

UNITED STATES PATENT OFFICE

2,564,768

YARN LUBRICANT

George W. Seymour, Maplewood, and Fred Fortess, Summit, N. J., and Marshall Duke, Cumberland, Md., assignors to Celanese Corporation of America, a corporation of Delaware

No Drawing. Application January 10, 1948,
Serial No. 1,663

4 Claims. (Cl. 252—8.75)

1

This invention relates to the treatment of textile materials and relates more particularly to the lubrication and conditioning of textile materials such as fibers and filaments having a basis of cellulose acetate or other organic derivative of cellulose.

An object of this invention is the provision of an improved conditioning agent or yarn lubricant for the treatment of textile materials 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, pining, hanking, weaving, knitting and the like.

Another object of this invention is the provision of an improved textile conditioning agent for yarns, filaments or fibers having a basis of cellulose acetate or other organic derivative of cellulose material which is stable, resistant to oxidation, and which renders the yarns, filaments or fibers to which it is applied anti-static and readily adapted to be woven or knitted into fabrics by suitable forming operations.

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 yarns, filaments and fibers having a basis of cellulose acetate or other organic derivative of cellulose to render the same more amenable to the several textile operations employed in processing the same into fabrics. These lubricating and conditioning agents reduce the friction of the yarns against guides and the like and against each other and also act to reduce the tendency of said yarns, when moving at very high speeds during winding and twisting, warping and other textile operations, to retain undesirably large charges of static electricity. While the use of many of the compositions previously employed has resulted in a definite improvement in the processing characteristics of said yarns, it has been observed that continued exposure to light and air has a slight tendency to oxidize the prior lubricating compositions present on the outside windings of cones and bobbins of said conditioned yarns. The slight oxidation not only causes a marked increase in running tension during warping but also interferes with the adhesion of casein or gelatin base weaving sizes which are usually applied to said yarn prior to weaving causing the fraying of the yarns during processing.

2

We have now found that the tendency toward oxidation and loss of size adhesion in certain lubricating and conditioning compositions applied to cellulose acetate or other organic derivative of cellulose yarns may be overcome by the use of a novel blended multi-component lubricating and conditioning composition. The novel lubricating and conditioning composition of our invention is formed by adding to a mixture of white mineral oil, castor oil, an alkylated phenol or other wetting agent, a long chain aliphatic acid such as oleic acid and an alkylolamine, which mixture is itself an excellent lubricating and conditioning agent under certain conditions, a suitable amount of the desalted, dehydrated reaction product with oleum of a mixture of a long-chain aliphatic acid, a vegetable oil and mineral oil containing a penetrating agent, which reaction product has been neutralized with an alkali and an alkylolamine prior to salt removal and dehydration. Where the long-chain aliphatic acid employed in the initial mixture is oleic acid, it is preferably purified to be low in linoleic acid. A blending agent such as oleyl alcohol or diglycol laurate may also be present in the mixture to which the salt-free, dehydrated composition is added. The diglycol laurate also exhibits excellent humectant and emulsifying properties and is valuable for controlling the scourability of the composition. The use of an acylated vegetable oil in the first-mentioned mixture has also been found to be somewhat advantageous when lubricating or conditioning organic acid ester of cellulose yarns.

The white mineral oil employed in forming the novel conditioning multi-component composition may be one having a paraffinic or naphthenic base and a viscosity of 50 to 75 seconds. This viscosity and those given hereinafter are Saybolt Universal at 100° F. However, the viscosity of the mineral oil employed is determined by the over-all viscosity desired in the final conditioning composition or, in other words, the limit of viscosity of the mineral oil is set by the over-all viscosity of the conditioning composition which can be handled in application. This over-all viscosity may be on the order of 80 to 200 seconds and preferably about 150 seconds. In the mixture comprising our novel conditioning composition, optimum results are obtained using a mineral oil having a paraffinic base and a viscosity of 50 seconds.

