1

3,518,184 TEXTILE FIBER FINISH COMPOSITION William N. Potter III, Scaford, Del., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware No Drawing. Filed May 4, 1967, Ser. No. 636,009 Int. Cl. D06m 15/10 U.S. Cl. 252-8.75 7 Claims

ABSTRACT OF THE DISCLOSURE

A finish for polyamide staple fibers which comprises

(a) a n-butyl monoether of a polyoxyalkylene glycol where the monoether has a viscosity of about 90 to 160 15 centipoises at 25° C.,

(b) a potassium salt of phosphate mono- and diesters of an aryl containing low weight polyoxyethylene alcohol,

(c) mineral oil,

(d) a sodium salt of sulfated peanut oil, and

(e) an alkali metal and/or an amine salt of oleic acid.

The art has long recognized the need of providing synthetic fibers with a coating of an antistatic lubricating 25 composition. These coating composition or, as they are commonly called, finishes repress the formation of static and minimize fiber damage during various processing steps. As indicated by the large number of such finishes in the prior art, the finding of a suitable finish composition is 30 analogous to the finding of a suitable catalyst in that specific activity cannot be predicted and otherwise similar compositions can give ineffectual or even deleterious results. Consequently, it has been found, as expected, that finishes of the prior art that have been used for the prepa- 35 like. Consequently, an antistatic material is needed. ration of yarns from heat-treated polyamide staple fibers have not been completely satisfactory.

It is, therefore, the primary objective of this invention to provide a finish having the characteristics necessary for productive processing of polyamide fibers.

It has now been discovered that improved polyamide staple fibers are obtained by the use of a finish composition comprising about 40 to 50 parts of the n-butyl monoether of a polyoxyalkylene glycol having a viscosity of 90 to 160 centipoises at 25° C., said ether being the re- 45 action product of n-butanol and a mixture of ethylene oxide and 1,2 propylene oxide in a weight ratio of about 1:1; about 20 to 40 parts of the potassium salt of a mixture of phosphate monoesters and diesters of an alcohol having the formula

wherein n is 3 to 5; about 12.0 to 15.0 parts of mineral 55 oil; about 4.0 to 6.0 parts of a sodium salt of sulfated peanut oil; and about 4.0 to 5.0 parts of an alkali metal and/or amine salt of oleic acid.

The above materials constitute the active ingredients of the compositions of this invention. In addition to these ingredients, there may be present minor amounts of additives such as buffering agents, tints, emulsification assistants, biocides and the like. The total amount of the additives should not exceed about 2% of the weight of the composition and preferably no individual additive will exceed about 1% of the total weight.

The finish composition is applied to polyamide fibers from an aqueous emulsion as an aid to processing and in controlling the accuracy of the amount deposited on the fibers. The concentration of the emulsion may range from $\,70\,$ about 1 to about 30 percent, by weight, of non-aqueous components, or "solids," so as to deposit from about 0.2

2

to about 1.0 percent "solids" on the fiber based on the weight of the fiber. Preferably, the emulsion will be deposited on the fibers by passing them across the face of a roll rotating in a trough containing the emulsion so that the fibers have about 0.5 to 0.7 percent of the "solids." If desired, application may be made by spraying or dipping and the like as known to those skilled in the art to produce the desired result.

In the production of yarns from polyamide staple fibers, 10 it is necessary that the fibers be workable, i.e., capable of movement with respect to neighboring fibers so that they may be carded, blended, drafted and the like. However, such movement should not be so readily obtained that a serious strength loss occurs. Consequently, fiber finishing compositions must provide a cohesive mass rather than slickened individual fibers. Further, this result, i.e., mass cohesion, must be obtained without any concomitant production of a material having a tacky or gummy nature. Moreover, the desired level of mass cohesion must be obtained after a heat treatment when the fibers are subjected to heat during and following drawing. Thus, it is apparent that a finish composition suitable for producing yarns from heat-treated polyamide staple fibers must possess thermal stability, lubricity, cohesion and antistatic characteristics without any one of these characteristics suffering unduly to provide an acceptable level for one of the others and that this combined result be achieved without any unacceptable consequence.

