ANTISTATIC FIBER LUBRICANT

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References Cited
UNITED STATES PATENTS
2,496,631 2/1950 Leupold et al..........................252/8.8
2,690,426 9/1954 Jefferson et al......................252/8.8
2,730,498 1/1956 Fortess et al..........................252/8.8
3,056,744 10/1962 Copes et al..........................252/8.8
3,170,877 2/1965 Beiswanger et al....................252/8.8

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Attorney—Wallenstein, Spangenberg, Hattis & Strampel

ABSTRACT
An emulsifiable antistatic fiber lubricant composition, which exhibits excellent long term emulsion stability, comprising a mixture of a mineral oil, a methyl ester of a long chain fatty acid, a neutralized phosphoric acid ester of an ethoxylated alkyl phenol or alkyl alcohol, a polyoxyethylene sorbitan tristearate and fatty alkanolamide.

2 Claims, No Drawings
ANTISTATIC FIBER LUBRICANT

This invention relates to novel compositions for use in the textile art, and, more particularly, to novel mineral oil compositions capable of forming stable oil-in-water emulsions for imparting an antistatic finish to fibers.

It is well known in the art that the application of a mineral oil lubricant to fibers greatly facilitates manufacturing techniques such as carding, weaving, spinning, etc., so that wear on the machinery is reduced and breakage of fibers is diminished.

Compositions applied to textile fibers must also have antistatic properties. The problem of preventing the accumulation of static charges on textiles has troubled the industry for a number of years. This property of retaining static electrical charges is most often encountered in the processing of synthetic fibers such as are manufactured from polyesters and polyamides, polyvinyl chloride, polyvinyl acetate and other vinyl polymerization products as well as polymers of acrylic acid derivatives.

It is advantageous to apply a textile lubricant in the form of an oil-in-water emulsion and a particular problem found in utilizing prior art oil-in-water emulsion compositions not infrequently is poor emulsion stability over prolonged periods of time. Numerous formulations have been proposed which combine known antistatic and fiber lubricants in an emulifiable composition, there is still a demand in the textile industry for economical compositions which retain uniformity after emulsification and at the same time effectively transfer to textile fibers the required antistatic and lubricating qualities.

In accordance with this invention there is provided a novel emulifiable antistatic fiber lubricant which comprises in combination one or more of each of the following materials in the stated parts by weight:

a. A mineral oil having a viscosity of from about 50 to 125 Saybolt Universal Seconds (S.U.S.) at 100°F, from about 60 to 70 parts;

b. A methyl ester of a C12 to C18 fatty acid, from about 10 to 15 parts;

c. A phosphoric acid ester of an ethylene oxide adduct (1) of a C10 to C16 linear alkyl alcohol, or (2) of a C12 to C18 branched chain alkyl alcohol, or (3) of a mono- and/or di-alkyl phenol in which the alkyl radical contains from five to 12 carbon atoms, said adducts containing from two to 20 mols of ethylene oxide per mol of the alcohol or alkyl phenol, as the case may be, said phosphoric acid ester being present as the neutralized alkali metal salt, from about six to 10 parts;

d. A polyoxyethylene sorbitan triester, from about eight to 10 parts; and

e. An alkanoamide of a C16 to C18 normally liquid fatty acid, from about 1.3 to 2 parts.

The phosphoric acid esters, which, as prepared, are generally mixtures of mono- and di-esters, are preferably made by reacting 1 mol of PO3 with from 2 to 4.5 mols of the ethylene oxide adducts of the linear alkyl alcohol or the alkyl phenol, as the case may be, at a temperature in the range up to about 100°C, in the presence or in the absence of an inert organic liquid diluent, most advantageously in the range of about 50 to 65°C. Such phosphating procedures are disclosed, for instance, in U.S. Pat. Nos. 2,441,295; 2,676,975; 2,701,258 and 3,004,056; Chemical Industries, Oct. 1942, pp. 516–521 and 557; and Organo Phosphorous Compounds, John Wiley & Sons, New York, 1950, pp. 220–225.

