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TEXTILE LUBRICANT

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This invention relates to the treatment of textile materials and relates more particularly to the lubrication and conditioning of fibers and filaments having a basis of cellulose acetate or other organic derivative of cellulose, or having a basis of partially saponified cellulose acetate or other partially saponified organic acid ester of cellulose, to render the same more amenable to textile operations such as carding, drafting, spinning, twisting, coning, pining, hanking, weaving, knitting and the like.

An object of this invention is the provision of an improved conditioning agent for the treatment of textile materials such as filaments and fibers having a basis of cellulose acetate or other organic derivative of cellulose or of partially saponified cellulose acetate or other organic acid ester of cellulose to render said textile materials more amenable to textile operations.

Another object of this invention is the provision of a single application conditioning agent adapted to be applied to cellulose acetate or other organic derivative of cellulose filaments, as said filaments emerge from the spinning cabinet in which they are formed, for lubricating and conditioning the same for the numerous textile operations to which said materials are subjected, either in continuous filament or staple fiber form, prior to being processed to a finished fabric.

A further object of this invention is the conditioning of cellulose acetate or other organic derivative of cellulose staple fibers in unsaponified or partially saponified form with a new and improved conditioning agent which renders the cellulose acetate or other organic derivative of cellulose staple fibers treated therewith anti-static and eminently suitable for processing into spun yarns on the cotton system.

Other objects of this invention will appear from the following detailed description.

Various compositions comprising vegetable, animal and mineral oils, both normal and specially treated, in combination with various other substances have been employed for lubricating and conditioning filaments and staple fibers having a basis of cellulose acetate or other organic derivatives of cellulose in unsaponified or partially saponified form to render the same more amenable to the various textile operations generally employed in forming said materials into fabrics. In order to process continuous filaments of cellulose acetate or other organic derivative of cellulose material into staple fiber suitable for the production of fine spun yarns, the continuous filaments are usually treated with a suitable lu-

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bricant composition as they emerge from the spinning cabinet or metier in which they are formed and then, after a plurality of said lubricated continuous filaments are associated together in the form of a bundle or tow, a separate conditioning agent is applied thereto in the desired amount. The lubricated and conditioned continuous filaments thus obtained are then severed into staple fibers of the desired length. In the case where partially saponified fibers of cellulose acetate or other organic acid ester of cellulose are desired, the continuous filaments are cut into staple fiber form, the resulting staple fibers subjected to a partial saponification process whereby the outer portions of the individual staple fibers are converted to regenerated cellulose, and the staple fibers thus obtained treated with a lubricant composition and then spun into yarn.

The lubricating composition is applied to impart a suitable degree of flexibility as well as desirable frictional qualities so that the staple fibers obtained when the continuous filaments are cut may be processed satisfactorily into fine spun yarns. The conditioning agent is applied to said filaments to render the staple fibers anti-static so as to avoid the generation of excessive static electricity which would render processing exceedingly difficult if not impossible. The separate application of lubricating composition and a conditioning composition is not only uneconomical but is also quite inconvenient. The development of a satisfactory single composition which could be applied, for example, to continuous filaments of cellulose acetate or other organic derivatives of cellulose as they emerge from the spinning cabinet or metier where they are formed or to partially saponified cellulose ester staple materials as well, which will produce the desired lubricating and conditioning action on cellulose acetate or other organic derivative of cellulose staple fiber materials and which is prepared with only a limited number of easily available components has long been a desideratum in the art.

We have now found that staple fibers of both cellulose acetate or other organic derivative of cellulose materials, or partially saponified cellulose acetate or other partially saponified organic acid ester of cellulose may be satisfactorily lubricated and conditioned for spinning operations by the application thereto of a single-application lubricating and conditioning composition comprising a partial mixed alkyl phosphate, an alkylamine ester and/or amide of a long chain aliphatic acid, a tertiary amine such as diethyl

cyclohexylamine, dibutyl ethanolamine, or triethanolamine and an alkyl phenol such as diamyl phenol, triamyl phenol or dibutyl-m-cresol all mixed with mineral oil in suitable proportions. Although our novel composition is free of sulfonated and/or sulfated compounds, not only is the composition thoroughly dispersed over the fibers on application thereto but the fibers obtained are completely anti-static so that a superior degree of lubrication and conditioning is achieved.