In converting staple fibers into yarns, the fibers are first carded into a web so as to open up the baled material. Due to this high degree of fiber working, appreciable static can be generated leading to a severe deterioration of carding performance due to clinging of the fibers to metal parts of the card, poor web formation and the

Further, antistatic protection must be provided without adversely affecting the cohesive strength of the web. Carded webs are wispy, low density masses of staple fibers and, as such, have very little strength. The cohesive properties imparted by finish compositions contribute significantly to the strength of the web and thus can contribute to handling performance by reducing web breakage as the web is being converted to sliver.

This invention is largely based on the discovery that a certain type of alkylene oxide condensate having a critical level of viscosity can be successfully compounded with an antistat to produce a novel blend suitable for use in the production of non-tacky carded webs having high cohesive strength and low static levels. The polyalkylene oxide condensate constitutes a major weight portion of the compositions of this invention and the viscosity of the condensate should be within the range of about 90 to 160. In general, if the viscosity is too low, the cohesive strength of the web will be low; and, if the viscosity is too high, the workability of the fibers will be poor.

The alkylene oxide condensate used in this invention is obtained by mixing about equal parts, by weight, of of ethylene oxide and propylene oxide which is then recacted with n-butanol to produce a monobuytl ether of the alkylene oxide reaction product to give an ether having an average molecular weight from about 1,000 to 1,500, preferably about 1,100 to 1,300. As is known, molecular weight is related to viscosity and since viscosity can be more meaningful when related to finish performance, it is viscosity that is of primary importance herein.

The potassium salt of a phosphate ester is obtained by neutralizing the partial esters obtained by reacting phosphorus pentoxide with the aliphatic alcohol compound. Preferably the salt will be a mixture of the monoester and diester of the partial phosphates formed from the product obtained by condensing 4 mols of ethylene oxide with 1 mol of 1-phenyl-1(4-hydroxyphenyl) ethane.

Suitable mineral oils are any highly refined paraffinic based mineral oil having a viscosity of about 5 to 15 centipoises at 25° C. Preferably the mineral oil will have a viscosity of about 10 centipoises at 25° C. to lower the high frictional properties contributed by the sulfated peanut oil salt.

The salt of the sulfated peanut oil can be obtained by reacting sulfuric acid with the glycerides in peanut oil followed by neutralization with sodium hydroxide.

The alkali metal salt of oleic acid employed in this $_{10}$ invention is preferably the potassium salt. Lower alkyl amines and lower alkanol amines may be used as the salt former; however, a preferred amine salt is formed from triethanolamine and oleic acid. Mixtures of these two salts are a preferred embodiment.

In the practice of this invention, the fiber may be formed from any of the nylons but is preferably of polyhexamethylene adipamide.

The following example is given to illustrate the invention. All parts are by weight and all percentages are by 20 weight based on the total weight of the components.

EXAMPLE

Freshly spun polyhexamethylene adipamide fibers are converged into a yarn bundle, the yarn bundle is passed 25 over a roll coated with a finish composition, so as to deposit 0.6% of "solids" based on the weight of the treated yarn, and the bundles combined into a tow havnig a denier of 580,000.

The finish composition is prepared by mixing two 30 separate components. The first component is made by mixing 598 grams of distilled water, 84.6 grams of a solution containing 45% potassium hydroxide and 193 grams of oleic acid at room temperature. To this soap solution is added, with agitation at room temperature, 191 pounds 35 (86.7 kilograms) of a material obtained by condensing equal portions of ethylene oxide and oxy-1,2-propylene with n-butanol to form the monobutyl ether of polyoxyalkylene glycols having a viscosity of 155.8 centipoises at 250 C. To the aqueous mixture of the condensate 40 and potassium oleate is added, at room temperature with agitation, 173 pounds (78.6 kilograms) of the potassium salt of a mixture of partial phosphate esters of an alcohol obtained by condensing four molecular proportions of ethylene oxide with one molecular proportion of 1-phenyl-1(4-hydroxyphenyl) ethane heated to a temperature high enough to fluidize the greasy mass; a temperature of about 60° C. being satisfactory.

