

1

3,048,539

ANTISTATIC TEXTILE LUBRICANT FINISHES
Witold R. Kocay and Derek J. Brady, Stamford, Conn.,
assignors to American Cyanamid Company, New
York, N.Y., a corporation of Maine
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This invention relates to the finishing of textiles and has for its principal object the provision of water-soluble finishing compositions capable of imparting both lubricity and antistatic properties to textiles wherein static charge is a problem. The invention includes the novel antistatic textile lubricants themselves and also the treatment of textiles such as filaments, fibers and the like therewith.

The presence of static charges of electricity on textiles is a problem of long standing that became acute with the development of fibers from thermoplastic synthetic resins, since such fibers frequently develop such strong electrostatic charges as to make their handling difficult or impossible. The problem was solved, however, with the discovery that small quantities on the order of about 0.1-5% or more of a water-soluble quaternary ammonium salt having cationic surface-active properties would obviate or minimize the tendency of filaments, fibers, yarns, films and other textiles and related objects to develop static charges. In particular it was found that alkylamido-alkyl-substituted quaternary ammonium salts containing at least one hydroxyalkyl substituent on the quaternary nitrogen atom were among the best antistatic agents. A preferred class of compounds of this type is described for the purpose in U.S. Patent No. 2,626,876; other similar compounds that may be used are described in U.S. Patent No. 2,345,570.

Although the surface-active quaternary ammonium salts of the type described above are effective antistatic agents they possess little or no lubricating properties. The problem of lubricating the textiles therefore still remains. In textile processing, and particularly in the production of continuous filament yarn from thermoplastic resins, it is necessary to treat the yarn or other textile with a suitable fiber lubricant both to attain an efficient rate of production and to prevent abrasion and other harmful results of excessive friction. Unfortunately, many of the lubricants that have been used as coning oils and for other related purposes in textile finishing are either incompatible with the surface-active quaternary ammonium salts used as antistatic agents or present other difficulties such as the formation of hard deposits on the winding equipment and on the guides and other parts of the spinning machine. These deposits cause frictions and erratic tensions on multiple speed setups resulting in non-uniformities in processing.

Our present invention overcomes these and other related difficulties by providing water-soluble mixtures of surface-active quaternary ammonium antistatic agents with water-soluble textile lubricants that are compatible therewith. We have found a class of water-soluble materials that do not interfere with the antistatic action of cationic quaternary ammonium surface-action agents, that are compatible therewith in dilute aqueous solutions, and that have excellent lubricating action on textile filaments, fibers, threads, yarns and the like. The lubricants possessing this combination of properties are water-soluble condensation products of castor oil with ethylene oxide. The novel antistatic textile finishes of our invention therefore consist essentially in mixtures of these castor oil-ethylene oxide condensation products with water-soluble quaternary ammonium surface-active antistatic agents, preferably in the proportions hereinafter described.

2

The ethylene oxide condensation products of castor oil are well known, and a wide variety are available commercially. Those which are water-soluble contain a minimum of about 25 mols of combined ethylene oxide for each mol of castor oil; i.e., of ricinoleic acid triglyceride. From this minimum the condensation products may contain up to 300-500 or more mols of combined ethylene oxide per mole of castor oil. It will be understood that any of these water-soluble condensation products may be used in admixture with any suitable quaternary ammonium antistatic agent in preparing the compositions of our invention, and it is an important advantage that the proportion of combined ethylene oxide may be varied throughout a wide range to obtain varying degrees of hydrophilic properties in the lubricants. Thus, one manufacturer of castor oil-ethylene oxide condensates provides a scale designated as a "Hydrophile-Lipophile Balance" or HLB that increases with increasing ethylene oxide content. This permits selection of the most suitable lubricant for the type and denier of textiles to be lubricated; for example, continuous filament fibers composed predominantly of polyacrylonitrile seem to require an HLB of about 13-18, corresponding to about 40-200 mols of combined ethylene oxide for best results, whereas fibers of other materials might require a different hydrophile-lipophile balance for optimum lubricity.

