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2,806,804

METHOD OF TREATING WOOL

Lloyd W. Davis, Upper Montclair, and John P. Redston, Montclair, N. J., assignors to E. F. Drew & Co., Inc., New York, N. Y., a corporation of Delaware

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The present invention is directed to a process of fulling of wool under acid conditions to obtain the same desirable hand usually associated with ordinary soap and alkali fulling.

Up to the present time, in commercial practice, woolen piece goods and fibers were fulled and scoured by mechanically working the same in a moist state in an alkaline medium such as a combination of soap and alkali with or without synthetic detergent or in a mixture of synthetic detergent and alkali. The procedure was carried out at a pH above 7, and usually not over 11.2 in such a manner as to cause felting of the fibers to obtain shrinkage of the fabric, with a resulting increase in thickness or density. This was usually followed by washing or scouring, and the resulting fabric had a desirable hand. If the fabric contained undesirable cellulosic material, it was impregnated in a solution of acid material, such as sulphuric acid, aluminum chloride, or other acidic substances, followed by drying at high temperatures to destroy the cellulose by carbonization. Thereafter the acidity was neutralized and the fulling operation in an alkaline medium was conducted.

Such procedure had a number of disadvantages in that a number of operations were necessary to carry out the process so that it was relatively expensive and required considerable time. A typical procedure would be five distinct steps such as, full, scour and rinse, carbonize, crush, neutralize. In this case it is somewhat difficult to remove the burrs and other cellulosic materials as they become imbedded in the fabric by virtue of the fulling or milling operation. Previously it had been somewhat difficult to dye evenly fabric which had been fulled, scoured and carbonized due to the fact that the acid present in the fabric was excessive and not always evenly combined with the wool throughout the piece, so much so that many dyers prefer to carbonize after dyeing.

The present invention is intended and adapted to overcome the difficulties and disadvantages inherent in prior methods, it being among the objects thereof to provide a fulling agent for wool which is stable in acid medium.

It is also among the objects of the present invention to provide an acid stable fiber lubricant or softener which has a cushioning effect, imparts slip to the fiber, does not inhibit the swelling action or plasticizing of the wool during the acid fulling action.

It is further among the objects of the present invention to provide a treatment of wool which permits a more even dyeing than prior alkaline processes and which renders the fabric light stable in damp storage before the dyeing operation.

In practicing the present invention, applicants provide an acid stable fiber lubricant having approximately the following composition:

	Parts
Polyethyleneglycol 325-850 monoester of higher fatty acid	10-20
Ethanolamine (1 to 1) condensation product with higher fatty acid	5-10
Unsaturated glyceryl triester of higher fatty acid	5-10
Paraffin oil	67-75

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The figures 325-850 refer to the molecular weight of the glycol; similarly, throughout the specification and claims, the figures following the glycols indicate the molecular weights thereof.

The higher fatty acids contained in the products usually have from 12 to 22 carbon atoms. The ethanolamine is usually a diethanolamine. The unsaturated higher fatty acids are those normally found in vegetable and animal oils. Usually there is added to the composition from .5 to 1.0% of a higher fatty acid which acts as a neutralizing agent for any excess amine which may be present in the product. Together with the first two named substances, the fatty acid so formed acts as an emulsifying agent. The third and fourth named constituents act as lubricants during the process of picking, carding, spinning and weaving the wool fibers. The whole composition actually nourishes the leather rub aprons and condenser tapes on the fiber carding machine in the process preliminary to spinning.

Usually after this sequence of operations the fabric is carbonized as usual without removal of the acid-stable lubricant. Thereafter the fabric is treated with the following agent, which is an alkoxypolyethyleneglycol 325-850 monoester of higher fatty acid. The higher fatty acid usually has from 12 to 22 carbon atoms. The alkoxy radical has 1 or 2 carbon atoms.

If the acid stable or any other suitable fiber lubricant is removed by dry cleaning or washing prior to carbonizing, a satisfactory acid stable cushioning agent or softener is added to the fulling bath together with the alkoxypolyethyleneglycol 325-850 mono ester of higher fatty acid.