The second component is prepared by mixing together 2416.5 pounds (1095 kilograms) of No. 50 mineral oil, 50 1132 pounds (514 kilograms) of the sodium salt of sulfated peanut oil (a commercial product containing about 23% of water), 97.2 pounds of diethylene gylcol (44 kilograms), 97.2 pounds (44 kilograms) of a solution containing 45%, by weight, of potassium hydroxide, 553 55 pounds (251 kilograms) of oleic acid, 209 pounds (95 kilograms) of triethanol amine, 209 pounds (95 kilograms) of a solution containing 40% of the sodium salt of o-phenylphenol, and 147 pounds (66.8 kilograms) of utes and then cooled to room temperature. This material is then used in the preparation of an aqueous emulsion by slowly adding it at a ratio of 111.8 parts to 378.2 parts of water, with vigorous agitation, to give an emulsion having about 20% "solids."

To the aqueous mixture of the first component is then added, with agitation, 490 pounds (222 kilograms) of the 20% "solids" emulsion designated as the second component. Enough water is then added to the mixture to form a final emulsion containing about 25% "solids."

The fibers are partially drawn by the use of snubbing pins and drawing is completed, by passing the fibers over a hot plate heated to a temperature of 185° C., to a d.p.f. of 2.3. The drawn fibers pass directly into an oven having a roll at the far end for reversal of the tow. The tempera- 75

ture of the oven is 185° C. at the entry/exit end and 195° C. at the roll end. The fibers are exposed to heat for a period of about 20 seconds. The fibers are cooled and cut into staple fibers 1.5 inches (3.8 centimeters) in length. The relatively smooth cylindrical staple fibers are then packaged by being compressed in a high-pressure baler so as to deform the filaments.

When the staple fibers described above are carded into a web, the web has a static level of 0.5 millivolt of static electricity as measured by a Type 42W Electrostatic Locator manufactured by the Sunco Co. Inc. of Lansdale, Pa. In addition to a reduction in static level as compared to a similarly used commercial finish composition, an increase in web strength of over 20% is obtained.

As a result of this increase in web strength, markedly improved performance is obtained since fewer web breaks are experienced between the doffer comb and the trumpet that condenses the web into sliver.

In evaluation of similar compositions it was found that they did not perform in the desired manner. As examples of compositions failing to provide the advantages of this invention there may be mentioned (1) the composition obtained by removing the phosphate ester, and substituting a like amount of the alcohol from which it is prepared, from the finish composition described in the example, and (2) the composition obtained by substituting comparable amounts of a salt of an alkyl phosphate ester and the same alkylene oxide condensate having a viscosity of 64 centipoises at 25° C. for the phosphate and condensate of the example.

What is claimed is:

1. A textile fiber finish composition which consists essentially of

(a) about 40 to 50 parts by weight of the n-butyl monoether of a polyoxyalkylene glycol having a viscosity of 90 to 160 centipoises at 25° C., said ether being the reaction product of n-butanol and a mixture of ethylene oxide and 1,2-propylene oxide in a weight ratio of about 1:1;

(b) about 20 to 40 parts by weight of the potassium salt of a mixture of phosphate monoesters and diesters of an alcohol having the formula

$$\begin{array}{c|c} H \\ \hline -C \\ CH_3 \\ \hline \end{array} - (0-CH_2-CH_2-)_nOH \\$$

wherein n is 3 to 5;

45

(c) about 12.0 to 15.0 parts by weight of mineral oil; (d) about 4.0 to 6.0 parts by weight of a sodium salt

of sulfated peanut oil; and

(e) about 4.0 to 5.0 parts by weight of an oleic acid salt of an alkali metal, an amine selected from the group consisting of lower alkyl amines and lower alkanolamines or mixtures of oleic acid salts of said alkali metal and said amine.