Illustrative alcohols and alkyl phenols from which the aforesaid ethylene oxide-adducts are made and which are reacted, for example, with PO3 to produce said phosphoric acid esters are n-octyl alcohol, n-nonyl alcohol, n-decyl alcohol, n-tridecyl alcohol, n-pentadecyl alcohol, n-hexadecyl alcohol, oleyl alcohol, n-stearyl alcohol, OxO alcohols containing from 10 to 20 carbon exemplified by OxO tridecyl alcohol, OxO hexadecyl alcohol, and OxO pentadecyl alcohol (and others as shown in U.S. Pat. No. 2,965,678; 2-ethyl octanol, and branched chain dodecanols, tetradecanols, hexadecanols and octadecanols; amyl phenol, dodecyl phenol, and didecyl phenol. Of especial usefulness are the phosphoric acid esters of the 2 to 8 mol ethylene oxide adducts of C16 to C18 linear alkyl alcohols (decyl undecyl, dodecyl, tridecyl and tetradecyl alcohols) and mixtures of two or more of said alcohols.

The phosphoric acid ester may be neutralized with an alkali (e.g. hydroxide of an alkali metal such as sodium, potassium, lithium or ammonium) in situ during the preparation of the lubricant composition or it may be added in the neutralized form. Preferably, potassium hydroxide is used as the neutralizing agent because of its better solubility, and it is conveniently added as a 45 percent solution. The exact quantity of base added, of course, is dependent upon stoichiometric considerations.

Generally, a slight excess over the stoichiometric amount is used since the desired pH range of the ultimate emulsion composition is in the range of 7.5–8.5. This final pH has been found particularly suitable because of emulsion stability demands and, also, to prevent or inhibit corrosion when the composition is contacted with textile fibers and machinery.

The methyl esters of fatty acids useful in the compositions of the present invention comprise the methyl esters of C12 to C18 saturated and unsaturated fatty acids, illustrative examples of said acids being lauric acid, myristic acid, palmitic acid, stearic acid, oleic acid, palmitoleic acid, tall oil fatty acids, and commercial and other mixtures of said acids such as coconut oil mixed fatty acids and various narrower fractions of mixed fatty acids. A particularly desirable commercial mixture of fatty acid methyl esters is that sold under the designation "CE-18" by The Procter & Gamble Company and consisting of about 70 percent oleate, linolate and stearate, 25 percent palmitate and the remainder varying amounts of laurate and myristate methyl esters.

The polyoxyethylene sorbitan triester is generally produced by condensing sorbitan triester with ethylene oxide. For the purposes of the present invention, this compound should have an HLB value of from about 8 to 15, or 40 to 75 weight percent ethylene oxide. Especially suitable is a polyoxyethylene sorbitan triester containing about 52 weight percent ethylene oxide.

The alkanolamides of the C16 to C18 normally liquid fatty acids which constitute one of the ingredients of the compositions of the present invention include, by way of illustration, oleic diethanolamide, tall oil fatty acid diethanolamide, linoleic acid diethanolamide and the corresponding fatty acid amides of glycerol mono- and di-amines, dipropanolamine, diisopropanolamine, aminoethyl ethanolamine, dibutanolamine and dibutanolamine. Oleic diethanolamide is especially satisfactory and it may be made, for instance, by condensing equal mols of diethanolamine and oleic acid, or by condensing about 1.5 to 2 mols of diethanolamine with 1 mol of oleic acid, at temperatures of 150° to 165°C. For several hours, or by condensing the oleic acid, in the form of an ester thereof, such as the triglyceride ester, with diethanolamine, or by condensing methyl oleate with diethanolamine in the presence of sodium methylate as a catalyst, or by oxymethylating the amide of oleic acid.

The order of mixing the ingredients in preparing the emulifiable lubricant compositions of the present invention is not critical but it is usually more convenient to add the several ingredients, with mixing, to the mineral oil. In the preparation of the emulsions for use in the practice of the present invention, emulsification occurs readily at temperatures above as well as below room temperatures in water, including hot water, by simply mixing the lubricant composition with the water. For example, an emulsion containing 10 percent by weight of the lubricant composition may be prepared quite readily at 10°C with no separation observable over a 24 hour period.

The preferred oil-in-water emulsions consist of 10 weight parts of the lubricating composition and 90 weight parts of water. However, this ratio may be varied considerably as, for example, 5 to 20 parts of the lubricating composition and 95
to 80 parts of water. Generally, it is not desirable to exceed about 30 percent by weight of the lubricating composition since the emulsion tends to thicken at these higher levels and stability tends to become marginal. Hence, generally speaking, said emulsions will comprise 70 to 99 parts of water and 30 to 1 parts of the lubricant composition.