One such type of acid stable cushioning agent or softener which may be used has the following composition:

	Parts
Polyethyleneglycol 450 mono oleate	44
Diethanolamine coconut fatty acid condensation product	22
Mustard seed oil	22
Oleic acid	12

Another acid stable cushioning agent or softener which may be used is the reaction product of higher fatty acid (1 to 1) with aminoethylethanolamine, further reacted with diethyl sulphate. The higher fatty acid has from 12 to 22 carbon atoms. The product may be formed as described in the co-pending application of Lloyd W. Davis, Serial No. 323,707, filed December 2, 1952, entitled "Anti-static Agent for Synthetic Textile Materials", and now abandoned. The reaction product is made, for example, by heating a mixture of oleic acid with aminoethylethanolamine to a temperature of about 205° C. for about two hours. Then the reaction mixture is maintained at the specified temperature until a test indicates that the reaction is complete. Thereafter the intermediate product is treated with diethyl sulphate with agitation and sufficient cooling to prevent the temperature from rising above 100° C. The product is soluble in water.

In the present process only three steps are required, namely carbonizing, crushing and fulling, scouring and rinsing. Furthermore, the burrs are easily carbonized in the open state of the fabric and removed in the fulling and crushing, followed by scouring and rinsing.

In applicant's process the even distribution of the acid throughout the piece is facilitated by the surface active agent present in the fulling solution. The removal of loosely combined acid in the subsequent scouring and rinsing operation represents a perfect ground containing an even distribution of acid combined with the wool and possessing affinity for all dyes usually used on wool. This shows up at the very commencement of the dyeing operation where it is observed that the primary component dyestuffs all start to exhaust on to the wool to-

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gether. This is the ideal condition for any level dyeing operation. In normal acid dyeing where the dye is added before the acid, some possible unevenness is encountered because some of the dye starts to exhaust without the presence of acid, whereas the components that require acid, do not start to exhaust until the acid is added. Also it is some time before the acid and dye have time to exhaust themselves on the wool by the not entirely perfect circulation present in ordinary piece dyeing machines. The advantages of presenting to the dyestuff a piece of woolen fabric absolutely evenly impregnated with acid is therefore of primary importance in obtaining a level dyeing. The complete absence of soap or of any soap-like product in the fulling and scouring operation is a fact that will be readily appreciated by all those versed in the art who are familiar with uneven dyeing results brought about by the presence of traces of residual soap left in the woolen fabric prior to acid dyeing.

The following are specific examples of the operation of the present invention.

Example 1

There is provided a fiber lubricant which is stable to acid, having the following composition:

Polyethyleneglycol 400 mono-oleate.....	parts--	15.0
Diethanolamine-coconut fatty acid condensation product	parts--	7.5
Peanut oil.....	do....	7.5
Pale paraffin oil (100 Saybolt at 100° F.)....	do....	70.0
Oleic acid	percent..	0.5-1.0

It is made up into an emulsion containing from 10% to 50% of the lubricant in water. An amount of the emulsion varying from 1% to 10% based on the weight of fibers is applied thereto either by hand or by atomization. Thereafter the fibers are processed as usual through the operation of picking, carding, spinning and weaving.

Then in order to carbonize the resultant fabric it is passed through a sulphuric acid solution containing from 2% to 10% of the acid dissolved in water. The fabric is dried at 180° F. and then baked at 260° to 270° F. to carbonize cellulosic matter.

The fulling agent is methoxy polyethylene glycol 350 monolaurate, which is dissolved in water at the rate of 2 to 8 oz. per gallon. The fabric is run through an impregnating device so that it takes up from 50% to 125% of its weight of the solution. If desired, the solution may be applied in said amounts to the dry fabric in the fulling mill. The fulling takes place at normal temperatures without any added heat, although the temperature may rise to 100° F. or higher during the operation.

The fulling having been completed the fabric is rinsed in water, leaving the same in acid condition as the fabric still carries some acid from the carbonizing bath. The scouring and rinsing may be accomplished in the ordinary scouring machine by adding water cold to 130° F. to the fabric in a short liquor ratio and running for 10 to 20 minutes, followed by rinsing with fresh water in the usual manner.

