Patented Feb. 7, 1950 2,496,631

UNITED STATES PATENT OFFICE

2,496,631

WOOL TREATMENT

Henry F. Leupold, Nutley, and Maurice J. Kelley, Bloomfield, N. J., assignors to Nopco Chemical Company, Harrison, N. J., a corporation of New Jersey

No Drawing. Application July 8, 1946, Serial No. 682,162

15 Claims. (Cl. 252—8.8)

1

The present invention relates to a composition of matter and a process for the treatment of wool or wool mixtures. More particularly it relates to the lubrication, fulling, and scouring operations in the manufacture of cloth from wool fibers alone or wool fibers mixed with other natural or synthetic staple fibers.

A conventional process for the manufacture of woolen cloth includes shearing the sheep, Angora goats and other wool producing animals, washing the fleece to remove grease and dirt, drying and sorting the fibers by color and quality. Then the selected fibers are mixed alone or, if desired, blended with other materials such as cotton, silk, nylon, rayon or other natural or synthetic fibers. Following this, the selected fibers may be treated with olive, teaseed, rice bran, or like fluid oils by sprinking or immersion in a dispersion of the oil in water. The oil serves to lubricate the fibers during the spinning and carding processes. The oiled fibers are next combed or carded to arrange them in parallel formation and also to remove any foreign matter. Thereafter they are pulled or drawn into loose strands having the fibers still parallel to one another. In the spinning operation, the fibers are twisted into yarn in one or more steps, depending upon the desired degree of twist. Customarily a steam treatment sets the twist in the yarn. The spun yarn is woven into cloth on looms, and this treatment is followed by an optional crabbing process wherein the cloth is tumbled in a water bath. At this time the woven fabric is of comparatively loose and thin structure, so fulling is performed to shrink and thicken the cloth thereby reducing the size of the interstices therein. Fulling is accomplished by working the fabric in a soap solution in a mill designed for the purpose. Then the fulled material passes into another detergent bath where it is scoured or washed to remove the lubricant applied to it prior to carding. In the next operation the material is dyed and then dried. To finish the cloth, it is usually steamed or in some cases treated with a water repellant agent.

In the prior art it was conventional to apply a lubricant at one stage in the manufacture of cloth, a fulling detergent at another stage and a final scouring detergent at still another stage. Difficulty was frequently encountered in the carding process due to the accumulation of charges of static electricity on the fibers. Oxidizable oils tended to discolor the yarn or to become set in the yarn upon oxidizing during steaming or crabbing. Whenever any appreciable quantity of

2

the lubricating oil remained in the cloth, the fabric was rather sticky. Uneven dyeing often resulted from excessive soap residues being left in the fabric after the final scouring step. Moreover, the wet finishing included a harsh steaming or water treatment which was necessary to restore the soft handle of the cloth.

An object of the invention is to provide an improved treatment and treating composition for wool fibers or mixtures thereof with other fibers.

A second object of the invention is the preparation of a wool lubricant which is self-scouring, self-fulling, prevents or reduces the accumulation of static electricity and readily forms an emulsion with water.

A third object of the invention is the preparation of a lubricating oil for wool or wool mixtures which may be more easily and more completely removed from the fibers.

A fourth object of the invention is to provide a faster and more economical process for the production of woolen goods.

A fifth object of the invention is to reduce the number of agents required for the treatment of woolen fibers during the manufacture of cloth.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The present invention concerns the treatment of wool fibers alone or wool fibers blended with other staple textile fibers with a composition which possesses lubricating, antistatic and detergent properties. Broadly the composition includes a mixture of a nonresinifying oil, an ester and a condensation product of selected fatty acids with certain alkylolamines, the molar ratio of the latter to the former being at least 1.5 to 1.0 and preferably at least 2.0 to 1.0. This agent performs like an oil on the fibers during carding and spinning operations and like a soap in the subsequent fulling and scouring operations.