As is indicated above, the antistatic agents to be mixed with castor oil-ethylene oxide condensation products in forming the compositions of the invention are known quaternary ammonium surface-active agents. These are compounds having the general formula

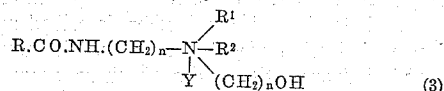


in which R is an organic radical containing a hydrophobic group and R¹, R² and R³ are short-chain alkyl, hydroxyalkyl, alkoxyalkyl or ethenoxy (polyethylene oxide) radicals, and Y is an anion such as halide, nitrate, phosphate or other acid salt group. For the compounds of this class to be surface-active the grouping



must be hydrophilic in character, which means that the R radicals should be either hydrocarbon or hydroxyalkyl chains of from 1 to not more than 3 carbon atoms or that any group having a larger number of carbon atoms should also contain a hydrophilic substituent such as a hydroxy radical or an ether radical, or both. The radical R of formula (1) may be an aliphatic hydrocarbon chain of from 7 to about 22 and preferably from 10 to 18 carbon atoms or it may consist of two or more aliphatic hydrocarbon chains connected by an ether or acid amide group. Representative quaternary ammonium antistatic agents of these classes are dodecyltrimethylammonium chloride, octadecyltriethylammonium chloride, trimethyl-(gamma-dodecyloxy - beta - hydroxypropyl) ammonium chloride and other ether-containing compounds as described in U.S. Patent No. 2,087,132 and alkylamidoalkyl-trialkylammonium salts in which the acyl radical contains at least 7 carbon atoms.

The preferred compounds, however, are those described in U.S. Patents Nos. 2,589,674, 2,626,876 and 2,345,570, which are described accurately by the formula



in which R is an aliphatic or alicyclic hydrocarbon radical containing at least 7 and preferably 12-18 carbon atoms,

R^1 and R^2 are alkyl or hydroxyalkyl radicals containing from 1 to 3 carbon atoms, inclusive, n is a whole number from 1 to 3, inclusive, and Y is an anion such as the halogen, nitrate or phosphate radical. The quaternary ammonium salts of this class have proved to be particularly effective antistatic agents for textiles composed of or containing thermoplastic synthetic resins such as nylon, acrylic fiber, polyester fiber, vinylchloridevinylacetate copolymers, Saran and cellulose acetate rayon fibers, threads and yarns.

The ratio of castor oil-ethylene oxide condensation product to quaternary ammonium salt antistatic agent in the compositions may be varied throughout a considerable range, but ordinarily there will be at least twice as much antistatic agent as castor oil-ethylene oxide lubricant in the composition. From this minimum the quantity of antistatic agent may range upwardly to as much as ten times the weight of the lubricant. It will be understood that the lubricant-antistatic mixture is ordinarily used as a solution in water, and this solution may advantageously contain other finishing agents such as inorganic salts, bleaching agents such as hydrogen peroxide and the like.

The antistatic textile lubricant compositions of the invention are particularly well suited for application to continuous filament yarn in the last stages of its manufacture. Lubricants of this type are applied to the yarn prior to or during drying on heated drying rolls; from these rolls the yarn passes through a relaxing furnace where it is heated momentarily to a high temperature. A suitable antistatic lubricant must therefore meet the following requirements. It must:

- (1) Provide good yarn lubricity.
- (2) Provide low frictional characteristics in both fiber to fiber contacts and fiber to other materials such as steel, ceramic, glass, etc.
- (3) Be effective in concentrations low enough to avoid oiliness in the treated fibers; preferably not more than about 1-2%.
- (4) Be compatible with the cationic antistatic agent used; i.e., will not inhibit or impair materially its antistatic efficiency nor precipitate it from water solution.
- (5) Have a high boiling point so that it will not volatilize in the relaxing furnace.
- (6) Not discolor or stain the yarn.
- (7) Be easy to scour.