The alkylated phenol employed on the present conditioning composition has a beneficial effect on all of the individual ingredients thereof. This

ingredient increases the lubricating value of the oils, reduces the development of static, increases the mutual solubility of the several constituents, lowers the viscosity of the conditioning composition as a whole and improves the wetting power of the same. While we prefer to employ branched chain diamyl phenols such as di-secondary- or di-tertiary-amyl phenol in our conditioning composition, other alkylated phenols such as a branched chain dibutyl and di-secondary- and di-tertiary-amyl derivatives of cresols and xylenols are also advantageously employed. These branched chain dibutyl and diamyl derivatives may be prepared from any of the individual pure cresols and xylenols or from technical grades of mixed cresols and cresylic acids. The branched chain dibutyl and diamyl derivatives of any of the mono-chlorophenols may also be satisfactorily employed in our novel conditioning composition. The alkylated phenol not only acts as an anti-oxidant but also governs the penetration and spread of the conditioning composition in and on the yarn and stabilizes the same.

These alkylated phenols normally have a compatibility with cellulose derivatives of 1 to 50%, the above diamyl phenols having a compatibility of about 20%. The compatibility of the phenol derivatives and of acylated vegetable oils with cellulose derivatives is determined by preparing films of the cellulose derivative and the material or compound whose compatibility is being tested. The films are prepared from solutions of the cellulose derivative and the phenol or vegetable oil derivative in a suitable volatile solvent, such as acetone, by pouring said solutions on a glass or metal plate. After the solvent has evaporated, the film remaining will be clear if the limit of compatibility has not been exceeded. The maximum percentage by weight of the phenol or vegetable oil derivative present in a clear film is the compatibility.

As stated above, the presence of an organic ester of vegetable oil in the initial conditioning fluid mixture to which the desalted and dehydrated reaction product is added to form the final composition is highly desirable, particularly when the yarn being treated is one having a basis of an organic acid ester of cellulose. Examples of such vegetable oil organic esters are the formyl, acetyl, propionyl and butyryl esters of castor oil or castor oil derivatives, specific examples being acetylated castor oil and butyl acetyl ricinoleate. The acetylated vegetable oils have a greatly increased degree of compatibility with the yarn over the unacylated vegetable oils, thereby enhancing the spreading power and wetting out action of the conditioning fluid or dressing. The acetylated vegetable oils may be employed in amounts of from 1 to 10% by weight of the conditioning fluid mixture.

The emulsifiability of the initial fluid mixture to which the salt-free, dehydrated reaction product is added is also improved by the presence of an amine soap which is preferably formed in the initial fluid mixture itself by adding thereto a hydroxylated amine, such as triethanolamine or mixed isopropanolamines, and a fatty acid such as oleic acid. The amine and the fatty acid are preferably added in stoichiometric proportions to form triethanolamine oleate. The amine soap also contributes to the anti-static properties of the composition, aids the blending of the components and has a wetting action on cellulose acetate or other organic derivative of cellulose

textile materials conditioned with our novel composition.

In forming the initial fluid mixture, the proportions of the several components may be varied somewhat depending on the properties it is desired to emphasize in the treated yarn. Where it is desired to increase the spreading and wetting properties, the amount of alkyl phenol may be increased, where the hand of the yarn is particularly important, the amount of softening agent present should be increased. The mineral oil content of the initial mixture may vary from 50 to 75% by weight.

The de-salted, dehydrated component of our novel lubricating and conditioning composition, which contains sulfate and/or sulfonate groups, is 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 a dibutyl or 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 batch 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. Pat. No. 2,406,408.

In order to form the improved lubricating and conditioning composition of the present invention, the sulfated and/or sulfonated reaction product obtained in the manner described above is subjected to a de-salting and dehydration treatment before it is blended with the initial mixture to form the final lubricating and conditioning composition. The de-salting and dehydration is effected by diluting the neutralized reaction product with an organic solvent such as anhydrous isopropyl alcohol which is a non-solvent for the inorganic salts present and which forms an azeotrope with water. The alcohol addition causes the salts to be precipitated from solution. The precipitated salts are then 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, for example, 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 salt-free, dehydrated component of our novel lubricating and conditioning composition.

To form the lubricating and conditioning composition of our invention, from 1 to 10 parts by weight of the salt-free, dehydrated component prepared as described above are added to 90 to

5

99 parts by weight of the initial fluid mixture and, after thorough mixing, the resulting composition may then be employed for the lubrication and conditioning of cellulose acetate, or other organic derivative of cellulose textile materials. Examples of other organic derivative of cellulose materials are cellulose esters such as, cellulose propionate, cellulose butyrate, cellulose acetate-propionate and cellulose acetate-butyrate as well as cellulose ethers such as ethyl cellulose and benzyl cellulose.

