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Karg

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[54] ANTISTATIC FIBER LUBRICANT

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[58] **Field of Search**.....252/8.8, 8.9; 117/139.5 F, 117/139.5 CQ

[56] **References Cited**

UNITED STATES PATENTS

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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
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[57] **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 changes 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 emulsifiable composition, yet 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 emulsifiable 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 C_{12} to C_{18} fatty acid, from about 10 to 15 parts;

c. A phosphoric acid ester of an ethylene oxide adduct (1) of a C_8 to C_{18} linear alkyl alcohol, or (2) of a C_{10} to C_{20} 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 tristearate, from about eight to 10 parts; and

e. An alkanolamide of a C_{16} to C_{18} normally liquid fatty acid, from about 3 to 4 parts.

The phosphoric acid esters, which, as prepared, are generally mixtures of mono- and di-esters, are preferably made by reacting 1 mol of P_2O_5 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 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. 220–223.

Illustrative alcohols and alkyl phenols from which the aforesaid ethylene oxide-adducts are made and which are reacted, for example, with P_2O_5 to produce said phosphoric acid esters are n-octyl alcohol, n-nonyl alcohol, n-decyl alcohol, n-dodecyl alcohol, n-tridecyl alcohol, n-tetradecyl 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, diamyl phenol, nonyl phenol, dinonyl phenol, dodecyl phenol and didodecyl phenol. Of especial usefulness are the phosphoric acid esters of the 2 to 8 mol ethylene oxide adducts of C_{10} to C_{14} 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 C_{12} to C_{18} 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, linoleate and stearate, 25 percent palmitate and the remainder varying amounts of laurate and myristate methyl esters.

The polyoxyethylene sorbitan tristearate is generally produced by condensing sorbitan tristearate 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 tristearate containing about 52 weight percent ethylene oxide.

The alkanolamides of the C_{16} to C_{18} 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 diisobutanolamine. 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 oxyethylating the amide of oleic acid.

The order of mixing the ingredients in preparing the emulsifiable 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 tap 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 range 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 limitative of its scope. All parts listed are by weight.

EXAMPLE I

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

	Parts
a. White mineral oil (55 S.U.S. at 100° F)	64.5
b. Methyl esters consisting of 70% oleate, linoleate and stearate; 25% palmitate; 5% laurate and myristate	13.5
c. Phosphoric acid ester of linear aliphatic alcohol (C_{16}-C_{14} alkyl radicals) of average molecular weight 180 adducted with 4 mols of ethylene oxide per mole of alcohol	6.1
d. 45% aqueous potassium hydroxide solution	1.9
e. Polyoxyethylene sorbitan tristearate (52.5% by weight ethylene oxide)	10.0
f. Oleic diethanolamide	4.0

This formulation had a pH of 8.3 in an aqueous 10 percent emulsion. A 10 percent emulsion was readily prepared with tap water at 10°, 25° and 50° 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.

Component	Parts by Weight			
	II	III	IV	V
(a)	62.5	60.5	66.5	70.0
(b)	13.5	13.5	13.5	13.5
(c)	10.0	10.0	6.1	8.0
(d)	3.0	3.0	1.9	2.5
(e)	8.0	10.0	8.0	6.0
(f)	3.0	3.0	4.0	1.9

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

	Control Fiber	Half-Life of Static Charge Fiber Treated with 25% by Weight Emulsion of Example I
nylon	>7,200 secs.	120 secs.
dacron[1]	>10,000 secs.	135 secs.
polypropylene	>1,800 secs.	78 secs.

[1] Trademark for polyethylene terephthalate polyester fiber.

[2] 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

	Grams of Tension Yarn-to-metal	Yarn-to-Yarn
Control Fiber	30–40	20–30
Fiber Treated with 25% by Weight Emulsion of Example I	40–50	12

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)

	Grams of Tension Yarn-to-Metal	Yarn-to-Yarn
Control Fiber	31–45	27–39
Fiber Treated with 25% by Weight Emulsion of Example I	35–44	16–20

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[1] (70 denier, 34 filament)

	Grams of Tension Yarn-to-Metal	Yarn-to-Yarn
Control Fiber	70–77	77–87
Fiber Treated with 25% by Weight Emulsion of Example I	48–50	29–30

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

[1] Trademark for polyethylene 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 antistatic 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 essentially 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 C_{12} to C_{18} 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) C_8 to C_{18} linear alkyl alcohol, (ii) C_{10} to C_{20} 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 C_{16} to C_{18} normally liquid fatty acid, said alkanolamide being derived from an alkanolamine selected from the group consisting of diethanolamine, glycerol mono- and diamines, dipropanolamine, diisopropanolamine, aminoethyl ethanolamine, dibutanolamine, and diisobutanolamine;

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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