The compositions of the present invention achieve highly satisfactory fiber to fiber and fiber to metal frictional properties. An accumulation of electrostatic charges on treated fibers is largely or substantially eliminated. Application to the fibers may be by any conventional means such as by immersion, spraying or roll application.

The following examples are illustrative of the invention and are not to be construed as in any way restrictive of its scope. All parts listed are by weight.

**EXAMPLE I**

A liquid emulsifiable antistatic fiber lubricant was prepared by combining the following components:

<table>
<thead>
<tr>
<th>Component</th>
<th>Parts By Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. White mineral oil (55 S.U.S. at 100° F)</td>
<td>64.5</td>
</tr>
<tr>
<td>b. Methyl esters consisting of 70% oleate, linoleate and stearate; 25% palmitate; 5% linoleate and myristate</td>
<td>13.5</td>
</tr>
<tr>
<td>c. Phosphoric acid ester of linear aliphatic alcohol (C&lt;sub&gt;18&lt;/sub&gt;-C&lt;sub&gt;22&lt;/sub&gt;; methyl radicals) of average molecular weight 180 adducted with 4 moles of ethylene oxide per mole of alcohol</td>
<td>6.1</td>
</tr>
<tr>
<td>d. 45% aqueous potassium hydroxide solution</td>
<td>1.9</td>
</tr>
<tr>
<td>e. Polyethylene sorbitan triacetate (52.5% by weight ethylene oxide)</td>
<td>10.0</td>
</tr>
<tr>
<td>f. Oleic diethanolamide</td>
<td>4.0</td>
</tr>
</tbody>
</table>

This formulation had a pH of 8.3 in an aqueous 10 percent emulsion. A 10 percent emulsion was prepared with tap water at 10°, 25° and 30° C. with slight agitation. After 24 hours, no evidence of creaming or other instability was observed.

**EXAMPLES II-V**

The same ingredients of example I were used, the proportions in parts by weight being varied as set forth below. The letter designations refer to the materials of example I.

<table>
<thead>
<tr>
<th>Component</th>
<th>Parts by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>II</td>
<td>III</td>
</tr>
<tr>
<td>(a)</td>
<td>62.5</td>
</tr>
<tr>
<td>(b)</td>
<td>13.5</td>
</tr>
<tr>
<td>(c)</td>
<td>10.0</td>
</tr>
<tr>
<td>(d)</td>
<td>3.0</td>
</tr>
<tr>
<td>(e)</td>
<td>8.0</td>
</tr>
<tr>
<td>(f)</td>
<td>3.0</td>
</tr>
</tbody>
</table>

A 10 percent emulsion was prepared using each of the compositions of examples II-V and stored at room temperature for visual observations of the stability of the emulsion. After 24 hours, little or no evidence of creaming, oiling or ring formation was perceived.

The following examples demonstrate the beneficial effects in regard to lubricity and resistance to accumulation of static charges when the compositions of the present invention are applied to particular fibers.

**EXAMPLE VI**

<table>
<thead>
<tr>
<th>Control Fiber</th>
<th>Half-Life of Static Charge Fiber Treated with 25% by Weight Emulsion of Example I</th>
</tr>
</thead>
<tbody>
<tr>
<td>nylon</td>
<td>&gt;7,200 secs.</td>
</tr>
<tr>
<td>dacron&lt;sup&gt;4&lt;/sup&gt;</td>
<td>&gt;10,000 secs.</td>
</tr>
<tr>
<td>polypropylene</td>
<td>&gt;1,800 secs.</td>
</tr>
</tbody>
</table>

<sup>1</sup> Trademark for polystyrene terephthalate polyester fiber.

<sup>2</sup> 210 denier, 35 filament polypropylene fiber.

These data demonstrate the advantageous antistatic properties of the compositions of the present invention. The tests were conducted by the resistivity method using Rothschild equipment. The value recorded as an indication of the discharge rate is the half-life of a 100 electrostatic volt charge applied through 20 strands of fiber. The Nylon and Dacron control fibers had been treated so as to remove the manufacturer's finish, while the polypropylene fiber contained this original finish.

**EXAMPLE VII**

The following results demonstrate the friction characteristics of fibers which were treated with emulsions prepared using compositions of the present invention. The tests were conducted using a Rothschild tension analyzer, which furnishes a continuous recording of tension in grams as the fibers are tested.