2. A textile fiber finish composition as defined in claim water. This mixture is heated to 60° C., stirred 30 min- 60 1 where the salt of said oleic acid is the potassium salt, the triethanolamine salt or mixtures of said salts.

3. A textile fiber finish composition as defined in claim 1 where said mineral oil has a viscosity of about 5 to 10 centipoises at 25° C.

4. Polyamide fibers carrying about 0.2 to 1.0 percent based on the weight of said polyamide of a composition having

(a) about 40 to 50 parts by weight of the n-butyl monoether of a polyoxyalkylene ether having a viscosity of 90 to 160 centipoises at 25° C., said glycol being the reaction product of n-butanol and a mixture of ethylene oxide and 1,2-propylene oxide in a weight ratio of about 1:1,

(b) about 20 to 40 parts by weight of the potassium salt of a mixture of phosphate monoesters and diesters 5

of an alcohol having the formula

wherein n is 3 to 5;

(c) about 12.0 to 15.0 parts by weight of mineral oil;

(d) about 4.0 to 6.0 parts by weight of a sodium salt of 10 sulfated peanut oil; and

(e) about 4.0 to 5.0 parts by weight of an oleic acid salt of an alkali metal, an amine selected from the group consisting of lower alkyl amines and lower alkanolamines or mixtures of oleic acid salts of said 15 alkali metal and said amine.

5. Polyamide fibers as defined in claim 4 where said polyamide is polyhexamethylene adipamide.

6. In a process for preparing yarn from polyamide staple fibers the improvement of depositing on said polyamide fibers before carding about 0.2 to 1.0 percent based on the weight of said polyamide of a composition having

(a) about 40 to 50 parts by weight of the n-butyl monoether of a polyoxyalkylene ether having a viscosity of 90 to 160 centipoises at 25° C., said glycol being the reaction product of n-butanol and a mixture of ethylene oxide and 1,2-propylene oxide in a weight ratio of about 1:1;

(b) about 20 to 40 parts by weight of the potassium salt of a mixture of phosphate monoesters and di-

esters of an alcohol having the formula

wherein n is 3 to 5;

(c) about 12.0 to 15.0 parts by weight of mineral oil;

(d) about 4.0 to 6.0 parts by weight of a sodium salt of sulfated peanut oil; and

(e) about 4.0 to 5.0 parts by weight of an oleic acid salt of an alkali metal, an amine selected from the group consisting of lower alkyl amines and lower alkanolamines or mixtures of oleic acid salts of said alkali metal and said amine.

7. A process as defined in claim 6 where said polyamide is polyhexamethylene adipamide.

References Cited

UNITED STATES PATENTS

2,150,568	3/1939	Whitehead 252-8.7 X
2,407,105		
2,425,755	8/1947	Roberts et al 252-8.9 X
2,865,855	12/1958	Chandler 117—139.5 X
3,170,877	2/1965	Beiswanger et al 252—8.9 X

HERBERT B. GUYNN, Primary Examiner

U.S. Cl. X.R.

30 117—138.8, 139.5; 252—8.7, 8.9; 260—951

UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No	3,518,184	Dated	June 30,	1970
Inventor(s)	William N. Pot	ter, III	- 	

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In Col. 4, line 69, after "polyoxyalkylene" and before "having" the word "ether" is deleted and the word --- glycol --- is substituted therefor.

In Col. 4, line 70, after "said" and before "being" the word "glycol" is deleted and the word --- ether --- is substituted therefor.

In Col. 5, line 24, after "polyoxyalkylene" and before "having" the word "ether" is deleted and the word --- glycol --- is substituted therefor.

In Col. 5, line 25, after "said" and before "being" the word "glycol" is deleted and the word --- ether --- is substituted therefor.

SIGNED AND SEALED DEC 8 - 1970

(SEAL)
Attest:

Edward M. Fletcher, Jr.
Attesting Officer

WILLIAM E. SCHUYLER, JR. Commissioner of Patenta