The invention accordingly comprises the several steps and relation of one or more of such steps with respect to each of the others, and a composition of matter possessing the characteristics, properties and relation of components; all of which will be exemplified in the following detailed disclosure, and the scope of the invention will be indicated in the claims.

Suitable agents in accordance with the present invention contain about 15 to 50% by weight of the reaction product of 1 equivalent weight of a fatty acylating substance having an acyl chain of at least 8 carbon atoms with at least 1.5 moles of an alkylolamine selected from the group consisting of diethanolamine ethyl monoethanolamine,

diisopropanolamine, and 2-amino 2-methyl propanol-1, diethanolamine being highly preferred. Of the fatty compounds, the mixture of fatty acids as obtained from coconut oil is highly preferred, but caprylic, capric, lauric, myristic, palmitic, stearic, decylenic, dodecylenic, palmitoleic, oleic, ricinoleic and like acids, their acyl halides, alkyl esters and glycerides, are also operative. While the fatty acid and alkylolamine will react in equimolecular proportions, the resulting condensation product is inferior to those employed in the present invention wherein substantial proportions of the condensation product result from the reaction of two or more molecules of the alkylolamine with a single molecule of the fatty acid. Inasmuch as the structure of the radial formed by the alkylolamine molecules apart from the basic amide linkage thereof with the fatty radical in the reaction product has not been established, the reaction product as a whole is termed herein merely "condensation product" for brevity and is used to denote the product or products of the chemical reaction in which a fatty acylating substance combines with an alkylolamine accompanied by a splitting off of water alone or with a hydrohalide or aliphatic alcohol. In the case of a glyceride reactant, the aliphatic alcohol will be glycerine or a glyceryl partial ester. The condensation product may be produced directly from fatty acylating substances by reacting them with the designated alkylolamines in the proportions indicated herein. When using glycerides, the fact that one mole contains more than one equivalent weight of fatty acids must be taken into consideration.

In addition to the condensation product, a nonresinifying oil constitutes another 25 to 80% by weight of the treating agent. Any oil may be used with the exception of drying oils which harden by oxidation or polymerization to resinous films. The expression "nonresinifying oil" is used in a sense including all mineral oils and raw, blown or hydrogenated oils of the non-drying and semi-drying types derived from vegetable, animal and marine sources but excluding all drying oils. While semi-drying oils introduce a quality of tackiness in the treating composition that is prized by many woolen manufacturers, drying oils do not provide satisfactory lubricating qualities in the agent, since they tend to form gums. Mineral oils of viscosities ranging from 45 to 200 Saybolt seconds at 100° F. are preferred, especially where the treating agent contains no glyceride oils. The nonresinifying oil may be a single oil or a mixture of various oils of the classes stated. A few of the many suitable oils are olive, rape seed, rice bran, teaseed, soybean, peanut, corn, neat's-foot, sperm, cottonseed, castor, lard, white mineral (55 Saybolt sec. at 100° F.) and yellow paraffin (100 Saybolt sec. at 100° F.) oils. Of such oils, blown vegetable oils are preferred, and mineral oils are useful as inexpensive diluents for the blown fatty oils. Partially saponified oils such as peanut oil may also be employed.

An ester of a low titer fat or cycloaliphatic compound is incorporated in the treating composition in a quantity of about 10 to 40% of the total weight. The low titer fat should have a melting point below 25° C., and the fatty ester should be liquid at all temperatures encountered in actual woolen manufacturing operations. Suitable esters include, inter alia, the methyl, ethyl, propyl, isopropyl, butyl, isobutyl, amyl, etc. esters of stearic, oleic, palmitic, lauric, caprylic, capric, ricinoleic, naphthenic, etc, acids, as well as octyl

formate, decyl acetate, lauryl acetate, myristyl propionate, palmityl acetate, oleyl butyrate, stearyl acetate, naphthenyl propionate, ricinoleyl formate, etc. Esters of polyhydric alcohols, as exemplified by diethylene glycol monolaurate, may also be used.

