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CONDITIONING AGENT

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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, to render the same more amenable to textile operations such as carding, drafting, spinning, twisting, coning, plying, 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 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 said materials are subjected to 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 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 to render the same more amenable to the various textile operations generally employed in converting 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 lubricant composition as they emerge from the spinning cabinet or metier in which they are formed

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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.

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 to avoid the generation of excessive static electricity which would render processing exceedingly difficult if not impossible. The lubricating and conditioning compositions are applied separately because they are incompatible and, if said compositions are mixed prior to application, they do not produce the desired lubricating and conditioning action. The separate application of each of said agents is not only uneconomical but is also quite inconvenient. The development of a single composition which may be applied, for example, to continuous filaments of cellulose or other organic derivatives of cellulose as they emerge from the spinning cabinet or metier where they are formed and which will produce the desired lubricating and conditioning action on cellulose acetate or other organic derivative of cellulose staple fiber materials has long been sought.

We have now found that staple fibers of cellulose acetate or other organic derivative of cellulose materials may be satisfactorily lubricated and conditioned for spinning operations by the application thereto of a single-application lubricating and conditioning composition which may be applied while said materials are in continuous filament form prior to the cutting operation whereby said filaments are severed into staple fiber lengths.

The single-application conditioning agent of our invention comprises a salt-free, dehydrated mixture of a plurality of components prepared individually and then blended into mineral oil. Our novel composition includes a long chain aliphatic alcohol, an alkyl phenol, an alkylamine, a sulfonated long chain aliphatic acid, a sulfonated vegetable oil, the alkylol-amino-ester of a

long chain aliphatic acid and a neutralized partial ester of phosphoric acid with a long chain aliphatic alcohol. Our novel composition, however, is not formed by merely effecting a mechanical mixing of the several components included therein; the composition is obtained by the blending in suitable proportions of several separate reaction products which, when taken together in their final blended form, yield the improved single-application, lubricating and conditioning agent of our invention.

Thus, the components of said lubricating and conditioning composition which contain sulfate groups are obtained by reacting a mixture of mineral oil, a vegetable oil such as olive oil, rice oil or peanut oil, and a long chain aliphatic acid, such as oleic acid, with fuming sulfuric acid such as 20% oleum at a temperature no greater than about 20° C. Under these conditions at least part of the aliphatic acid and the vegetable oil employed are sulfated and/or sulfonated. At the completion of the sulfation and/or sulfonation reaction an alkylated phenol, such as diamyl phenol, is added to the reaction mixture to stabilize the same and to act as a penetrant and an anti-oxidant. The addition of the alkyl phenol is followed by the addition of an alkylamine, water and a sufficient amount of an alkali-metal hydroxide to neutralize all of the sulfuric acid remaining and the major portion of the free aliphatic acid. The bath is then stirred until reaction ceases and a clear oil is formed. The process for obtaining this sulfated and/or sulfonated product is more particularly described in U. S. Patent No. 2,406,408.

In accordance with the present invention the sulfated and/or sulfonated reaction product obtained in the manner described above is not employed in the form in which it is initially obtained but is subjected to a de-salting and dehydration treatment before it is blended with the other components. The de-salting and dehydration is effected by diluting the neutralized reaction product with an organic solvent which forms an azeotrope with water, such as anhydrous isopropyl alcohol, which is a non-solvent for the inorganic salts present. The alcohol addition causes the salts to be precipitated from solution. The precipitated salts are filtered out and the isopropyl alcohol distilled off, the water which is present in the mixture coming off as an azeotrope with the isopropyl alcohol. Preferably, the temperature of the sulfonated mixture being distilled should not exceed about 110° C. during the distillation operation. If any water remains, an additional quantity of anhydrous isopropyl alcohol is then added and the diluted mixture again distilled to remove the remaining water as well as the alcohol. Prior to distillation, a filter aid such as diatomaceous earth or bentonite, etc. may be added and the solution filtered to improve the clarity and remove any inorganic salts still present. The still residue remaining after distillation comprises the composition employed as the sulfonated component of our novel composition.

