United States Patent

Habib

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[54] **ANTISTATIC FIBER TREATMENTS**

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[57] **ABSTRACT**

Static problems created during the processing of keratinous fibers and mixtures of fibers containing at least 10 percent by weight of keratinous fibers are minimized by applying to the fibers a conductive silicone lubricant composition containing at least about 0.2 percent by weight, based on the weight of the fibers, of a silicone lubricant. The silicone lubricant can be either a conductive or a non-conductive silicone, and supplementary antistatic additives can be included in the composition.

12 Claims, No Drawings

ANTISTATIC FIBER TREATMENTS

BACKGROUND OF THE INVENTION

This invention relates to a process for improving the characteristics of keratinous fibers and mixtures of fibers containing keratinous fibers to minimize static effects, increase lubricity and improve uniformity of drafting of the fibers during processing, and more particularly, to a process utilizing a conductive silicone lubricant composition.

It is well-known that fibers and filaments used in the preparation of textile materials readily accumulate electrostatic charges by friction. Certain textile processing operations such as carding, pin drafting, roving and spinning produce a considerable amount of electrostatic charge because of friction produced during the drafting operations required to attenuate the slivers and orient the fibers. Static is also produced during winding of the spun yarn at the high speeds this operation is usually run. Such an accumulation of static results in shedding of fibers which seriously affects these operations. Some lost fibers contaminate the atmosphere, while others are picked up by the rolls and create a lapping problem. The laps formed are highly objectionable because of distortion and loss of continuity of the sliver resulting in non-uniform yarn.

Certain textile fibers are particularly subject to the accumulation of static charges. Wool fibers, because of the presence of serrations and scales on the surface, generate a particularly large amount of electric charge due to friction. The problems related to the processing of woolen fibers are increased when the wool fibers have been dyed. The dyed fibers appear to generate or accumulate excessive amounts of static charges, probably due to some deleterious effect by absorption of the lubricant by the dye, and the presence of dye particles on the wool surface which increases the friction between the fibers during processing.

The accumulation of electric charges on the fibers during the processing operation is assumed to be due to the inability of the textile fibers to dissipate the charges as fast as they are generated by friction. The amount of static formed, and consequently the amount of the hairing, lapping, and shedding of fibers during carding, drafting, and spinning increases with (1) the mass of the fibers being processed, (2) the speed of the fibers through the operating zone, (3) the draft ratio, and (4) the degree of friction between the fibers in motion.

Many lubricating finishes have been suggested as useful for treating textile fibers during such processing operations as carding, drafting, and spinning, and the particular nature of the finish usually depends on the type of fibers being processed. However, the advent of new high speed, high draft ratio equipment has increased the problem of static electricity in processing fibers and rendered many of the known antistat agents and lubricant compositions obsolete. In many instances, it has been necessary to operate the new high speed machinery at lower speeds and low draft ratios because of the deficiencies of the known lubricants and antistats.

Lubricating oils utilized to improve the processing of fibers have included both natural and synthetic oils such as mineral oil and silicone polymers. The natural mineral oils have been particularly useful for treating natural fibers such as wool, whereas the synthetic polymer lubricants have found particular utility in the treatment of synthetic polymer fibers. Additionally, it has been proposed to employ water-soluble or water-dispersible surface-active agents to assist in the processing of textile fibers and yarns. These agents are valuable because of their ability to lubricate the fibers and to reduce static.

It also has been proposed to employ surface-active agents in combination with the conventional hydrocarbon oil lubricants. However, it has been found that these combinations do not spread uniformly over synthetic polymer fibers. When used on natural fibers such as wool, they are not effective at high speeds and high draft ratios, and the fibers lose their antistatic capability, probably due to absorption of the supplementary additives into the wool fibers. In some instances, this problem is overcome by spraying wool fibers with water just before or during each processing operation. Experience has shown, however, that when the antistatic properties are lost, this solution to the problem is only temporary since it is necessary to spray the fibers with water at every or nearly every operation in order to maintain the antistatic properties.