In the case where the wool has been dyed at some stage before the weaving, the rinsing is done in the presence of an alkaline material. Usually sodium carbonate or ammonium hydroxide is used. This counteracts any change in the shade of the color on the wool brought about by the acid in the carbonizing bath. Rinsing in the presence of an alkaline material applies also when the type of dyes to be used require pieces to be neutral or alkaline at the start of the dyeing operation.

Example 2

In some cases the fiber lubricant in the fabric is removed by dry cleaning prior to the fulling operation. In such case there is added to the fulling bath an acid

stable softener having a cushioning action. The following composition is used:

	Parts
Polyethyleneglycol 400 mono-oleate.....	44
Diethanolamine coconut fatty acid condensation product	22
Mustard seed oil.....	22
Oleic acid.....	12

The operation is conducted as set forth above in the fulling mill or other suitable apparatus.

Example 3

As an alternative to the composition used in Example 2 as a softening and cushioning agent, there may be used a reaction product of 1 mol. of stearic acid with 1 mol. of aminoethylethanolamine and the condensation product subsequently reacted with diethyl sulphate.

There are numerous advantages inherent in the present process. The acid used in the carbonizing is utilized to provide favorable acidic conditions to promote the fulling action and is further utilized in the later dyeing operation. The acid-stable lubricant assists the fulling action and the acid itself has a strengthening effect on the wool fiber. The residual acid, after the surplus has been rinsed out with water, is actually combined evenly with the wool fibers and produces a very satisfactory condition in which the fabric absorbs the dye uniformly. The fabric is more light stable during storage, in a damp condition while awaiting dyeing than in the usual alkaline condition, and thus a more even dyeing is possible.

Most of the detergents and emulsifiers which are acid stable are not satisfactory for applicant's purpose because they do not provide the necessary detergent properties in combination with the cushioning effect necessary in fulling operations. In the present invention the compositions and procedures used have these properties and thus an excellent result is obtained.

Although the invention has been described setting forth several specific embodiments thereof, it will be apparent to those skilled in the art that various changes in the details of the invention may be made within the spirit hereof. For instance, the several steps in the operation may be reversed, some of the steps may be omitted and other steps may be added. The compositions used in the several examples were illustrative only as the proportions of the constituents may be varied in substantial degree providing the mixture used remain homogeneous.

The unsaturated oils which may be of animal or vegetable origin remain liquid throughout the operation and insure that the compositions will remain homogeneous. The diethanolamine higher fatty acid condensation products are formed by the condensation at elevated temperatures of equimolecular proportions of the constituents. Such reagents are described in a number of patents in the name of Wolf Kritchevsky, for instance, No. 2,173,909. In the treatment of worsted fabric, the procedure is the same as in Example 1 but the emulsion may be applied to the fibers after the backwashing operation or at the first or second pin drafting operation, or at any other suitable stage.

What is claimed is:

1. A method which comprises treating wool fibers by impregnating said fibers with a mixture which is substantially free from soaps and which consists of an aqueous emulsion of about 10-20 parts of polyethylene glycol 325-850 monoester of a fatty acid having 12-22 carbon atoms, about 5-10 parts of the liquid glyceryl triester of fatty acids having 12-18 carbon atoms and in which some of said acids are unsaturated, about 5-10 parts of the condensation product in mol to mol ratio of an ethanolamine with the fatty acids of animal and vegetable oils, and about 65-75 parts of a paraffin oil, and thereafter treating said fibers by contact with an alkoxy polyethylene glycol 325-850 monoester of a fatty acid having 12-22

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carbon atoms in which the alkoxy radical has 1-2 carbon atoms.

2. The method of claim 1 in which the wool subjected to carbonization of cellulose present therein at a point in the process between said treatments.

3. The method of claim 1 in which said fatty acids are contained in coconut oil.

4. The method of claim 1 in which said ethanolamine is diethanolamine.

5. The method of claim 1 in which said triester is a natural glyceride oil.

6. The method of claim 1 in which said alkoxy group has 1-2 carbon atoms.

7. The method of claim 1 in which said glycol has a molecular weight of about 350-550.

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