The long chain aliphatic acid alkylamine ester which we employ in our novel composition is obtained by reaction of triethanolamine with a mixture of straight chain aliphatic acids comprising essentially lauric acid and having an average of about 11 carbon atoms in the alkyl chain, exclusive of the carboxyl group present. This aliphatic acid mixture may be obtained as a product of the saponification of commercial coconut

oil. The saponification reaction yields glycerin and the desired mixture of long chain aliphatic acids which may be separated from the glycerin in a suitable manner and then esterified to yield the desired alkylamine ester. In forming the alkylamine ester, the hydroxy radical of the carboxyl group of the long chain aliphatic acid reacts with one of the hydroxy groups of the triethanolamine splitting off water and forming the diethanolamino-ethyl ester of the long chain aliphatic acid. The reaction may be effected employing the necessary stoichiometric proportions of the long fatty acid mixture and triethanolamine at a temperature of about 150° C. and, preferably, in an atmosphere of an inert gas such as carbon dioxide or nitrogen.

In forming the desired phosphoric acid partial ester of a long chain aliphatic alcohol, the desired alcohol, such as lauryl alcohol, for example, is reacted with a slight stoichiometric excess of phosphorus pentoxide to form a primary phosphoric acid ester, the reaction temperature being maintained at about 110° C. with stirring until reaction is completed.

When the several reaction products forming part of our novel composition have been obtained in a form suitable for compounding, they are then blended with mineral oil and an additional quantity of an alkylamine, alkyl phenol and long chain aliphatic alcohol to form a smooth, homogeneous composition. Thus, from 55 to 65 parts by weight of mineral oil are placed in a suitable vessel equipped with a stirrer and then 2 to 12 parts by weight of the long chain aliphatic alcohol phosphoric acid partial ester are added. The de-salted, dehydrated sulfonation reaction product is then added in an amount of from 2 to 15 parts by weight, followed by the addition of 10 to 30 parts by weight of the diethanolaminoethyl ester of the long chain mixed aliphatic acids. From 2 to 5 parts by weight of alkylamine, 2 to 10 parts by weight of alkyl phenol and 2 to 10 parts by weight of the long chain aliphatic alcohol are then added and the resulting mixture thoroughly blended by continued stirring until homogeneous. The alkyl phenol and the long chain aliphatic alcohol act to impart a greater spreading and penetrating action to said composition. A diatomaceous earth filter aid such as that sold commercially under the name "Filtercel," may then be added and the mixture clarified by filtration. The clarified mixture is ready for use as a lubricating and conditioning agent.

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

Example I

The de-salted, dehydrated sulfonation mixture of our composition is prepared as follows:

A mixture of 117 parts by weight of acid refined white mineral oil of about 50 Saybolt viscosity at 100° F., 87 parts by weight of oleic acid, and 43 parts by weight of raw peanut oil are cooled to 8 to 10° C. in a suitable vessel provided with a cooling jacket and then 27 parts by weight of 20% oleum are added over the course of one-half hour. The temperature rises but the maximum temperature is held to below 25° C. by cooling. The reaction mixture is then stirred for an additional 45 minutes with the temperature held at 20° C. To the reaction mixture, there are now added 21 parts by weight of diamyl phenol, followed by the addition of 47 parts by weight of triethanolamine, 37 parts by weight of water and

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18 parts by weight of a 22% by weight aqueous solution of sodium hydroxide. The resulting neutralized mixture is cooled to room temperature with stirring.

The sulfonated reaction mixture obtained is then desalted by the addition thereto of anhydrous isopropyl alcohol to precipitate the inorganic salts present and the latter are then removed by filtration. The filtered solution is distilled under vacuum and the isopropyl alcohol and water were taken off as an azeotropic mixture with the still temperature held at a maximum of 110° C. The still residue is diluted with an equal volume of anhydrous isopropyl alcohol, about 0.1 to 0.4% by weight of "Filtercel" are added and the mixture filtered again. The isopropyl alcohol present and any remaining water are removed by repeating the distillation under vacuum at a maximum still temperature of 110° C. The still residue comprises the sulfonated reaction product forming one of the components of our novel lubricating and conditioning agent, the final preparation of which is described below.