In order further to illustrate our invention, the following examples are given:

Example I

The de-salted, dehydrated component of our novel lubricating and conditioning 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 a period of one-half hour. The temperature rises due to the exothermic nature of the reaction 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 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 are 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 a diatomaceous earth filter aid 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 my novel lubricating and conditioning composition.

Example II

A fluid mixture is prepared by thoroughly mixing the following components in the proportions given:

	Parts by weight
Paraffin base white mineral oil (50 seconds' viscosity) -----	55.6
Castor oil -----	18.5
Oleic acid -----	10.7
Di-tertiary-amyl phenol -----	9.7
Triethanolamine -----	4.98
Oleyl alcohol -----	0.55

and to the mixture obtained are added 5 parts by weight of the salt-free dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The

6

composition has a Saybolt Universal viscosity of about 185 seconds at 100° F.

Example III

A fluid mixture is prepared by thoroughly mixing the following components in the proportions given:

	Parts by weight
10 Paraffin base white mineral oil (50 seconds' viscosity) -----	54.5
Castor oil -----	20
Oleic acid -----	10.25
Butyl acetyl ricinoleate -----	5
15 Di-tertiary-amyl phenol -----	4.5
Triethanolamine -----	4.75
Oleyl alcohol -----	1

20 and to the mixture obtained are added 5 parts by weight of the salt-free, dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The composition has a Saybolt Universal viscosity of about 165 seconds at 100° F.

Example IV

30 A fluid mixture is prepared by thoroughly mixing the following components in the proportions given:

	Parts by weight
35 Paraffin base white mineral oil (50 seconds' viscosity) -----	55.05
Castor oil -----	20.2
Oleic acid -----	10.35
Triethanolamine -----	4.80
Butyl acetyl ricinoleate -----	5.05
40 Di-tertiary-amyl phenol -----	4.55

45 and to the mixture obtained are added 5 parts by weight of the salt-free, dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The composition has a Saybolt Universal viscosity of about 150 seconds at 100 F.

Example V

A fluid mixture is prepared by thoroughly mixing the following components in the proportions given:

	Parts by weight
Paraffin base white mineral oil (50 seconds' viscosity) -----	56
Castor oil -----	21
60 Oleic acid -----	5
Cocanut oil diethanolamide -----	5
Acetylated castor oil -----	5
Di-tertiary-amyl phenol -----	4.5
Triethanolamine -----	1.5
65 Dodecyl amine -----	1
Oleyl alcohol -----	1

70 and to the mixture obtained are added 5 parts by weight of the salt-free, dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The composition has a Saybolt Universal viscosity of about 150 seconds at 100 F.

Example VI

A fluid mixture is prepared by thoroughly mixing the following components in the proportions given:

	Parts by weight
Paraffin base white mineral oil (50 seconds' viscosity) -----	52.38
Castor oil -----	14.29
Triethanolamine -----	4.29
Oleic acid -----	11.90
Butyl acetyl ricinoleate -----	6.67
Di-tertiary-amyl phenol -----	5.71

and to the mixture obtained are added 4.76 parts by weight of the salt-free, dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The composition has a Saybolt Universal viscosity of about 150 seconds at 100° F.

Example VII

The following components are thoroughly mixed in the proportions given:

	Parts by weight
Paraffin base white mineral oil (50 seconds' viscosity) -----	55
Castor oil -----	15
Di-secondary-amyl phenol -----	10
Oleic acid -----	12.5
Triethanolamine -----	4.5

and to the mixture obtained are added 5 parts by weight of the salt-free, dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The composition has a Saybolt Universal viscosity of 152.1 at 100° F.

Example VIII

The following components are thoroughly mixed in the proportions given:

	Parts by weight
Paraffin base white mineral oil (50 seconds' viscosity) -----	55
Castor oil -----	15
Oleic acid -----	12.5
Triethanolamine -----	4.5
Butyl acetyl ricinoleate -----	7
Di-secondary-amyl-phenol -----	6

and to the mixture obtained are added 5 parts by weight of the salt-free, dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The composition has a Saybolt Universal viscosity of 146 at 100° F.