Although 15 to 50% condensation product, 25 to 75% nonresinifying oil and 10 to 40% ester are suitable proportions by weight for accomplishing the purposes of this invention, considerably better results are secured when the ranges are narrowed down to 20 to 50%, 25 to 40% and 25 to 40% respectively.

The textile treating compositions in the examples below may be compounded by merely mixing the listed ingredients at room temperature, but the resulting product usually appears as a cloudy suspension when dispersed in water. Therefore, it is preferable to heat the mixed constituents to temperatures ranging from 60 to 80° C. for 2 to 3 hours to produce a composition capable of forming a true, transparent or translucent emulsion in water. The nature of the change occasioned by the heating is unknown.

The composition may be applied in the form of an aqueous emulsion or in solution in hydrocarbon or chlorinated hydrocarbon solvents by spraying the emulsion or solution or immersing the wool therein, or the wool may be coated directly with the agent by means of rollers, wicks or the like. To realize all advantages of the new agent it should be applied prior to carding, and preferably as an aqueous emulsion, since the composition requires no added emulsifiers to form a transparent or translucent emulsion in water. In preparing this emulsion, water should be heated to a temperature of from 100 to 150° F. and the composition of the present invention then stirred in gradually until it is thoroughly emulsified. Under no circumstances should this emulsion be boiled at any time as this will break the emulsion. The concentration of the emulsion may be varied widely but should be such that approximately from 1 to 5 parts by weight of the treating agent will be deposited on each 100 parts of dry, scoured fibers. About 2.5 to 3.0 parts per 100 parts of raw stock is recommended. Where it is desired to utilize the agent only as a lubricant and to prevent static in the combing and drawing operations, less than 1 part per 100 parts of the raw fibers may be employed. These quantities are much lower than experience has shown to be desirable in the case of conventional wool oils.

As will be described hereinbelow, numerous advantages accrue to the new composition in addition to its emulsifying qualities and ability to perform as both a lubricant and soap.

No additional equipment is involved and it is unnecessary for the mills to change their procedure except for omitting the harsh water or steam finishing treatment usually employed for restoring a soft handle to the cloth. The agent contains no sulfonates, salts or ash; hence it does not corrode metals. In the case of a sulfonate, there is always the danger of the compound breaking down to form sulfuric acid. The present composition is stable in both alkaline and mild acid solutions and is compatible with all phases of woolen manufacturing.

In utilizing the new treatment of the invention, superior carding results have been obtained due to the excellent lubricating and static-eliminating properties of the agent. It is believed that due to its ionic character, the condensation

product conducts all static electricity to grounded members of the equipment as fast as the static charges are produced. The carding seems to take place nearer the tip of the wire or clothing, and the elimination of static has greatly reduced the fly. Further, the fibres are laid more nearly parallel and there is less loading up of the cards. Since there is less stripper waste, it is possible to speed up the cards with appreciable reduction of costs in this operation. In some instances, it has been found that it is no longer necessary to strip the cards at all in contrast with the former practice of stripping every 15 to 18 hours. The improved composition has no tendency to oxidize and thus produce gumminess; hence it is much superior in this regard to the conventional wool lubricants. Instead of glazing the leather of the tape condensers and rubroll aprons, the new agent softens them and a loftier and rounder roving thereby results. This softening and preservation of the aprons greatly reduces the shutdowns for stripping or oiling the leather aprons. Since the roving retains the original loft and resiliency of the wool fibers, the final yarn has greater tensile strength and is free of "bearding" or fuzziness.

In spinning, the present composition performs equally well as a lubricant on either mule spinners or frame spinners. Due to the improved carding, an improvement in spinning naturally occurs, as this operation depends to a large extent on the quality of the carding. It has been found that far fewer broken ends occur when the treatment of the present invention is employed. Fadometer and Sudam violet tests have proven that this new agent is far less susceptible to oxidation than other lubricants which have given a great deal of trouble by producing a sticky and discolored yarn. Moreover, the improved composition does not become set in the yarn as do conventional oils where filling bobbins require steaming. Again, it does not become set in fabric when greige goods are crabbed, as is the case with mineral oils and many types of fatty oils.