SUMMARY OF THE INVENTION

The static problems relating to the processing of keratinous fibers and mixtures of fibers containing at least 10 percent by weight of keratinous fibers have been minimized by the process of this invention which comprises applying to the fibers, a conductive silicone lubricant composition containing at least about 0.2 percent by weight, based on the weight of the fibers, of a silicone lubricant. If the silicone lubricant is electrically conductive, supplementary additives may not be required. However, if the silicone lubricant is not itself sufficiently conductive, supplementary antistats such as, for example, water-soluble ethylene oxide condensates are incorporated into the composition. Keratinous fibers treated with these compositions are easily processed through such textile operations as carding, drafting, spinning and winding.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The fibers which are treated in accordance with the process of this invention include keratinous fibers such as woolen fibers and mixtures of fibers containing at least 10 percent by weight of keratinous fibers. The process is particularly applicable to the treatment of keratinous fibers. The fiber mixtures which can be treated include mixtures of various natural fibers as well as mixtures of natural and synthetic fibers. Synthetic fibers which have been combined with keratinous fibers and successfully treated include polyester fibers such as "Dacron" of E. I du Pont de Nemours and Company, and "Fortrel" of the Celanese Corporation; polyamides such as Nylon; polyacrylonitriles such as "Orlon;" polypropylene such as "Herculon;" and polyvinyl chloride copolymers such as "Verel" of the Eastman Chemical Company.

The fibers may be treated in any desired form, for example, as loose fibers, filaments, rovings and yarns. For a continuous process, however, it is desirable to have the fibers in rope-like form such as sliver, slubbing, top, roving or yarn. Early uniform application to the fibers of the lubricating compositions of the invention, for example, prior to the first pin drafting, is desired so that the fibers are protected against static during the subsequent processing operations.

The loose fibers subjected to the process of the invention include those fibers which have been top or stock dyed, i.e., dyed prior to being subjected to the usual processing operations, such as drafting, spinning, etc. The compositions of the invention are particularly useful for treating such dyed fibers which ordinarily are difficult to process due to the presence of dye on the surface of the fibers and the resulting increased fiber-to-fiber friction.

In the process of this invention, the fibers to be processed are treated with a conductive silicone lubricant composition containing at least about 0.2 percent by weight, and preferably from about 0.4 to about 0.8 percent by weight, based on the weight of the fibers, of a silicone lubricant. The silicone lubricant can be either electrically conductive or non-conductive, but when non-conductive silicone lubricants are utilized, supplementary antistatic additives must be applied to the fibers. These may be added to the lubricant composition or to the fibers in a separate step.

Conductive silicone lubricants include the siloxane oxyalkylene block or graft copolymers obtained by condensing alkylene oxides with organic polysiloxanes. The preparation of exemplary blocked copolymers of polyethers and cyclic siloxanes is described in British Pat. No. 802,688; No. 804,369; and No. 880,022. Other copolymers are described in U.S. Pat. No. 3,140,198.

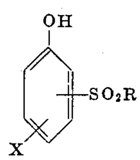
In addition to the siloxane oxyalkylene blocked copolymers in which the polysiloxane and polyoxyalkylene blocks are united through a Si—O—C-linkage, other copolymers in which the blocks are united through a Si—C-linkage may be utilized. Suitable copolymers of this type are made by reaction of polysiloxanes containing silicon bonded hydrogen with unsaturated polyethers in the presence of known catalysts for this type of condensation. Alternatively, these siloxane based polyoxyalkylene copolymers linked by a Si—C bond can also be made by the addition of alkylene oxides to silanes or siloxanes containing Si—R—OH bonds. This latter procedure is described in U.S. Pat. No. 2,846,458.

Examples of non-conductive silicones include alkylhydrogen-polysiloxanes and dialkyl-polysiloxanes as described in "Chemistry of the Silicones" by Rochow, (2nd edition, 1951). Both types of silicone lubricants are available commercially under a variety of trade names from such companies as Dow Corning Corporation, Sun Chemical Company, and Union Carbide Corporation either as pure chemicals or in the form of aqueous emulsions or dispersions.

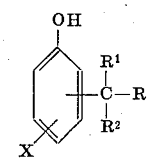
As mentioned previously, it is desirable, and in the case of lubricating compositions prepared from non-conductive silicones necessary, to include a supplementary antistatic compound. The conductive silicone lubricating compositions useful in the process of the invention will comprise, therefore, from about 1–5 parts by weight of a silicone polymer and from 0–15 parts by weight of the antistatic agent, and where the silicone polymer is not conductive, the composition should comprise from about 1–15 and preferably from about 5–15 parts of the supplementary antistat.

Any antistatic composition which is a liquid at the temperatures of operation can be utilized in combination with the silicone lubricant. Probably the largest single source of antistatic compositions are surface-active agents which can be either non-ionic or cationic. Non-ionic surface-active agents include the reaction products of compounds containing one or more reactive hydrogen atoms, for example, certain fatty acids, alcohols, phenols, amines, amides, and mercaptans, with varying amounts of an alkylene oxide, particularly ethylene oxide. Although these compounds containing reactive hydrogen atoms can be reacted with varying amounts of alkylene oxide, it must be remembered that the final product must have the liquid character and antistatic properties described previously, and for these reasons the ethylene oxide condensation products are preferred. Alcohol- and phenol-derived condensation products have been found to be particularly useful, and these include polyethylene glycols having molecular weights of about 300–500 and substituted phenolic compounds reacted with from about 8–19 moles of ethylene oxide per mole of phenolic hydroxyl group contained within said compound.