While the prior textile lubricating and conditioning compositions, comprising mixtures of a simple partial alkyl phosphate, a tertiary amine, an alkyl phenol and mineral oil, have good stability and lubricating and conditioning properties, the compositions produced according to the instant invention have even better anti-static properties, so that considerably less of the lubricating and conditioning composition need be applied to the textile. At the same time, the stability and viscosity characteristics of our lubricating compositions are also greatly improved.

In the compositions of our invention, there is present at least about 12% of a partial mixed alkyl phosphate in which the total weight of any one alkyl radical does not exceed 75% of the total weight of all of the alkyl radicals in the phosphate. In our partial mixed alkyl phosphate each of the alkyl radicals contains 8 to 13 carbon atoms, the average number of carbon atoms per alkyl radical is 9 to 11, preferably about 10, and the average number of alkyl radicals per phosphorus atom is $1\frac{1}{2}$ to $1\frac{3}{4}$. These partial mixed esters may be produced by reacting phosphorus pentoxide with a blend of aliphatic monohydric alcohols in which each alcohol has 8 to 13 carbon atoms, and in which the average number of carbon atoms is 9 to 11. Preferably, this reaction is carried out by finely dispersing the phosphorus pentoxide in an inert diluent, such as mineral oil, adding thereto the blend of alcohols, and then heating the resulting mixture to about 50 to 100° C. according to the method described in the copending application of Fred Fortess et al., Serial No. 182,614, filed August 31, 1950 to produce a mixture of the inert diluent and the mixed mono-and-di-phosphates of the blend of alcohols. The mole ration of the alcohol blend to the phosphorus pentoxide should range between about 3:1 and about $3\frac{1}{2}$:1.

Alcohols which may be present in the blend include n-octyl alcohol, 3,5,5 trimethylhexanol, n-decanol, 2 n-butyl hexanol-1, 2-ethyl octanol-1, 2-n-butyl octanol-1, tridecanols, 2-ethylhexanol, octanol-2, diisobutyl carbinol and undecanols. The blend of alcohols should contain no more than 75% of any one single alcohol. Suitable blends of alcohols which may be reacted with phosphorus pentoxide are, for example, a mixture of about 1 part by weight of 2-n-butyl hexanol-1, 1 part by weight of 2-ethyl octanol-1, and 2 parts by weight of n-decanol; a mixture of 1 mole of 2-butyl octanol-1 and 1 mole of octanol-2; a mixture of 1 mole of 2-butyl octanol-1 and 1 mole of a mixture of 1 mole of 2-butyl octanol-1 and 1 mole of trimethylhexanol. Preferably, the blend should contain at least one branch chain alcohol, with the branch chain alcohols constituting at least 25% by weight. It is also desirable, for best results, to use primary alcohols.

The long chain aliphatic acid alkylolamine ester and/or amide which we employ in our novel composition is obtained by reaction of an alkylolamine, such as triethanolamine, diethanolamine,

triisopropanolamine or 2-amino-2-methyl-1-propanol with a higher fatty acid, more particularly a mixture of straight chain aliphatic acids comprising essentially lauric acid and having an average of about 12 to 13 carbon atoms in the alkyl chain, exclusive of the carboxyl group present. The carbon chain length usually varies from about 8 to 18 carbon atoms. This aliphatic acid mixture may be obtained as a product of the saponification of commercial coconut oil which yields glycerin and the desired mixture of long chain aliphatic acids, which acids may be separated from the glycerin in a suitable manner and then esterified to yield the desired alkylolamine ester. In forming the aliphatic acid alkylolamine ester, the hydroxy radical of the carboxyl group of the long chain aliphatic acids reacts with one of the hydroxy groups of the triethanolamine, for example, splitting off water and forming the diethanolamine-ethyl ester of the long chain aliphatic acid. The reaction may be effected employing the necessary stoichiometric proportions of the long chain aliphatic acid mixture and triethanolamine at a temperature of about 160° C. and continuing the reaction until the viscosity of the reaction product is reduced to about 320 seconds as determined in the Saybolt Universal viscometer at 100° C. Where the term "viscosity" is employed hereinafter, it is to be understood as being that obtained in the Saybolt Universal viscometer at a temperature of 100° F. The reaction may be effected at temperatures up to about 200° C. employing any alkylolamine and fatty acid or mixture of fatty acids which is not volatile at the temperature employed.