Example II

The long chain aliphatic acid diethanolaminoethyl ester is formed as follows:

A mixture of 77 parts by weight of mixed coconut oil fatty acids, comprising essentially lauric acid, and 49 parts by weight of triethanolamine are charged into a reaction vessel. The mixture is heated rapidly to 150° C. and maintained at this temperature with stirring under a blanket of gaseous carbon dioxide for about five hours or until the viscosity of the esterified product reaches about 430 seconds as determined in the Saybolt Universal viscometer at 100° F. The reaction mixture is then cooled immediately with cold water. The product obtained comprises essentially the diethanolamino-ethyl ester of lauric acid.

Example III

The long chain aliphatic alcohol partial phosphoric acid ester is obtained as follows:

To a jacketed reaction vessel provided with a stirrer and a steam coil for heating are added 96 parts by weight of lauryl alcohol and then 24 parts by weight of phosphorus pentoxide are added gradually thereto with stirring over the course of 15 minutes. The temperature rises to about 120° C. The reaction mixture is stirred and circulated for five hours during which time the temperature is held at 105 to 110° C. A smooth, homogeneous reaction product is obtained. The product comprising primary lauryl phosphate is cooled to 50 to 60° C. and is discharged to storage where it solidifies on standing.

Example IV

The single application lubricating and conditioning agent of our invention is obtained by blending the components whose preparation is described in Examples I, II and III into mineral oil in suitable proportion.

Thus, 195 parts by weight of acid refined white mineral oil having a viscosity of 50 Saybolt at 100° F. are introduced into a blending vessel, 29 parts by weight of melted lauryl phosphoric acid obtained as described in Example III are added thereto, followed by the addition of 26 parts by weight of the de-salted, dehydrated sulfonated reaction product obtained in the manner described in Example I. To the mixture obtained are added 65 parts by weight of the diethanolaminoethyl ester of lauric acid prepared as de-

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scribed in Example II followed by 9 parts by weight of triethanolamine, 8 parts by weight of diamyl phenol and 18 parts by weight of lauryl alcohol. The final mixture is stirred for one-half hour, one and one-half parts by weight of "Filtercel" are added and the whole filtered. The final filtered mixture comprises the novel single application lubricating and conditioning agent of our invention. 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 meter or spinning cabinet where said filaments are formed. In addition, said composition imparts a very desirable hand to treated staple fiber, 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. The elimination of lickerin loading appears to be due directly to the presence in our novel composition of the neutralized long chain aliphatic alcohol partial ester of phosphoric acid, which is neutralized, of course, in the final composition by the alkaline agents present. Examples of other organic derivative of cellulose materials which may be lubricated and conditioned with said composition are cellulose esters such as cellulose propionate, cellulose butyrate, cellulose acetate - propionate, cellulose acetate - butyrate 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:

1. A single-application lubricating and conditioning composition for rendering textile materials more amenable to textile operations, comprising essentially a homogeneous mixture of 2 to 15 parts by weight of the de-salted and dehydrated reaction product of a mixture of oleic acid, peanut oil and mineral oil with oleum, containing diamyl phenol and which is neutralized with triethanolamine and an aqueous solution of sodium hydroxide blended into 55 to 65 parts by weight of mineral oil with from 2 to 12 parts by weight of primary lauryl phosphate, from 10 to 30 parts by weight of the diethanolamino-ethyl ester of lauric acid, from 2 to 5 parts by weight of triethanolamine, from 2 to 10 parts by weight of diamyl phenol and 2 to 10 parts by weight of lauryl alcohol.

2. Cellulose textile materials lubricated and conditioned with a composition comprising essentially a homogeneous mixture of 2 to 15 parts by weight of the de-salted and dehydrated reaction product of a mixture of oleic acid, peanut oil and mineral oil with oleum, containing diamyl phenol and which is neutralized with triethanolamine and an aqueous solution of sodium hydroxide, blended into 55 to 65 parts by weight of

mineral oil with from 2 to 12 parts by weight of primary lauryl phosphate, from 10 to 30 parts by weight of the diethanolamino-ethyl ester of lauric acid, from 2 to 5 parts by weight of triethanolamine, from 2 to 10 parts by weight of diamyl phenol and 2 to 10 parts by weight of lauryl alcohol.

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