The aromatic compound containing at least one phenolic group which is to be condensed with ethylene oxide to produce the antistatic compounds useful in the invention include those having the formula



wherein R is an aryl or substituted aryl radical containing from about six to 24 carbon atoms such as phenyl, chlorophenyl, octylphenyl, or hydroxybenzyl, and X is hydrogen, chlorine, bromine, or a straight or branched chain alkyl radical containing from about one to 15 carbon atoms. The aromatic compound may also have the following structure



wherein R is an aryl or substituted aryl radical containing from six to about 24 carbon atoms, X is hydrogen, chlorine, bromine, or straight or branched chain alkyl radicals containing from about one to 15 carbon atoms, R^1 is hydrogen or an alkyl radical having from one to five carbon atoms, and R^2 is an alkyl radical having from one to five carbon atoms. The preparation of such phenolic compounds and their alkylene oxide condensation products is described in U.S. Pat. No. 3,333,983.

Examples of cationic surface-active agents which are suitable in the process of the invention include non-polymeric ternary sulphonium and especially quaternary ammonium organic compounds. Particularly useful are those compounds in which at least one of the organic radicals is a long chained aliphatic radical. The anion associated with the cation may be any convenient anion such as chloride and bromide anions. Such compounds and their preparation are described in "Surface Activity" by Moilliet, Collie, and Black (2nd edition, 1961) on pages 445 to 464. Cetyl trimethyl ammonium bromide, lauryl dimethyl benzyl ammonium chloride and tetradecyl pyridinium chloride are specific examples of such compounds.

Anionic antistatic compounds which also are useful include sulfated and sulfonated oils and fatty acids such as sulfonated castor oil and other vegetable oils, and sulfated fatty esters. Also useful as a supplementary antistat in the compositions of the invention are those condensation products obtained by condensing fatty acids with hydroxy amines. Examples of such products include the condensate obtained from oleic acid and 1,2-diethanol amine.

Mixtures of many of these supplementary antistats also are useful in the compositions of the invention, and a particularly preferred combination is obtained from polyethylene glycols and ethylene oxide condensates of aromatic phenols. In such mixtures the weight ratio of polyethylene glycol to condensed phenol is preferably in the range of from about 1 to 0.1 and 0.1 to 1.

In general, the conductive silicone lubricating compositions of this invention are prepared and applied as aqueous dispersions or emulsions although they can be applied neat. The lubricating compositions can be applied by any of the conventional means such as by spraying, immersion in a bath, brushing, or roller coating. The excess liquor can be removed, if desired, by squeezing, for example, between rollers, or by hydro-extraction, and then, if desired, the material can be dried.

The compositions of this invention can be applied from a single dispersion or emulsion of all of the ingredients, or the silicone lubricant dispersion can be applied first, dried, and the lubricant-treated fibers thereafter sprayed with the antistat. In any event it is preferred to add the silicone lubricant prior to the first drafting since this provides a method for coating all the fibers prior to drafting.

The total amount of composition applied to the fibers, and the concentration of the components in the composition, are selected to provide, after removal of the excess liquor in drying, from about 0.2 to about 7.5 percent on the fiber on a dry basis. For worsted stocks, from about 0.2 to 2.5 percent of the composition is preferred while the higher amounts, up to 7.5 percent are applied to woolen stocks which are carded. At least 0.2 percent and preferably from about 0.4 to about 0.8 percent of the coating is the silicone lubricant component. If the composition contains lesser amounts of the silicone lubricant component, the processing during drafting is non-uniform due to lack of lubricity and so much static is generated that serious distortion of the sliver results.

Emulsions prepared from the following silicone compositions are illustrative of the compositions useful in the process of the invention.