A. Nylon

<table>
<thead>
<tr>
<th>Control Fiber</th>
<th>Fiber Treated with 25% by Weight Emulsion of Example I</th>
</tr>
</thead>
<tbody>
<tr>
<td>30-40</td>
<td>20-30</td>
</tr>
<tr>
<td>40-50</td>
<td>12</td>
</tr>
</tbody>
</table>

Conditions: 100 ft./min.; Relative Humidity 34 percent at 70° F.

The manufacturer's finish had been extracted from the control fiber. Here, superior performance is indicated in a substantial reduction of yarn-to-yarn tension with a simultaneous elimination of fluctuation in the tension value.

B. Polypropylene Fiber (210 denier, 35 filament)

<table>
<thead>
<tr>
<th>Control Fiber</th>
<th>Fiber Treated with 25% by Weight Emulsion of Example I</th>
</tr>
</thead>
<tbody>
<tr>
<td>31-45</td>
<td>27-39</td>
</tr>
<tr>
<td>35-44</td>
<td>16-20</td>
</tr>
</tbody>
</table>

Conditions: 20 ft./min.; Relative Humidity 34 percent at 70° F.

The manufacturer's finish was allowed to remain on the control fiber. For both yarn-to-metal and yarn-to-yarn friction, fluctuation was reduced and the yarn-to-yarn tension was substantially lowered at the same time.

C. Dacron<sup>1</sup> (70 denier, 34 filament)

<table>
<thead>
<tr>
<th>Control Fiber</th>
<th>Fiber Treated with 25% by Weight Emulsion of Example I</th>
</tr>
</thead>
<tbody>
<tr>
<td>70-77</td>
<td>77-87</td>
</tr>
<tr>
<td>48-50</td>
<td>29-30</td>
</tr>
</tbody>
</table>

Conditions: 20 ft./min., Relative Humidity 38–41 percent at 68°–72° F.

<sup>1</sup> Trademark for polystyrene terephthalate polyester fiber.

The manufacturer's finish had been removed from the control fiber. Again, substantial reduction of tension with simultaneous minimization of fluctuation was indicated.
It may be noted that it is known in the art to produce anti-
static textile assistants utilizing various phosphate esters such
as are disclosed, for example, in U.S. Pat. Nos. 2,730,498;
2,842,462; 3,170,877 and 3,056,744. Neither in these patents
nor elsewhere, so far as we are aware, however, is there any
disclosure or suggestion of the compositions which we have
evolved which we have found to possess highly advantageous
properties and which are quite low in cost.

What is claimed is:
1. An oil-in-water fiber lubricant emulsion consisting essen-
tially of 70–99 parts by weight of water and 30–1 parts by
weight of a mineral oil composition consisting essentially of,
by weight,
   a. from about 60 to 70 parts of mineral oil having viscosity
      of from about 50 to 125 S.U.S. at 100°F.;
   b. from 10 to 15 parts methyl ester of a C₁₈ to C₁₉ fatty acid;
   c. from about 6 to 10 parts of an alkali metal salt of a
      phosphoric acid ester of an ethylene oxide adduct of at
      least one member selected from the group consisting of
      (i) C₄ to C₁₀ linear alkyl alcohol, (ii) C₁₀ to C₁₉ branched
      chain alkyl alcohol, and (iii) mono- and and/or di-alkyl
      phenols in which the alkyl radical contains from five to 12
      carbon atoms, said adduct containing from 2 to 20 mols
      of ethylene oxide per mol of the alcohol or alkyl phenol,
      as the case may be;
   d. from about 8 to 10 parts polyoxyethylene sorbitan
      tristearate containing 40 to 75 weight per cent ethylene
      oxide;
   e. from about 3 to 4 parts of an alkanolamide of a C₁₈ to C₁₉
      normally liquid fatty acid, said alkanolamide being
derived from an alkanolamine selected from the group
consisting of diethanolamine, glycerol mono- and di-
amines, dipropanolamine, diisopropanolamine, aminoethyl
ethanolamine, dibutanolamine, and diisobut-
olanolamine;
   f. a slight excess of alkali sufficient to yield a final emulsion
      pH between about 7.5–8.5.
2. An aqueous emulsion comprising, by weight, about 90
   parts of water and about 10 parts of the composition of claim
1.

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