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LUBRICATING AGENTS

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This invention relates to lubricating agents and relates more particularly to lubricating agents employed in the processing of filamentary materials.

In the commercial production of textile fabrics by circular knitting, the yarns being knitted must pass rapidly over many metallic guide surfaces. The "drag" of the yarns, that is, the friction between the yarns and these guide surfaces, is an important factor in the efficiency of the knitting process. In fact, in some types of circular knitting machines this factor causes the articles produced thereby to show runs or holes when the drag is too great.

It is an object of this invention to provide new and improved lubricating agents for application to filamentary materials, which agents will act to decrease greatly the drag of said filamentary materials and will enable said materials to be knitted on circular knitting machines at high speeds to produce fabrics of excellent quality.

Another object of this invention is the provision of novel and highly effective lubricating agents for yarns of cellulose acetate or other organic derivative of cellulose.

Other objects of this invention will be apparent from the following detailed description and claims.

The lubricating agents of this invention contain a mixture of eicosane and an amine salt of an acid phosphate of a higher fatty alcohol. Advantageously, they also contain an amine salt of a higher fatty acid.

The lubricating compositions of this invention are preferably prepared by mixing the eicosane in molten condition with an ester-forming phosphorus acid derivative, such as phosphorus pentoxide, and adding to the mixture a higher fatty alcohol, in amount sufficient to form an acidic phosphate of said alcohol, together with a higher fatty acid, while maintaining the said mixture at an elevated temperature. Thereafter an amine is added to the mixture to form the salts of the acidic phosphate and of the higher fatty acid.

The amine used in the production of the lubricating compositions preferably contains a hydroxyl group. Examples of suitable tertiary amines of this type are dibutylethanolamine, triethanolamine, diethylethanolamine and triisopropanolamine. Amines containing no hydroxyl substituents, e. g. N,N-diethylcyclohexylamine, may be used, if desired. Although optimum results have been obtained using tertiary amines, secondary amines such as diethylamine or diethanolamine may be employed instead.

The alcohol used to form the acidic phosphate ester advantageously contains at least 12 carbon atoms, preferably about 14 to 20 carbon atoms. Mixtures of alcohols for example, blends of homologous or isomeric alcohols, may be employed if desired. Examples of suitable alcohols are octadecyl (stearyl), octadecenyl (oleyl), and hexadecyl (cetyl), or mixtures of the same. Examples of ester-forming phosphorus acid derivatives suitable for reaction with the alcohol are phosphorus pentoxide, which is preferred, and phosphorus tetroxide. Phosphorus

pentachloride may also be used but is somewhat less desirable since it liberates corrosive hydrochloric acid during the esterification reaction. The relative amounts of the ester-forming phosphorus acid derivative and the higher fatty alcohol may be such that the resulting ester contains about 1 to 2, preferably 1.3 to 1.6, higher fatty alcohol radicals per phosphorus atom.

The higher fatty acid used in the production of the compositions of this invention may contain, for example, 14 to 20, preferably 18 carbon atoms. Examples of suitable higher fatty acids are stearic acid and oleic acid.

The proportions of the ingredients of the compositions of this invention are advantageously within the following ranges: eicosane, about 20 to 45%, preferably about 30 to 35%; amine salt of acidic higher fatty phosphate, about 25 to 45%, preferably about 30 to 40%; amine salt of higher fatty acid, about 10 to 40%, preferably about 20 to 25%. If desired, there may be employed a slight excess of amine over that necessary to form the salts of the acidic constituents. For best results the amount of amine is such that the pH of an aqueous dispersion of the composition, of 20 to 30% concentration, is about 7.5 to 8.5.

The lubricating compositions of this invention may be applied to the filamentary material in any desired manner. For example, the composition may be applied as a molten mixture, as a solution in a volatile solvent, or, most desirably, as a dispersion in water. The amine salts of the phosphate esters and fatty acids act, in part, as emulsifying agents so that the composition disperses easily in water. Suitable concentrations of the composition in water are, for example, 5 to 35%, preferably 20 to 30% by weight. Advantageously, the aqueous dispersions are prepared by adding the heated molten lubricating composition to cool water under strong agitation. If desired, an additional emulsifying agent may be incorporated into the mixture to improve the stability of the dispersion. Examples of such emulsifying agents are those polyethylene oxide derivatives known as "Igepal CA–630," "Brij 30," "Lipal 30W," "DS–160" and "Span 85."