It has been found that the mixtures of castor oil-ethylene oxide condensation products and quaternary ammonium antistatic agents described above will meet all of these requirements and give good performance when tested as coning oils. In this application the oil is applied as a dilute aqueous solution of about 2% to 5% solids, the concentration increasing with the denier of the yarn. Application is by means of a wick or a roller running in a shallow trough. The yarn contacts the roller just prior to the positive differential tension gate, and an effective coning oil aids in dampening non-uniformities in the supply package which may cause erratic tension variations during winding. It was found that a solution containing from 1.75% to 3.2% of stearamidopropyl dimethyl - beta-hydroxyethylammonium dihydrogen phosphate and from 0.3% to 0.5% of the condensation product of 1 mol of castor oil with 200 mols of ethylene oxide (HLB of 18) produced excellent results on a continuous filament yarn composed essentially of polyacrylonitrile and varying from 200/120 to 40/24 denier. This yarn was coned without further oiling and evaluated on a commercial knitting machine; and the knitting proceeded in a satisfactory manner.

The invention will be further described and illustrated by the following specific examples. It should be understood, however, that although these examples may describe certain preferred embodiments of the invention, they are given primarily for purpose of illustration and

the invention in its broader aspects is not limited thereto.

Example 1

A continuous filament yarn composed of a copolymer of 70-90% by weight of acrylonitrile and 30-10% of a mixture of vinyl acetate and other vinyl monomers was treated with a series of antistatic textile lubricant mixtures and tested for lubricity and static charge. The mixtures were applied to the yarns as aqueous solutions of 2-5% solids by means of a roller applicator.

Water-soluble castor oil-ethylene oxide condensation products are prepared by charging the castor oil into an autoclave, closing and flushing it several times with nitrogen to insure the absence of oxygen, heating to about 130°-150° C. and introducing the ethylene oxide continuously at a rate such as to maintain about 150-175 lbs. per square inch gage pressure. If desired a small quantity of an alkaline catalyst such as from 0.1% to about 1% of sodium or potassium hydroxide may be added, preferably as a 40% aqueous solution. In this case the reaction temperature and pressure may be reduced to about 100°-130° C. and 4-5 atmospheres or less.

Lubricants of this type were dissolved to an 0.1% to 1% solution in water which also contained from about 0.5% to 5% by weight of stearamidopropyl dimethyl-beta-hydroxyethylammonium dihydrogen phosphate. The solution was maintained at about 40°-60° C. during its application to the fiber. The yarn was then dried and heated and wound into cones. These were examined to determine the extent to which the yarn stuck to the cone. Knitting conditions were simulated by running the yarn from each supply cone through a constant tension gate and two porcelain eyelets of $\frac{3}{64}$ " inside diameter. The path of the yarn through the eyelets was angled so that any wax present would be scraped off and the deposit could be noted.

The best results were obtained with oils having the compositions indicated in the following table, which were rated as shown therein.

No.	Lubricant		Antistatic, percent Conc.	Rating as—	
	E.O.-C.O. Ratio ¹	Percent Conc.		Lubricant	Antistatic
1	40:1	0.5	3.2	Excellent	Excellent.
2	54:1	0.5	3.2	do	Good.
3	81:1	3.0	3.0	Oily	Poor.
4	200:1	0.5	3.2	Excellent	Excellent.
5	300:1	0.5	3.2	Fair	Fair.

¹ Mols ethylene oxide per mol of castor oil.

It will be seen that on the particular yarns under test, which range from 75 to 200 denier, the optimum molar ratios of ethylene oxide to castor oil were 40:1 and 200:1. With these yarns the best combination of lubricity and antistatic properties was obtained using about 6 parts by weight of antistatic agent for each part of the lubricant, but it will be understood that with other yarns or even with different thicknesses of the same yarn the optimum ratios might well be different. Tests of varying proportions of antistatic agent to lubricant should therefore be made with each individual type of yarn to determine those yielding the best results.

