TREATMENT OF TEXTILE FIBERS WITH ANTI STATIC AGENT AND PRODUCT THEREOF


No Drawing. Application February 25, 1954; Serial No. 412,661

6 Claims. (Cl. 117—138.8)

This invention relates to hydrophobic, non-cellulosic textile materials having improved qualities and more particularly to hydrophobic, non-cellulosic fibers, filaments, tapes or yarns of textile material having improved antistatic properties and to a process of imparting these antistatic properties to the textile material.

Hydrophobic textile fibers tend to accumulate electrostatic charges due to their poor electrical conductivity. This accumulation of electrostatic charges generally occurs during the working of the fibers in the textile mills, where through repeated friction of the fibers, filaments or yarns with one another and with the equipment, these electrostatic charges accumulate and seriously interfere with the spinning, drawing, twisting, weaving and knitting operations. These charges can also be annoying and troublesome when fabrics made from these hydrophobic textile fibers are in service in the form of carpets, drapes, clothing and automobile seat covers.

Although the general idea of treating textile fibers with antistatic agents is old, the problem of selecting a proper agent is by no means a simple one. In the first place, the nature of the fiber to be treated must be taken into account. Thus, agents which have been indicated in the art as suitable for wool, viscose or cellulose acetate, do not as a rule produce good anti-static effects on hydrophobic, non-cellulosic fibers. Secondly, when an effective agent has been found, it must have still other qualifications; for instance, it must be compatible with lubricants, sizes and other agents commonly applied to fibers in the mill. It must allow proper running tensions of filament during drawing and twisting operations. It must be non-corrosive to the apparatus in which the fiber will be worked, and it must be non-toxic and have no injurious dermatological effects upon the worker or upon the ultimate wearer.

This invention has as an object to improve the electrostatic qualities of hydrophobic, non-cellulosic textile fibers. A further object is to provide a hydrophobic, non-cellulosic textile fiber impregnated with an antistatic agent. A still further object is to provide a process for impregnating a hydrophobic non-cellulosic textile fiber with an antistatic agent as defined below, to improve the electrostatic qualities. Other objects will appear hereinafter.

These objects are accomplished according to the present invention by impregnating hydrophobic, non-cellulosic fibers with a special group of antistatic agents which may be defined broadly as amine salts of mixtures of alky1 esters of pentavalent phosphorus acids or acid anhydrides, wherein the amine portion is an alkanolamine, more specifically diethanol amine. These mixtures of alky1 esters of pentavalent phosphorus acids or acid anhydrides may be represented by the following general formula:

where n is a small whole number of from 1 to 6 and R is selected from the group consisting of hydrogen atoms and normal alkyl radicals of from 8 to 18 carbon atoms, and wherein on the average one half of the R's of the mixture are normal alkyl radicals and the other half are hydrogen atoms.

These phosphate esters result from the reaction of 2 moles of alcohol or mixture of alcohols, with one mole of phosphoric anhydride. In the composition obtained, about 75 to 100 wt. percent of the phosphorus is connected through an oxygen linkage to another phosphorus atom, as shown by titration with a standard alkali solution both before and after hydrolysis with water. It is believed that the greater portion of the composition is in the pyrophosphate form, although up to 25% of the composition may be in the form of orthophosphates. These compositions are subsequently added to diethanol amine in order to produce the desired salt.

These compositions are readily soluble in alcohol and are soluble or self-dispersible in water. Consequently, they can be applied to fibers from either medium with the most common and preferred method of application being padding from a dibute aqueous solution or dispersions. They can also be applied by spraying or from rollers which are in contact with the fibers at one point and the treating bath at another.

The following examples will better illustrate the nature of the present invention; however, the invention is not intended to be limited to these examples. Parts mentioned are by weight.

Example I

To 400 parts of a higher alcohol mixture having an average molecular weight of 200, and comprising a mixture whose composition is approximately 3% n-decanol, 61% n-dodecanol, 23% n-tetradecanol, 11% hexadecanol and 2% octadecanol, there was added 142 parts of phosphoric anhydride at a rate such that the temperature of the well-agitated charge was maintained at 75–80° C. while the reactor jacket temperature was held between 40° and 70° C. The reaction mass was then stirred for twelve hours at 80° C. The acid number of the product at this point was 227 and 23 mole percent of the total titratable acidic constituents was shown by potentiometric titration to be dibasic. A small sample of this intermediate was reacted with water for several hours at about 80° C. and the acid number rose to 355 and the mole percent of the total titratable acidic constituents was shown by titration to be 55%. The above described intermediate prior to reaction with water, of a high proportion of unreacted phosphorous-oxygen-phosphorus bonds.