It has been found that aqueous emulsions or dispersions produced in accordance with this invention have superior fluidity and stability when, in the preparation of the composition, the higher fatty acid is present during the esterification reaction between the higher fatty alcohol and the phosphorus pentoxide or other ester-forming phosphorus acid derivative. If the higher fatty acid is not present at this time, but is added after the esterification reaction has been substantially completed, the resulting emulsion is much more viscous.

The lubricating compositions of this invention are particularly useful for use on filamentary materials of cellulose acetate of about 53 to 55% acetyl value, calculated as combined acetic acid. They may also be applied to filamentary materials of other organic derivatives of cellulose, e. g. other cellulose esters, such as cellulose triacetate, cellulose propionate, cellulose butyrate, cellulose acetate-propionate and cellulose acetate-butyrate, or cellulose ethers, such as ethyl cellulose or benzyl cellulose. They are also useful for the lubrication of other filamentary materials, such as polyamides, e. g. nylon, polyesters such as polyethylene terephthalate and polymers and copolymers of acrylonitrile. The compositions may be applied to continuous filaments as they emerge, in the form of a yarn, from the spinning cabinet or metier in which they are formed. Similarly, they may be applied to tows, or relatively thick bundles, of continuous filaments before said bundles are severed into staple fibers of the desired length, or they may be applied to the staple fibers themselves at any desired stage in the processing of said staple fibers. Generally, the amount of the composition

applied to the filamentary material is about 1 to 4%, based on the weight of said filamentary material.

The use of the lubricating compositions of this invention greatly reduces the drag occurring when filamentary materials are passed over guide surfaces. In fact, their application to cellulose acetate yarns has made it possible to knit such yarns in a highly successful manner on circular knitting machines under such drastic conditions as had previously precluded the use of cellulose acetate yarns carrying the conventional lubricating agents.

In addition, the lubricating compositions of this invention show practically no change in characteristics on aging, either in the presence or absence of small amounts of water, have practically no tendency to corrode black iron, have excellent antistatic properties, improve the flex abrasion characteristics of the filamentary material, and may be very easily removed from the filamentary material by scouring in water.

The following examples are given to illustrate the invention further. All proportions are by weight unless otherwise indicated.

Example I

33.6 parts of the crystalline product consisting predominantly of n-eicosane, having a melting point of 96.5 F. and having 10% and 90% boiling points of 383° F. and 410° F., respectively, measured at 10 mm. Hg absolute, sold under the name "Eicosane" by Atlantic Refining Company, are melted and 4.3 parts of finely divided phosphorus pentoxide are added thereto and mixed for 5 minutes to form a slurry. The temperature of the slurry is adjusted to 60° C. and then 24.4 parts of oleyl alcohol and 13.0 parts of oleic acid are added quickly. The ensuing reaction causes the temperature to rise. Then the mixture is heated further and maintained at a temperature of 80° C. for 30 minutes while it is stirred vigorously. Thereafter 19.8 parts of dibutylethanolamine and 4.9 parts of the emulsifying agent known as "Igepal CA–630" (a non-ionic nonyl phenoxy ether of a polyethylene glycol containing 8 to 12 ethoxy linkages) are blended into the mixture. On cooling the product is a soft wax. This soft wax is emulsified in water by heating the wax to 80° C., at which temperature it is molten, and then adding it, while stirring, to 4 times its weight of water having a temperature of 25° C. The emulsion is applied to dull cellulose acetate continuous filament yarn in amount sufficient to deposit thereon 2.5%, based on the weight of the yarn. The yarn is then knitted on a Wildman Interlock circular knitting machine. Knitted fabrics of excellent quality are produced.

Example II

Example I is repeated except that the "Igepal CA–630" is omitted. The resulting mixture is applied in molten condition, at 35 to 40° C., to the yarn. The lubricating effect is substantially the same as in Example I.

Example III

Example I is repeated except that 35 parts of eicosane are employed; 24.5 parts of stearyl alcohol and 13.2 parts of stearic acid are substituted for the oleyl alcohol and oleic acid, respectively; 18 parts of triethanolamine are substituted for the dibutylethanolamine; and the stearic acid is added after the stearyl alcohol has been reacted in the mixture for the 30 minute period. The product is a hard wax. This wax is emulsified by first mixing it thoroughly with twice its weight of water having a temperature of 80° C., to form a paste, cooling the paste and adding the desired amount of cold water. The resulting composition has substantially the same lubricating properties as that of Example I.

