous pad bath containing 15% by weight, based on the bath, of dimethylolurea containing about 20 parts monomer and 4 parts low polymer and about 0.5% by weight of ammonium phosphate catalyst, drying the textile material thoroughly, and then immersing it in an aqueous solution containing (1) from 0.01% to 0.1% by weight of a polyacryloyloxyalkyltrialkyl ammonium alkyl sulfate salt in which the alkyl radicals contain from 1 to 4 carbon atoms; and (2) about a molar equivalent thereof of a water-soluble salt from the group consisting of alkyl carboxylates and alkyl sulfates, the alkyl group of which contains from 8 to 20 carbon atoms, and thereafter drying the said textile material.

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