Composition A	Parts by Weight
FF–400 Fiber Finish (a conductive, ethoxylated organopolysiloxane having a viscosity of 300 centistokes at 24° C, available from Dow Corning Corp.)	1.5
Polyethylene glycol having a molecular weight of 400	2.5
Water	96.0
Composition B	
FF–400 Fiber Finish	1.25
Water	98.75
Composition C	
FF–400 Fiber Finish	1.25
Triton X–102 (Octyl phenoxy polyethoxy ethanol-nonionic wetting agent available from Rohm and Haas Co.)	2.5
Water	96.25
Composition D	
FF–400 Fiber Finish	1.25
Triton X–102	2.25
Carbowax 400 (Polyethylene glycol, of molecular weight of about 400, available from Union Carbide Co.)	0.4
Water	96.1
Composition E	
LE–45 (a 35% emulsion of a nonconductive organo polysiloxane available from Union Carbide Corp.)	3.5
Triton X–102	2.0
Carbowax 400	0.5
Water	94.0
Composition F	
FF–400	1.25
Nopcostat HS (an ethoxylated arylated phenol available from Nopco Chemical Co.)	1.85
Water	96.9
Composition G	
FF–412 Fiber Finish (a conductive, ethoxylated organo polysiloxane available from Dow Corning)	1.25
Carbowax 400	1.70
Triton X–102	0.15
Water	96.9
Composition H	
LE–45	3.5
Water	96.5

Emulsions of FF–400 are made by slowly pouring the emulsion into cold water using a high speed agitator. The concentration of the FF–400 in the emulsion should be maintained below about 6 percent and the temperature of the emulsion should be maintained below about 120° F. to prevent coagulation. Similar conditions are used to prepare stable emulsions of FF–412 fiber finish except that less agitation is required for emulsification.

When utilizing the process of this invention on blends of fibers such as a blend of 55 percent polyester and 45 percent wool, it has been found desirable to apply the silicone lubricant composition containing the silicone lubricant and the antistatic agents to the wool fibers, and thereafter blend the treated wool fibers with the polyester fibers which have been treated with the conductive silicone lubricant. Blends treated in this manner have been found to be easily processed with no significant static formation.

The following Examples illustrate the process of the invention. Unless otherwise indicated, all parts and percentages are by weight.

EXAMPLE 1

Wool top (64's grade) is passed through the aqueous emulsion of Composition A, maintained at a temperature of about 30° C. and squeezed between a pair of rolls and dried. These fibers, containing 0.5 percent silicone and 1 percent of the glycol, are run through a pin drafter set at a draft ratio of 8:1, an entering speed into the pin drafter of 11 yards/minute, and an entering wool mass into the pin drafter of approximately 1,200 grains/yd. No significant hairing or fiber drop-out is observed. On the other hand, when the wool top is passed through the same pin drafter without the silicone composition treatment, considerable fiber drop-out and lapping due to static is observed.

EXAMPLE 2

The procedure of Example 1 is repeated except that the 64's grade wool top is dyed with a neutral premetallized pearl dye using standard worsted top dyeing procedures prior to immersion in the silicone dispersion. Only slight hairing is observed for the pearl dyed wool top treated with Composition A whereas the similarly dyed but untreated wool top cannot be pin drafted at this ratio without an excessive amount of hairing, lapping and fiber loss due to static.

EXAMPLE 3

The procedure of Example 2 is repeated except that the polyethylene glycol is not included in the emulsion but is sprayed on to the dried silicone treated fibers immediately prior to the pin drafting. The fibers treated in this manner drafted satisfactorily.

EXAMPLE 4

The pearl dyed 64's wool top which is treated and pin drafted in accordance with procedure described in Example 3 is allowed to stand in air for 10 days. This top, containing 8 percent water, is again pin drafted at a draft ratio of 8:1. Even after this prolonged standing and with no additional antistat treatment, the wool top pin drafted with no evidence of static formation.

EXAMPLE 5

Pearl dyed 64's wool grade top is treated with Composition B to provide a pick-up of about 0.75 percent of the silicone lubricant and dried. This silicone coated top is then sprayed with a 25 percent aqueous solution of Syn-Fac N–95 (an ethoxylated nonyl phenol containing about 9.5 moles of ethylene oxide per mole of nonyl phenol, available from Sylvan Chemical Company, Spartanburg, South Carolina) to provide a pick-up of the phenolic compound of about 1 percent based on the weight of the fiber. These treated fibers are passed through a gill box three times to blend in the phenolic compound whereupon the top is placed in a plastic bag. After standing for about 24 hours in the plastic bag, the fibers are run through a pin drafter as in Example 1 without any difficulty or observable static formation.

EXAMPLE 6

The procedure of Example 5 is repeated except that the Syn-Fac N–95 is replaced by an equivalent amount of Triton X–102. This top also is processed through the pin drafter with no evidence of static formation.

