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METHODS FOR PROCESSING TEXTILE MATERIALS

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This invention relates to improvements in textile processing procedures and more particularly the invention relates to improved methods of lubricating, fulling and scouring textile fibers, yarns and fabrics of wool or mixtures of wool and other materials.

It is conventional practice to apply a lubricating material to wool fibers to assist in picking, carding, spinning, weaving and the like and materials which have been employed for this purpose include emulsifiable vegetable oils, mineral oils and mixtures of such oils. After the lubricating material has served its purpose, it is thereafter necessary to remove the same with soap and/or a detergent since if even a small quantity of the lubricating material is allowed to remain in the finished fabric, it generally results in the fabric having an undesirable appearance, hand and/or odor and also interferes with the dyeing of the fabric. Generally, from about 4 to 6%, by weight of the wool, of lubricant is applied and this is thereafter wasted since as yet no satisfactory method of recovering the lubricating agent in re-usable form has been devised. In addition, conventional practice requires the use of 4% to 5% soap or detergent and 3% to 5% alkali, based on the weight of wool, to remove the lubricating agent after it has served its purpose.

According to this invention, the waste inherent in prior art procedures is eliminated, and improved results are otherwise obtained by applying to the wool a small percent of a surface active agent, of a class to be subsequently defined, which is capable of acting as an excellent lubricating agent during carding, spinning, and weaving, a fulling agent during fulling, and which is not only readily removed in scouring, but which actually assists in removing other soils that might be present on the wool fiber. In other words, according to this invention, a single material acts as lubricating, fulling and scouring agent.

The new procedure of this invention has several other important advantages, and a first such advantage is that even if scouring is not effective in completely removing the processing agent from the wool, the surface active lubricants employed in the process of this invention do not materially interfere with the dyeing procedure. Prior to this invention, inadequate scouring has been a frequent cause of difficulty since it is generally impossible to effect even and complete removal of the lubricating agent from the fabric and even small amounts of conventional lubricants remaining in the fabric can result in uneven dyeing or shading. When, however, woolen materials are processed according to this invention, any lubricant remaining in the fabric at the time of the dyeing operation seems to be uniformly distributed and causes no difficulty.

Another important advantage of this invention is that smaller quantities of lubricant can be used because of the high degree of effectiveness of the class of materials employed. In other words, the process of this invention not only eliminates the use of detergents and/or alkalies to remove the spinning oil from the textile fibers, but also eliminates the necessity of empolying such quantities of a lubricant in the first instance.

Still another advantage of the invention is that it simplifies problems of inventory and requires the stocking of only one material. The prior art process has heretofore required that a supply of at least two or three materials be maintained and the new process of this

invention eliminates the administrative work normally necessary for making certain that adequate supplies of the several materials are on hand at all times.

A further advantage of the new process of this invention is that it permits the use of conditions which result in less damage to the wool fibers. It is well known by those skilled in the art that subjecting wool fabrics to highly alkaline conditions results in damage to the wool fibers and yet prior to this invention, the use of a very alkaline and high temperature bath for scouring has generally been necessary to completely remove the lubricant conventionally applied to wool fibers. Numerous efforts have been made to employ acid or relatively neutral conditions in scouring but such procedures have not been widely employed because it is quite difficult to obtain a completely satisfactory "hand" under such conditions. According to the process of this invention the fulling and scouring operations can be conducted at any desired pH.

A still further advantage of the new process of this invention is that it permits goods to be carbonized in the grease with less discoloration than when conventional lubricants are employed. In many instances, carbonizing in the grease permits an overall simplification of the production procedure but prior efforts to carbonize before scouring have resulted in yellowing of the textile material to an extent which makes it unsuitable for light shades. By the process of this invention the textile material can be carbonized in the grease and still retain a satisfactory degree of whiteness.

The class of materials which are employed as processing agents according to this invention, are high molecular weight, water-dispersable, amine bases, and acid addition salts thereof, said bases being capable of being represented by the formula:

$$R_1[N(C_2H_4O)_xR_2]_nR_3N[(C_2H_4O)_yR_4]_2$$

wherein N is the symbol for nitrogen; C is the symbol for carbon; H is the symbol for hydrogen; O is the symbol for oxygen; R_1 represents a hydrophobic group; R_2 and R_4, in each instance, represent hydrogen or blocking groups; R_3 represents a divalent hydrocarbon connecting radical having from 1 to 6 carbon atoms; x and y represent integers of from 0 to 8 in each instance and the total of all x's and all y's is from 3 to 8; and n represents an integer from 0 to 1 inclusive. Mixtures of amine bases of the above formula and/or of acid addition salts thereof can be suitably employed and, in fact, the preferred processing agents according to this invention are generally mixtures, since it is exceedingly difficult and unnecessarily expensive to prepare compounds of the above formula in pure form.