When the above reaction is carried out using a secondary amine such as diethanolamine or a primary amine such as 2-amino-2-methyl propanol or monoethanolamine the corresponding amide is obtained which is also applicable in forming the novel compositions of this invention. When a commercial grade of a tertiary alkylamine is employed, the same usually contain both primary and secondary amino groups which gives rise to both ester and some amide formation.

Most advantageously, however, the esterification and/or amidization reaction described above is effected under a vacuum of about 10 to 14 millimeters of mercury and at a temperature of 140 to 160° C. The lower reaction temperatures possible under these reaction conditions minimizes the formation of color or undesirable by-products such as polymers or oxidation products.

For best results the mineral oil used in our composition should have a Saybolt Universal viscosity of 50 to 100 seconds at 100° F. Highly refined paraffinic based mineral oils are preferable.

Our novel composition preferably comprises at least 12%, more desirably from about 12 to 45% by weight of the partial ester of phosphoric acid and the blended alcohols, 5 to 25% by weight of the long chain aliphatic acid alkylolamine ester, 3 to 25% parts by weight of the tertiary amine, and 30 to 70% of mineral oil.

The lubricants of our invention may be applied to the textile material in the form of an aqueous emulsion or colloidal solution. To this end we may incorporate an emulsifying agent into the lubricant composition. Very satisfactory clear, stable, uniform emulsions or colloidal solutions may be obtained when the emulsifying agent is either a polyoxyethylene ether of a long chain

fatty alcohol or a polyoxyethylene ester of a long chain fatty acid, which may constitute 5 to 25% by weight of the lubricant composition.

The long chain fatty alcohols and long chain fatty acids from which may be prepared the emulsifying agents that are to be used in the stable aqueous emulsion of this invention may contain from about 12 to 18 carbon atoms in their alkyl chains and these chains may be straight or branched. The long chain fatty acids and the long chain fatty alcohols may be commercially pure single compounds or mixtures of such single compounds of one type, i. e. acids or alcohols, or they may be mixtures that are obtained from naturally occurring products. Suitable long chain fatty alcohols are, for example, oleyl alcohol, lauryl alcohol and the mixture of alcohols derived from sperm oil. Suitable long chain fatty acids are, for example, lauric acid, oleic acid and the mixture of acids obtained from coconut oil. In producing the polyethylene oxide ethers and esters, ethylene oxide may be permitted to act directly on the long chain fatty alcohols and long chain fatty acids. Alternatively, the long chain fatty acids and long chain fatty alcohols may be reacted with a polyethylene glycol. In the latter case, care should be taken to remove any unreacted glycol from the final product, as, for example, by washing the same with brine or the like. The length of the polyethylene oxide chain may be varied over a considerable range, satisfactory results having been obtained with chain lengths of from about 2 to 10 ethylene oxide units.

In order to further illustrate our invention, but without being limited thereto, the following examples are given.

Example I

A blend of decanols is made by mixing 158 parts by weight of n-decanol with 316 parts by weight of a blend of equal parts of 2-n-butyl hexanol-1, and 2-ethyl octanol-1.

A dispersion is formed by mixing 616 parts by weight of a mineral oil having a viscosity of 50 S. U. V. @ 100° F. with 142 parts by weight of phosphorus pentoxide. The blend of decanols is added rapidly to this dispersion, with stirring, and the resulting mixture is heated to 80° C. for 30 minutes, cooled and filtered. There is obtained a low viscosity light colored clear liquid.

Example II

46.3 parts by weight of mineral oil having a Saybolt Universal viscosity of 50 seconds at 100° F. are mixed, with stirring, with 26 parts by weight of the liquid product obtained in Example I. To the resulting mixture are added successively, with stirring, 20 parts by weight of diethanolaminoethyl ester of coconut oil fatty acids, 3.2 parts by weight of triethanolamine and 2.5 parts by weight of ditertiary ortho amyl phenol. The resulting smooth homogeneous mixture, which has a pH of about 7.3, constitutes a very effective lubricating and conditioning agent. It shows no cloudiness when cooled to 0° C. or heated to 100° C., even in the presence of 2% water. It has a Saybolt Universal viscosity of 170-200 seconds at 100° F. and very good resistance to change of viscosity in the presence of absorbed water on storage. Its excellent anti-static properties are shown by the fact that application of as little as 1.1% by weight of this lubricating and conditioning agent to cellulose acetate staple fibers is sufficient to com-

pletely prevent development of any static in the carding of such staple fibers.