Example 2

The yarns described in Example 1 were lubricated with aqueous solutions containing castor oil-ethylene oxide condensation products having ethylene oxide to castor oil molar ratios of from 40:1 to 200:1 and containing gamma-caprylamidopropyl dimethyl-beta-hydroxyethylammonium chloride as the antistatic agent. Aqueous solutions containing 0.5% by weight of the condensation product of 200 mols of ethylene oxide with 1 mol of castor

5

oil and 3% of the antistatic agent imparted good lubricity and antistatic properties to the fiber.

Example 3

The procedure of Example 1 was applied to "Vinyon N" continuous-filament yarn, using the lubricants and antistatic agent described in that example. It was found that with this fiber the optimum molar ratio of ethylene oxide to castor oil in the lubricant was about 100:1 and that the best ratio of antistatic agent to lubricant varied with the denier of the yarn, being about 5:1 when 200 denier yarn was used.

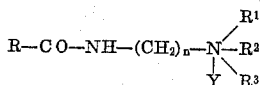
What we claim is:

1. A water-soluble antistatic textile finish composition consisting essentially of a mixture of about one part by weight of a water-soluble castor oil-ethylene oxide condensation product containing at least about 25 mols of combined ethylene oxide, and from about 2 to 10 parts of a quaternary ammonium cationic surface-active antistatic agent represented by the general formula



wherein R is an organic radical containing a hydrophobic group, R¹, R² and R³ are each radicals selected from the group consisting of hydroxyalkyl, alkoxyalkyl and ethenoxy radicals, and alkyl radicals containing from 1 to not more than 3 carbon atoms, and Y is an anion.

2. A water-soluble antistatic textile finish composition consisting essentially of a mixture of about one part by weight of a water-soluble castor oil-ethylene oxide condensation product containing at least about 25 mols of combined ethylene oxide, and from about 2 to 10 parts of a quaternary ammonium compound of the formula



wherein R is a member of the group consisting of aliphatic and alicyclic radicals containing at least 7 carbon atoms, R¹ and R² are members of the group consisting of alkyl and hydroxyalkyl radicals having from 1 to 3 carbon atoms, R³ is a hydroxyalkyl radical having from 1 to 3 carbon atoms, n is a whole number from 1 to 3, inclusive, and Y is an anion.

3. A textile finish composition according to claim 2 wherein the quaternary ammonium compound is a stearamidopropyl dimethyl-beta-hydroxyethyl-ammonium salt.

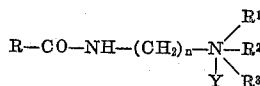
6

4. A water-soluble antistatic textile finish composition consisting essentially of a mixture of about one part by weight of the condensation product of one mol of castor oil with from about 40 to about 300 mols of ethylene oxide and from about 2 to 10 parts of a quaternary ammonium cationic surface-active agent represented by the general formula



wherein R is an organic radical containing a hydrophobic group, R¹, R² and R³ are each radicals selected from the group consisting of hydroxyalkyl, alkoxyalkyl and ethenoxy radicals, and alkyl radicals containing from 1 to not more than 3 carbon atoms, and Y is an anion.

5. A water-soluble antistatic textile finish composition consisting essentially of a mixture of about one part by weight of the condensation product of one mol of castor oil with from about 40 to about 300 mols of ethylene oxide and from about 2 to 10 parts of a quaternary ammonium compound of the formula



wherein R is a member of the group consisting of aliphatic and alicyclic radicals containing at least 7 carbon atoms, R¹ and R² are members of the group consisting of alkyl and hydroxyalkyl radicals having from 1 to 3 carbon atoms, R³ is a hydroxyalkyl radical having from 1 to 3 carbon atoms, n is a whole number from 1 to 3, inclusive, and Y is an anion.

6. A textile finish composition according to claim 5 wherein the quaternary ammonium compound is a stearamidopropyl dimethyl-beta-hydroxyethyl ammonium salt.

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