EXAMPLE 7

Dacron tow is cut on a converter and sprayed with Dow Corning's FF–400 Fiber Finish to deposit 0.5 percent by weight, based on the weight of the Dacron, of the silicone. Wool fibers are immersed in Composition B and passed through squeeze rolls to apply about 0.5 percent of silicone on the wool. After drying, the wool is sprayed with a mixture containing one part of Carbowax 400, a polyethylene glycol having a molecular weight of about 400, and 3 parts of Nopcostat HS to deposit about 1.0 percent, based on the weight of the fibers, of the mixture. After the first pin drafting operation of the wool, the "Dacron" and wool fibers are then blended in the ratio of 55 parts of "Dacron" and 45 parts of wool. The resulting blend can be pin drafted at a draft ratio of 10:1, an entering speed of 13.5 yds./min. and an entering blend mass of 3,500 grains/yd. without evidence of the formation of static.

EXAMPLE 8

Wool top (64's grade) is dyed with a black chrome dye using standard worsted top dyeing procedures. The dyed top is immersed in Composition B and passed through squeeze rolls to apply 0.5 percent FF–400 on the wool. After drying the wool is sprayed with a mixture containing 1 part Carbowax 400 and 3 parts Nopcostat HS to deposit about 1 percent of the mixture based on the weight of the fibers. The wool is pin drafted at a draft ratio of 6:1; the mass is 2,700 grains per yard; and the entering speed is 13.4 yards per minute. No significant hairing, lapping or static is observed. This treated wool is subjected to the normal processing operations thereafter and through high speed winding of the yarn without significant evidence of static.

EXAMPLE 9

Dacron tow is cut on a converter and sprayed with Dow Corning silicone FF–400 to deposit 0.6 percent by weight, based on the weight of the Dacron. Commercial wool (64's grade) is sprayed, as it enters a pin drafter, with a mixture containing 1 part Carbowax 400 and 3 parts Nopcostat HS to deposit about 1 percent of the mixture based on the weight of the fibers. The Dacron and wool fibers are then blended using a pin drafter in the ratio of 70 parts Dacron and 30 parts wool. The resulting blend can be processed normally through pin drafting, roving, spinning and high speed winding of the yarn without significant evidence of static.

That which is claimed is:

1. A process of treating loose keratin fibers to reduce the static effects during processing which comprises:

a. applying to the fibers at least about 0.2 percent of a siloxane oxyalkylene block or graft polymer in the form of an aqueous dispersion,
b. removing at least a portion of the water from the polymer treated fibers prior to drafting, and
c. spraying the polymer treated fibers with a water-soluble ethylene oxide condensate prior to or during drafting.

2. The process of claim **1** wherein the condensate is a polyethylene glycol.

3. The process of claim **1** wherein the condensate is an ethoxylated phenol.

4. The process of claim **1** wherein the condensate is a mixture of a polyethylene glycol and an ethoxylated aryl substituted phenol in the range of from about 1 to 0.1 and 0.1 to 1.

5. The process of claim **1** wherein the keratin fibers are dyed.

6. A process of treating dyed wool top to reduce the static effects during processing which comprises:

a. immersing the top in an aqueous dispersion containing sufficient silicone polymer lubricant to deposit from about 0.2 percent to about 0.8 percent by weight of the polymer on the top,
b. removing at least a portion of the water from the polymer treated top, and
c. spraying the dried top with a water-soluble ethylene oxide condensate prior to or during the carding, drafting or winding.

7. The process of claim **16** wherein the silicone polymer lubricant is a siloxane oxyalkylene block or graft polymer.

8. The process of claim **6** wherein the ethylene oxide condensate is a polyethylene glycol.

9. The process of claim **6** wherein the condensate is an ethoxylated phenol.

10. The process of claim **6** wherein the condensate is a mixture of a polyethylene glycol and an ethoxylated phenol substance.

11. A process of treating keratinous fibers and mixtures of fibers containing at least 10 percent by weight of keratinous fibers to minimize static effects during processing, which process comprises applying to the fibers, a conductive silicone lubricant composition comprising from about 1 to 5 parts by weight of a silicone polymer and from about 1 to 15 parts by weight of a mixture of a polyethylene glycol and a polyoxyethylene phenol in the range of from about 1 to 0.1 and 0.1 to 1.

12. A process of treating wool top to reduce the static effects during processing which comprises:

a. immersing the top in an aqueous dispersion containing sufficient silicone polymer lubricant to deposit from about 0.2 percent to about 0.8 percent by weight of the polymer on the top,
b. removing at least a portion of the water from the polymer treated top, and
c. spraying the dried top with a water-soluble ethylene oxide condensate prior to or during the carding, drafting or winding.

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