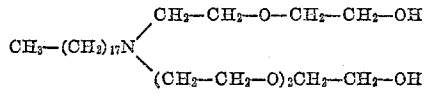
The nature and length of the hydrophobic group in compounds of the above formula are quite important since both the length of the group and its chemical nature affect the properties and partially determine the suitability of the compounds for use according to this invention. A first important consideration is that the length and nature of the group must be balanced against the number of oxyethylene groups in the compound so that the compound is readily water dispersable, but not truly water soluble. In other words, the hydrophile-lipophile balance of the compound must be such that it will readily form an aqueous dispersion which is stable for at least about 2 to 10 hours, but must not be such that the compound will form a molecular solution. Other important considerations are the nature of substituent groups on the hydrophobic radical, the degree of saturation, and the presence or absence of branched chains. Preferably the hydrophobic group is free of substituents and is a pure hydrocarbon radical, but certain groups can be present without making the compound unsatisfactory for purposes of this invention. For example, a hydroxy substituent

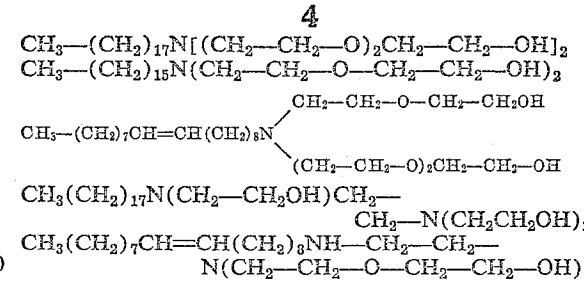
upon the hydrophobic group is permissible although it reduces the hydrophobic nature of the group and requires that the compound have a smaller number of oxyethylene groups than would otherwise be most advantageous. The hydrophobic group is also preferably saturated since it has been found that compounds containing a high degree of unsaturation are not so effective as lubricants, although the hydrophobic group may be ethylenically unsaturated at 1 or 2 points without destroying the lubricity of the compound and in certain instances compounds having a small degree of unsaturation might be preferred because of their liquid form at room temperature. Straight chain hydrophobic groups are generally preferred to branched chain aliphatic radicals. All of these factors not only affect the lubricity of compounds of the above formula but, at least in some instances, also have an effect upon the total number of carbon atoms which may be suitably present in the hydrophobic group. As a general rule, however, the number of carbon atoms in the hydrophobic group can suitably range from 10 to 20 with the preferred number being from 14 to 18.

The radical represented by R_3 in the above formula is a divalent aliphatic hydrocarbon connecting radical having from 1 to 6 carbon atoms. It will be apparent from the formula that R_3 can either link the hydrophobic group R_1 to the terminal amine group through an intermediate amine group or can directly link the group R_1 to the terminal amine group through a carbon to carbon linkage and that in the latter instance, R_1 and R_3 may suitably be considered as a single hydrophobic radical. In any event, R_3 also has a hydrophobic influence and its length partially determines the optimum number of carbon atoms in the primary hydrophobic group represented by R_1. In instances where R_3 links the primary hydrophobic group R_1 to the terminal amine group through an intermediate amine group, R_3 preferably represents an ethylene group and partially offsets the hydrophilic effect of the intermediate amine group, and in instances where R_3 directly links R_1 to the terminal amine group, R_1 preferably represents a methylene group so that the total number of carbon atoms in R_1 and R_3 is from 15 to 19.

Compounds of the above formula containing only one amine group are generally preferred and, as a rule, give better results than compounds containing two amine groups. If the compound contains only one amine group, however, it is generally necessary that it contain a larger number of oxyethylene groups than when the surface active agent contains two amine groups, since the compounds employed in the process of this invention should be water-dispersable and an amine group acts as a water solubilizing group. With compounds containing a single amine group, the total number of oxyethylene groups should normally be from 3 to 8, with the preferred number depending upon the nature of the aliphatic chain. In the instance of a compound containing a single amine group and in which the hydrophobic radical (R_1+R_3) is an octadecyl radical, the preferred number of oxyethylene groups is 5. In the case of compounds containing two amine groups, the total number of oxyethylene groups is generally from 2 to 5, with the preferred number being 3 in the case of a compound in which the hydrophobic radical R_1 is an octadecyl radical and the connecting radical R_3 is an ethylene radical. The oxyethylene chains in compounds suitable for use in this invention can terminate in free hydroxy groups or in hydroxy groups blocked by transformation into lower alkoxy groups, such as methoxy and ethoxy, or lower fatty acid acyloxy groups as illustrated by acetoxy and formoyl groups.

Specific examples of compounds which can suitably be employed in this invention include the following:





Compounds of this type can be prepared by procedures well known in the art and several mixtures, containing compounds of the above type and suitable for use in this invention, are commercially available.

The processing agent of this invention can be applied at any stage of operations where a lubricant, fulling agent or scouring agent is conventionally applied since it serves to replace all such agents employed in prior art procedures. For example, a small amount of the processing agent can advantageously be applied at the picker to lubricate the fibers during picking, carding, spinning, and weaving. An additional quantity of the processing agent can advantageously be applied immediately before or during fulling since if sufficient lubrication is applied prior to fulling to give the best results in this operation, it is generally in excess of the amount necessary for best lubrication of the fibers during picking, carding, spinning, and weaving.