Example IV

Thirty parts of the triethanolamine ester of stearic acid and thirty parts of the triethanolamine salt of stearyl phosphate are mixed in forty parts of molten eicosane until a homogeneous mixture results. On cooling, a hard wax results. This wax is emulsified by first mixing it thoroughly with twice its weight of water having a temperature of 80° C., to form a paste, cooling the paste and adding the desired amount of cold water. The product has good lubricating properties.

It is to be understood that the foregoing detailed description is merely given by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention, what we desire to secure by Letters Patent is:

1. A lubricating composition for filamentary materials, comprising about 20 to 45 parts by weight of n-eicosane and about 25 to 45 parts by weight of an amine salt of an acid phosphate of a higher fatty alcohol.

2. Textile fibers carrying a coating of the lubricating composition of claim 1.

3. A lubricating composition for filamentary materials, comprising about 20 to 45% of n-eicosane, about 25 to 45% of an amine sale of an acid phosphate of a higher fatty alcohol, and about 10 to 40% of an amine salt of a higher fatty acid.

4. A lubricating composition for filamentary materials, comprising about 20 to 45 parts by weight of n-eicosane, about 25 to 45 parts by weight of an amine salt of an acid phosphate of a higher fatty alcohol, and about 10 to 40 parts by weight of an amine salt of a higher fatty acid, dispersed to a concentration of about 20 to 30% in an aqueous medium in the presence of a non-ionic emulsifying agent.

5. A lubricating composition as set forth in claim 3, said amine in each case containing an alcoholic hydroxyl group.

6. A lubricating composition as set forth in claim 3, in which said amine is dibutylethanolamine.

7. A lubricating composition as set forth in claim 3, in which said amine is triethanolamine.

8. Cellulose acetate filamentary material carrying a coating of the lubricating composition of claim 3 in which the tertiary amine in each case contains an alcoholic hydroxyl group.

9. A lubricating composition for filamentary materials, comprising about 30 to 35% of n-eicosane, about 30 to 40% of a tertiary amine salt of an acid phosphate of oleyl alcohol, and about 20 to 30% of a tertiary amine salt of oleic acid, said tertiary amine in each case containing an alcoholic hydroxyl group.

10. A lubricating composition for filamentary materials, comprising about 35 to 45% of n-eicosane, about 25 to 35% of a tertiary amine salt of an acid phosphate of stearyl alcohol, and about 25 to 35% of a tertiary amine salt of stearic acid, said tertiary amine in each case containing an alcoholic hydroxyl group.

11. Process for the production of lubricating compositions, which comprises forming a mixture of about 20 to 45 parts by weight of molten n-eicosane and an ester-forming phosphorus acid derivative, adding to the mixture a higher fatty alcohol, in amount sufficient to react with said phosphorus acid derivative to form an acidic phosphate of said alcohol, while maintaining said mixture at an elevated esterification temperature, and thereafter adding an amine to the resulting mixture to form an amine salt of said acidic phosphate, said phosphorus acid derivative, higher fatty alcohol and amine being added in amount sufficient to form about 25 to 45 parts by weight of the amine salt of said acidic phosphate.

12. Process for the production of lubricating compositions, which comprises forming a mixture of molten n-eicosane and an ester-forming phosphorus acid derivative, adding to the mixture a higher fatty alcohol, in amount sufficient to react with said phosphorus acid derivative to form an acidic phosphate of said alcohol, together with a higher fatty acid, while maintaining said mixture at an elevated esterification temperature, adding

an amine to the resulting mixture to form amine salts of said acidic phosphate and said higher fatty acid, and thereafter emulsifying the mixture in water.

13. Process as set forth in claim 12 wherein a nonionic emulsifying agent is present during said step of emulsifying.

14. Process as set forth in claim 12 in which said amine contains an alcoholic hydroxyl group and said phosphorus acid derivative is phosphorus pentoxide.

15. Process for the production of lubricating compositions, which comprises forming a mixture of about 20 to 45 parts by weight of molten n-eicosane and an ester-forming phosphorus acid derivative, adding to the mixture a higher fatty alcohol, in amount sufficient to react with said phosphorus acid derivative to form an acidic phosphate of said alcohol, together with a higher fatty acid, while maintaining said mixture at an elevated esterification temperature, adding an amine to the resulting mixture to form amine salts of said acidic phosphate and said higher fatty acid, said phosphorus acid derivative, higher fatty alcohol, higher fatty acid and amine being added in amount sufficient to form about 25 to 45 parts by weight of the amine salt of said acidic phosphate and about 10 to 40 parts by weight of the amine salt of said higher fatty acid, and thereafter emulsifying the mixture in water to a concentration of about 20 to 30%.

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