In the fulling mill only water is required in carrying out the improved treatment as the new agent itself produces an adequate foam. The addition of a small quantity, as for instance one ounce per gallon of water, of an alkali such as sodium, potassium or ammonium carbonate is optional and appears to produce slightly better results. It is likely that these alkalies merely serve to soften the water. The amount or type of alkali added is in no sense critical, therefore any quantity can be dissolved so long as the pH is kept below values deleterious to woolens. However, no soap or detergent whatsoever is needed. The woven goods should be wet out in the usual manner to obtain the same degree of wetness as when soap or detergents are used. It will be generally found that one-third more water will be required to reach the same apparent degree of wetness. The treating composition already impregnated in the woven goods will start immediately to foam, and the operator can easily estimate when the same degree of wetness or slickness is reached by feeling the goods from time to time as water is added. Shrinkage, both filling-wise and warp-wise, will take place at the same time.

The material leaves the stuffing box and folds in the same manner as when soaps are used. It is scoured in the washer without the addition of any soap, detergents or further alkali. The agent and alkali already in the fabric are carried over to the washer in the usual way; and conventional temperatures and methods of sudsing and rinsing are employed. Another advantage of the new composition is that pitch and tar are removed from the fibers far more readily than with the ordinary soaps or detergents. In utilizing the present invention it has been found that the alkali and treating composition are quickly dispersed and removed in rinsing with resulting time savings of from 15 to 60 minutes in the final cold water rinse. In the past it was conceded that all detergents used in fulling and scouring were never completely washed out, and ether extractions showing a residual oil content of not more than 0.5 per cent were accepted simply because no lower figure had been obtainable. In carrying out the improved process, residual oil contents of around 0.2 to 0.3 per cent or lower are common, even with a much shorter rinsing time.

Where the goods are piece dyed, the present treatment insures much better results. Not only does the material go to the dye kettles with a materially smaller quantiy of residual oil, but that oil is far more evenly distributed throughout the cloth; these factors are conducive to levelness of dyeing. Moreover, it has been found that the residual oil of the new composition serves as a dyeing assistant rather than an obstacle to dyeing, and that it is more compatible with fugitive tints than any known wool oil.

Woolen cloth manufactured in accordance with this invention is stronger and has a distinctly softer handle. This is due to the retention of the original loft and resiliency of the raw fibers resulting from the improved fiber lubrication, the elimination of excessively alkaline fulling and scouring baths, and the elimination of harsh steaming treatments customarily employed to soften the handle of the fabric.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following examples which are given merely to further illustrate the invention and are not to be construed in a limiting sense all parts given being by weight.

Example I

	Parts
Condensation product (coconut acids-diethanolamine)	40
Methyl oleate	30
Blown peanut oil	30
	100

The condensation product was formed by reacting 2 moles of diethanolamine with 1 mole of cocoanut fatty acids at a temperature of 170° C. with agitation for one hour during which period water was evolved thus forming a coconut fatty amide.

In a laboratory test for detergent effect, 100 parts of flannel were impregnated with 3.8 parts of the composition of this example dispersed in an aqueous emulsion. The cloth was later washed with agitation in water only for 30 minutes at 140° F., and then dried. A thorough extraction with ether showed the residual matter on the cloth to amount to only 0.3 parts. This test affords a good basis for judging the performance of an agent in fulling and scouring operations.

In mill practice, ether extractions of 0.2% of the total fiber weight and lower have been obtained when 100 parts of the raw fibers were treated with 3 parts of the same composition.

The composition of this example constitutes the preferred embodiment of the invention.