The main body of the intermediate was added with agitation at temperatures not exceeding 80° C. to 45 parts of diethanolamine, giving a viscous paste. The pH of a 10% aqueous dispersion of this paste was 7.45.

Example II

In a manner similar to that described in Example I, a product was made in which the higher alcohol used was 260 parts of n-octanol. This product, after neutralization with an amount of diethanolamine to give pH 7.0–7.5 of a 10% aqueous solution, was soluble in water.

Example III

In a manner similar to that described in Example I, a product was made in which 316 parts of n-decanol was used. This product, after neutralization with an amount of diethanolamines to give pH 7.0–7.5 as a 10% aqueous solution, was soluble in water.

The evaluation of the antistatic quality of the treated...
Resistance: | Anti-static rating |
<table>
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<tbody>
<tr>
<td>Less than 50×10^8 ohms</td>
<td>Excellent</td>
</tr>
<tr>
<td>50-500×10^8 ohms</td>
<td>Very good</td>
</tr>
<tr>
<td>500-2500×10^8 ohms</td>
<td>Good</td>
</tr>
<tr>
<td>2500-10,000×10^8 ohms</td>
<td>Fair</td>
</tr>
<tr>
<td>10,000-100,000×10^8 ohms</td>
<td>Poor</td>
</tr>
<tr>
<td>More than 100,000×10^8 ohms</td>
<td>Nil</td>
</tr>
</tbody>
</table>

The rating of the untreated control in the noncellulosic, hydrophobic fibers herein discussed is usually nil.

Each agent whose preparation is described in Examples 1 to 3 was applied separately to taffeta fabrics of nylon, "Orlon" acrylic fiber and "Dacron" polyester fiber by padding from a dilute aqueous solution or dispersion. This was done by padding for approximately 30 seconds in 250 parts by weight of water containing 1 part by weight of the antistatic agent. After wringing and air-drying to active ingredient loadings of about 0.5% dry weight based on the weights of the fabrics, the following results were obtained (electrical resistance of the treated fabrics was measured by the above described method):

Product: | Anti-static rating |
<table>
<thead>
<tr>
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<tbody>
<tr>
<td>Ex. I</td>
<td>Excellent</td>
</tr>
<tr>
<td>Ex. II</td>
<td>Excellent</td>
</tr>
<tr>
<td>Ex. III</td>
<td>Excellent</td>
</tr>
</tbody>
</table>

With active ingredient loadings of about 0.1% based on the weights of the fabrics, the following results were obtained (electrical resistance of the treated fabrics was measured by the above described method):

Product: | Anti-static rating |
<table>
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</thead>
<tbody>
<tr>
<td>Ex. I</td>
<td>Very good</td>
</tr>
<tr>
<td>Ex. II</td>
<td>Very good</td>
</tr>
<tr>
<td>Ex. III</td>
<td>Very good</td>
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</tbody>
</table>

As evidenced by the above anti-static rating tests, the quantity of agent required per unit weight of fabric is not high. A loading of agent as low as 0.02 g. per 100 g. of fabric will produce a remarkable improvement in electrical conductivity. Loadings as high as 2% by weight of the fabric may be used. More commonly, however, loadings of 0.1 to 1% will be found both satisfactory and economical.

The preferred phosphate esters of the present invention are those in which the normal alkyd radical of the ester groups contain 12 and 14 carbon atoms. Also preferred are those made from commercial mixtures of these C12 and C14 alcohols which are marketed as technical lauril alcohol. A specific mixture available on the market is described in Example 1.