Example IX

The following components are thoroughly mixed in the proportions given:

	Parts by weight
Paraffin base white mineral oil (50 seconds' viscosity) -----	55
Castor oil -----	15
Oleic acid (not above 1% linoleic acid content) -----	12.5
Triethanolamine -----	4.5
Butyl acetyl ricinoleate -----	7
Di-tertiary-amyl phenol -----	6

and to the mixture obtained are added 5 parts by weight of the salt-free, dehydrated component obtained in accordance with Example I. After further mixing, the resulting lubricating and conditioning composition is ready for use. The com-

position has a Saybolt Universal viscosity of 149.4 at 100° F.

When applied to yarns, filaments or fibers our novel lubricating and conditioning composition is found to be highly resistant to oxidation, polymerization and gumming even after remaining on the yarns, filaments or fibers for extended periods prior to the utilization of said lubricated and conditioned yarns, filaments or fibers for weaving and knitting operations or for yarn formation. Size adherence is found to be excellent and fraying on the looms due to poor size adhesion is eliminated. In addition, cellulose acetate or other organic derivative of cellulose yarns lubricated and conditioned with the novel composition of our invention have substantially improved anti-static properties.

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 lubricating and conditioning composition for the treatment of textile materials to render the same more amenable to textile operations, comprising a mixture of about 55 parts by weight of a mineral oil, about 5 to about 12.5 parts by weight of castor oil, 1.5 to about 5 parts by weight of an alkylolamine, about 5 to about 10 parts by weight of a long chain aliphatic acid, about 5 parts by weight of an alkyl phenol and a blending agent selected from the group consisting of oleyl alcohol and diglycol laurate, said mixture containing 0.01 to about 0.1 part by weight of said mixture of the de-salted and dehydrated reaction product of oleum with a mixture of a long chain aliphatic acid, a vegetable oil and mineral oil neutralized with an alkali and an alkylolamine.

2. A lubricating and conditioning composition for the treatment of textile materials to render the same more amenable to textile operations, comprising a mixture of about 55 parts by weight of a mineral oil, about 5 to about 12.5 parts by weight of castor oil, 1.5 to about 5 parts by weight of triethanolamine, about 5 to about 10 parts by weight of oleic acid, about 5 parts by weight of a diamyl phenol and a blending agent selected from the group consisting of oleyl alcohol and diglycol laurate, said mixture containing 0.01 to about 0.1 part by weight of said mixture of the de-salted and dehydrated reaction product of oleum with a mixture of a long chain aliphatic acid, a vegetable oil and mineral oil neutralized with an alkali and an alkylolamine.

3. A lubricating and conditioning composition for the treatment of textile materials to render the same more amenable to textile operations, comprising a mixture of about 55 parts by weight of a mineral oil about 5 to about 12.5 parts by weight of castor oil, 1.5 to about 5 parts by weight of an alkylolamine, about 5 to about 10 parts by weight of a long chain aliphatic acid, about 5 parts by weight of an alkyl phenol and a blending agent selected from the group consisting of oleyl alcohol and diglycol laurate, said mixture containing 0.01 to about 0.1 part by weight of said mixture of the de-salted and dehydrated reaction product of oleum with a mixture of oleic acid, peanut oil and mineral oil neutralized with triethanolamine and an aqueous solution of sodium hydroxide and having a diamyl phenol incorporated therein.

4. A lubricating and conditioning composition for the treatment of textile materials to render the same more amenable to textile operations, comprising a mixture of about 55 parts by weight of a mineral oil, about 5 to about 12.5 parts by weight of castor oil, 1.5 to about 5 parts by weight of triethanolamine, about 5 to about 10 parts by weight of oleic acid, about 5 parts by weight of a diamyl phenol and a blending agent selected from the group consisting of oleyl alcohol and diglycol laurate, said mixture containing 0.01 to about 0.1 part by weight of said mixture of the de-salted and dehydrated reaction product of oleum with a mixture of oleic acid, peanut oil and mineral oil neutralized with triethanolamine and an aqueous solution of sodium hydroxide and having a diamyl phenol incorporated therein.

GEORGE W. SEYMOUR.

FRED FORTESS.

MARSHALL DUKE.

20

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,771,347	Prissell	July 22, 1930
1,871,927	Sullivan	Aug. 16, 1932
2,122,593	Stafford	July 5, 1938
2,328,600	Baggett	Sept. 7, 1943
2,328,931	Steik	Sept. 7, 1943
2,396,718	Moscowitz	Mar. 19, 1946
2,406,408	Seymour et al.	Aug. 27, 1946
2,479,644	Seymour et al.	Aug. 23, 1949