Example II

	Parts
Condensation product (oleic acid-diethanolamine)	30
Isobutyl oleate	11
Blown soybean oil	15
Paraffin oil (pale yellow—100 Saybolt sec. at 100° F.)	44
	100

In producing this condensation product, 2 moles of diethanolamine were condensed with one mole of oleic acid at 170° C. for one hour with the evolution of water thus forming an oleic fatty amide.

Example III

	Parts
Condensation product (coconut acids-diisopropanolamine)	30
Isobutyl oleate	11
Blown soybean oil	15
Paraffin oil (pale yellow—100 Saybolt sec. at 100° F.)	44
	100

One mole of the coconut fatty acids was reacted with two moles of diisopropanolamine at 190° C. for 30 minutes. Water distilled off during the reaction whereby a coconut fatty amide was formed.

Example IV

	Parts
Condensation product of Example I	40
Diethylene and glycol monolaurate	20
Blown soybean oil	20
Paraffin oil (pale yellow—100 Saybolt sec. at 100° F.)	20
	100

The same conditions as in Example I were employed in the laboratory detergency test. 3.6 parts of this composition left a residue of 0.33 part in 100 parts of the flannel.

Example V

	Parts
Condensation product of Example I	15
Isobutyl oleate	32
Blown soybean oil	43
White mineral oil (85 Saybolt sec. at 100° F.)	10
	100

Example VI

	Parts
Condensation product of Example I	16
Diethylene glycol	4
Methyl oleate	40
Paraffin oil (pale yellow—100 Saybolt sec. at 100° F.)	40
	100

Example VII

	Parts
Condensation product of Example I	20
Methyl oleate	20
Blown soybean oil	20
Paraffin oil (pale yellow—100 Saybolt sec. at 100° F.)	40
	100

Example VIII

	Parts
Condensation product of Example I	20
Methyl oleate	15
Blown soybean oil	15
White mineral oil (50 Saybolt sec. at 100° F.)	50
	100

Example IX

	Parts
Condensation product of Example I	37.5
Isobutyl oleate	24.0
Blown soybean oil	31.0
White mineral oil (55 Saybolt sec. at 100° F.)	7.5
	100.0

Example X

	Parts
Condensation product of Example I	40
Methyl oleate	30
Rice bran oil	30
	100

Example XI

	Parts
Condensation product of Example I	40
Methyl oleate	30
Partially saponified blown peanut oil	30
	100

The partially saponified oil was formed by mixing:

	Parts
Blown peanut oil	73.5
Aqueous potassium hydroxide (49% KOH)	6.4
Antioxidant	.1
Diethylene glycol	20.0
	100.0

Experiments indicated that the condensation products of the reaction of one mole of fatty acids with one mole of the alkylolamine were distinctly inferior in woolen lubricant compositions to those in which 1.5, 2, 3 or 4 moles of the alkylolamine were reacted with one of the acids.

Adjustments to improve the emulsifying properties of the compositions of the above examples are occasionally necessary due to variations in the ingredients. Minor quantities of red oil, caustic soda or mutual solvents, such as ethylene glycol, are employed for the purpose.

The expression "woolen textile" is used herein to connote any textile containing wool fibers such as obtained in the shearing of sheep, Angora goats and other wool producing animals.

Since certain changes in carrying out the above process and certain modifications in the composition which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all the generic and specific features of the invention herein described and all statements of the scope of the invention, which, as a matter of language, might be said to fall therebetween: and that they are intended to be inclusive in scope and not exclusive, in that if desired, other materials may be added to our novel composition of matter herein claimed without departing from the spirit of the invention. Particularly it is to be understood that in said

claims, ingredients or components recited in the singular are intended to include compatible mixtures of said ingredients wherever the sense permits.