Example III

To 65 parts by weight of the liquid product obtained in Example I are added with stirring 1 part by weight of ditertiary amyl phenol, 6 parts by weight of "Lipol 30-W" the ester of oleic acid with a polyethylene glycol having an average molecular weight of 300, 21 parts by weight of dibutyl ethanalamine, and 7 parts by weight of diethanolaminoethyl ester of coconut oil fatty acids. This lubricant composition, which has a Saybolt Universal viscosity of 550 seconds at 100° F., shows no cloudiness at 0° C. or 100° C. 100 parts by weight of this lubricant composition are mixed into 400 parts by weight of water. There is obtained a clear transparent emulsion or colloidal solution which is stable and retains its properties substantially unchanged for extended periods of time. This emulsion is particularly desirable for the lubrication of a tow of cellulose acetate filaments that are to be converted into fibers of staple length.

Example IV

A blend of 186 parts by weight of 2-butyl octanol-1 and 130 parts by weight of octanol-2 are added rapidly, with stirring, to a dispersion of 94.6 parts by weight of phosphorus pentoxide in 410.6 parts by weight of mineral oil, having a Saybolt Universal viscosity at 100° F. of 50 seconds. The resulting mixture is heated to 80° C. for 30 minutes, cooled and filtered. 300 parts by weight of the filtered mixture are mixed with stirring with 463 additional parts by weight of the mineral oil. To the resulting mixture are added successively, with stirring, 200 parts of the diethanolamino ethyl ester of coconut oil fatty acids, 25 parts of ditertiary amyl phenol and 35 parts of triethanolamine. The resulting smooth homogeneous mixture is clear and stable at 0° C. and 100° C., even in the presence of 2% water, has very good resistance to change of viscosity in the presence of absorbed water on storage and has excellent anti-static and conditioning properties on cellulose acetate staple fibers.

Our novel composition may be applied directly to continuous filaments of cellulose acetate or other organic derivative of cellulose to condition the same for all textile operations and more particularly, for spinning operations after said lubricated and conditioned continuous filaments have been cut to staple fiber lengths. The lubricating and conditioning agent of our invention is particularly advantageous in that it may easily be applied to said continuous filaments at the metier or spinning cabinet where said filaments are formed. In addition, said composition imparts a very desirable hand to treated cellulose acetate or other organic acid ester of cellulose staple fiber, whether partly saponified or unsaponified, prevents card loading and lickerin loading in processing said treated staple and imparts very satisfactory spinning characteristics thereto, particularly when said treated cellulose acetate or other organic derivative of cellulose staple fiber is spun on the cotton system. Furthermore, the composition is anti-static, non-corrosive and its lubricating and conditioning action is unaffected and unchanged over widely differing conditions of temperature and humidity.

Examples of other organic derivative of cellulose materials which may be lubricated and con-

ditioned with said composition are cellulose esters such as cellulose propionate, cellulose butyrate, cellulose acetate-propionate and cellulose acetatebutyrate and cellulose ethers, such as ethyl cellulose and benzyl cellulose. Our novel composition may also be employed for lubricating and conditioning other textile materials.

It is to be understood that the foregoing detailed description is given merely 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:

A lubricating and conditioning composition for textile materials comprising 12 to 45% by weight of a partial mixed alkyl phosphate produced by

esterifying a mixture of n-decanol, 2-n-butyl hexanol-1 and 2-ethyl octanol-1 in which the total weight of any one alcohol does not exceed 75% of the total weight of all the alcohols, and the average number of alkyl radicals per phosphorus atom is $1\frac{1}{2}$, 30 to 70% by weight of mineral oil, 3 to 25% by weight of a tertiary amine selected from the group consisting of triethanolamine, dibutyl ethanolamine, and dimethyl cyclohexylamine, 1 to 5% by weight of diamyl phenol, and 3 to 25% by weight of the diethanolamino-ethyl ester of coconut oil fatty acid.

References Cited in the file of this patent

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Number	Name	Date
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