As for the chemical nature of the fiber to be treated, this invention is operable on anything which has a tendency to accumulate an electrostatic charge. The preferred materials in the textile fibers field are polyamides (e.g., nylon), polyesters (e.g., polyethylene terephthalate) and polyacrylates (e.g., polyacrylonitrile). These can be in the form of fibers, filaments, yarns or fabrics. The present invention is particularly useful in the automobile seat cover trade, since these seat covers are often made of nylon and other hydrophobic materials and they have a great tendency to accumulate an electrostatic charge.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that this invention is not limited to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A hydrophobic, non-cellulosic textile fiber having incorporated therein about 0.02 to 2.5% by weight of an antistatic agent, the latter being a mixture of alkanol amine salts of alkyl esters of phosphorus compounds selected from the group consisting of pentavalent phosphorus acids and pentavalent phosphorus acid anhydrides, said alkyl esters being characterized by the presence of —P—O—P— linkages and wherein the alkyd radical contains at least 8 carbon atoms.

2. A hydrophobic, non-cellulosic textile fiber having incorporated therein from about 0.02 to 2.5% by weight of an antistatic agent, the latter being a mixture of diethanolamine salts of phosphate esters of the general formula

3. A hydrophobic, non-cellulosic textile fiber having incorporated therein from 0.1 to 1% by weight of an antistatic agent, the latter being a mixture of diethanolamine salts of phosphate esters of the general formula

4. A hydrophobic, non-cellulosic textile fiber having incorporated therein from 0.1 to 1% by weight of an antistatic agent, the latter being a mixture of diethanolamine salts of phosphate esters which are produced by the reaction of phosphoric anhydride with an alcohol mixture comprising approximately 3% n-decanol, 61% n-dodecanol, 23% n-tetradecanol, 11% hexadecanol and 2% octadecanol, said phosphate esters having the following general formula

5. A hydrophobic, non-cellulosic textile fiber having incorporated therein from 0.1 to 1% by weight of an antistatic agent, the latter being a mixture of diethanolamine salts of phosphate esters which are produced by the reaction of phosphoric anhydride with an alcohol mixture comprising approximately 3% n-decanol, 61% n-dodecanol, 23% n-tetradecanol, 11% hexadecanol and 2% octadecanol, said phosphate esters having the following general formula

6. A hydrophobic, non-cellulosic textile fiber having incorporated therein from 0.1 to 1% by weight of an antistatic agent, the latter being a mixture of diethanolamine salts of phosphate esters which are produced by the reaction of phosphoric anhydride with an alcohol mixture comprising approximately 3% n-decanol, 61% n-dodecanol, 23% n-tetradecanol, 11% hexadecanol and 2% octadecanol, said phosphate esters having the following general formula
to 18 carbon atoms and wherein on the average one-half of the R's in the mixture are hydrogen atoms.

5. Process of improving the electrostatic qualities of hydrophobic, non-cellulosic textile fiber which comprises impregnating the same with a mixture of alkanol amine salts of phosphate esters of the general formula

\[
\begin{align*}
\text{R-O} & \quad \text{O} \\
\text{R-O} & \quad \text{P-O} \\
\text{O} & \quad \text{OR}
\end{align*}
\]

wherein \( n \) is a small whole number of from 1 to 6 and \( R \) is selected from the group consisting of hydrogen atoms and normal, aliphatic hydrocarbon radicals of from 8 to 18 carbon atoms and wherein on the average one-half of the R's in the mixture are hydrogen atoms.

6. Process of improving the electrostatic qualities of hydrophobic, non-cellulosic textile fiber which comprises impregnating the same by padding from a dilute aqueous dispersion with a mixture of diethanolamine salts of phosphate esters of the general formula

\[
\begin{align*}
\text{R-O} & \quad \text{O} \\
\text{R-O} & \quad \text{P-O} \\
\text{O} & \quad \text{OR}
\end{align*}
\]

wherein \( n \) is a small whole number of from 1 to 6 and \( R \) is selected from the group consisting of hydrogen atoms and normal, aliphatic hydrocarbon radicals of from 8 to 18 carbon atoms and wherein on the average one-half of the R's in the mixture are hydrogen atoms.

References Cited in the file of this patent

UNITED STATES PATENTS

<table>
<thead>
<tr>
<th>Patent Number</th>
<th>Inventor(s)</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,676,122</td>
<td>McCarthy</td>
<td>Apr. 20, 1954</td>
</tr>
<tr>
<td>2,676,924</td>
<td>Fortress et al.</td>
<td>Apr. 27, 1954</td>
</tr>
<tr>
<td>2,676,975</td>
<td>Fortress et al.</td>
<td>Apr. 27, 1954</td>
</tr>
</tbody>
</table>