Having described our invention, what we claim as new and desire to secure by Letters Patent is:

1. A self-fulling and self-scouring wool treating composition which comprises sufficient amounts of a nonresinifying oil and a liquid monoacylated organic fatty ester to soften and lubricate wool fibers and a quantity sufficient to form a stable emulsion of the oil and the ester in water of a condensation product formed by heating in the ratio of one equivalent weight of a fatty acylating substance having an acyl chain of at least 8 carbon atoms with from 1.5 to 4.0 moles of an alkylolamine selected from the group consisting of diethanolamine, ethyl monoethanolamine, diisopropanolamine and 2-amino 2-methyl propanol-1 at a temperature and for a time sufficient to form a fatty amide.

2. A composition according to claim 1 wherein the alkylolamine comprises diethanolamine.

3. A composition according to claim 1 wherein the fatty acylating substance comprises coconut fatty acids.

4. A composition according to claim 1 wherein the nonresinifying oil comprises a blown fatty oil.

5. A composition according to claim 1 wherein the nonresinifying oil comprises a mineral oil having a viscosity of from 45 to 200 Saybolt seconds at 100° F.

6. A composition according to claim 1 which comprises from 25 to 75% by weight of the nonresinifying oil, from 10 to 40% by weight of the fatty ester and from 15 to 50% by weight of the condensation product.

7. A self-fulling and self-scouring wool treating composition which comprises from 25 to 40% by weight of a nonresinifying blown fatty oil, from 25 to 40% by weight of a liquid monoacylated organic fatty ester, and from 20 to 50% by weight of the condensation product formed by heating in the ratio of one equivalent weight of coconut fatty acids with from 1.5 to 4.0 moles of diethanolamine at a temperature and for a time sufficient to form a coconut fatty amide.

8. A self-fulling and self-scouring wool treating composition which comprises approximately 30% by weight of a nonresinifying blown fatty oil, approximately 30% by weight of a liquid monoacylated organic fatty ester, and approximately 40% by weight of the condensation product formed by heating in the ratio of one equivalent weight of coconut fatty acids with substantially 2 moles of diethanolamine at a temperature and for a time sufficient to form a coconut fatty amide.

9. A self-fulling and self-scouring wool treating composition which comprises sufficient amounts of a nonresinifying oil and a liquid monoacylated organic fatty ester to soften and lubricate wool fibers and a quantity sufficient to form a stable emulsion of the oil and the ester in water of a condensation product formed by heating in the ratio of one equivalent weight of a fatty acylating substance having an acyl chain of at least 8 carbon atoms with substantially two moles of an alkylolamine of the group consisting of diethanolamine, ethyl monoethanolamine, diisopropanolamine and 2-amino 2-methyl propanol-1 at a temperature and for a time sufficient to form a fatty amide.

10. A composition according to claim 9 wherein the alkylolamine comprises diethanolamine.

11. A composition according to claim 9 wherein the fatty acylating substance comprises coconut fatty acids.

12. A composition according to claim 9 wherein the nonresinifying oil comprises a blown fatty oil.

13. A composition according to claim 9 wherein the nonresinifying oil comprises a mineral oil having a viscosity of from 45 to 200 Saybolt seconds at 100° F.

14. A composition according to claim 9 which comprises from 25 to 75% by weight of the nonresinifying oil, from 10 to 40% by weight of the fatty ester and from 15 to 50% by weight of the condensation product.

15. A self-fulling and self-scouring wool treating composition which comprises from 25 to 40% by weight of a nonresinifying blown fatty oil, from 25 to 40% by weight of a liquid monoacylated organic fatty ester, and from 20 to 50% by weight of the condensation product formed by heating in the ratio of 1 equivalent weight of coconut fatty acids with from 2 to 4 moles of diethanolamine at a temperature and for a time sufficient to form a fatty amide.

HENRY F. LEUPOLD.
MAURICE J. KELLEY.

REFERENCES CITED

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

UNITED STATES PATENTS

Number	Name	Date
1,985,687	Nuesslein et al.	Dec. 25, 1934
2,096,705	Heckethorn	Oct. 19, 1937
2,333,770	Dickey et al.	Nov. 9, 1943
2,334,852	Weisberg et al.	Nov. 23, 1943