The amount of the processing agent most advantageously applied at the picker depends upon a number of factors, foremost among which is the amount of natural oils in the fibers, but generally a suitable amount of the processing agent will vary from 0.1% to 5% with the preferred range being from 1% to 2%. It will be noticed that this is considerably below the amount of wool oil conventionally applied at this point since at least about 5% of a conventional wool oil is normally required for best results. The processing agent is preferably applied at this point in the form of an aqueous dispersion since a dispersion enables one to make a relatively even application of the processing agent, and the water in the dispersion acts to hold down fly. A dispersion having a concentration varying within wide limits can suitably be employed and the only important considerations are that the dispersion be not so concentrated that an even application of the processing agent is difficult, nor so dilute that it is necessary to make the stock too wet in order to apply a satisfactory amount of the processing agent. As a general rule, a suitable concentration for the aqueous dispersion to be applied at the picker is from 3% to 30% with the preferred concentration being from 5% to 15% by weight. The aqueous dispersion may be applied by any suitable means and, for example, may be sprayed onto the stock from a nozzle or applied by means of an absorbent roll or the like.

It is generally advantageous to apply an additional quantity of the processing agent immediately preceding or during fulling to thereby provide better fulling and more effective scouring, and except for economic considerations, there is practically no upper limit as to the amount of the processing agent that can be applied at this point. One can, for example, apply an additional 20% of the processing agent at this point with satisfactory results but such a large amount of the processing agent is completely unnecessary and it is seldom, if ever, advantageous to apply more than about 5% of the processing agent during or immediately before fulling. The preferred amount of the agent to be employed at this time, assuming that the fabric already contains from 1% to 2% of the processing agent previously applied for purposes of fiber lubrication, is from about 0.5% to 3% based upon the weight of the goods. Of course, if the fabric contains less than about 1% of a processing agent according to this invention due to the fact that a conventional lubricant

was employed, alone or in combination with an agent according to this invention, for fiber lubrication in the preceding processing operation, or due to the fact that part of the agent previously applied has been removed, or for any other reason, larger amounts of the processing agent are generally advantageous, and an amount of the agent should be applied to bring the total amount of the agent present in the fabric up to at least about 1.5% to 5% by weight. The processing agent is also, in this instance, preferably applied in the form of an aqueous dispersion and again the concentration of the dispersion is relatively unimportant. Under proper conditions, a dispersion of the processing agent having a concentration of from 0.1% to 60% by weight can be employed, but there is a well-recognized optimum moisture content for fulling, and it is generally advantageous to employ a dispersion of a proper concentration to furnish the desired quantity of water for the fulling operation. Since an optimum moisture content for fulling is generally from 50% to 100% by weight of the goods, and since it is generally advantageous to add from about 0.5% to 3% of the processing agent, it will be seen that the preferred concentration for the aqueous dispersion to be employed immediately before fulling is from about 0.5% to 6%. The dispersion of the processing agent can be applied before or during fulling by any suitable means but a preferred procedure comprises applying the agent by means of a conventional "soaper" before the fabric is placed in the fulling mill.

A dispersion suitable for use in this invention can be readily prepared since the processing materials are readily water dispersable and in most instances no special apparatus is required. Some agitation is, of course, advantageous to insure uniformity of the dispersion, and in some instances better results are achieved if the water employed in forming the dispersion is heated, for example, to from 60° to 95° C., but in most instances all that is necessary is for the processing material to be added to a calculated amount of water to give a dispersion of the desired concentration and the resulting mixture slightly agitated.

Because the processing agents of this invention are readily water dispersable and do not interfere with dyeing when present in the dye bath in limited quantities, it is frequently possible to eliminate the scouring operation which conventionally precedes dyeing, and this is particularly true when the goods are of the type which are not subjected to a fulling operation. Fabrics formed from halogenated wool fibers and fabrics formed from a blend of wool with at least about 50% of a non-fulling fiber material such as nylon fibers, Dacron polyester fibers, or Orlon and Acrilan acrylic fibers, do not full and need not be subjected to a fulling operation. When employing a processing agent according to this invention, the conventional scouring operation can also be eliminated and the fabric can be taken directly from the loom to the dye bath.

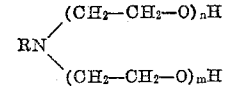
Conventional temperatures can be employed in the process of this invention although it is an advantage of the invention that the scouring operation can be conducted at room temperature. If the wool is contaminated with relatively large amounts of soil, it has been found that improved scouring is obtained if the bath is slightly warm and for this reason, a temperature of about 100° F. is generally preferred for scouring.

It is also an advantage of the invention that no pH adjustments are required in either the fulling or scouring operations. The amines employed in the new process of this invention are normally basic and give an unadjusted pH of about 9 or 10 in aqueous solution and generally it is advantageous to conduct both the fulling and scouring operations under such basic conditions without pH adjustment. If desired, however, both the fulling and scouring operations can be conducted under neutral or acidic conditions. For example, the dispersion applied immediately before or during fulling can be neutralized with an organic acid, such as acetic or formic, or with a non-oxidizing mineral acid, such as sulfuric acid or hydrochloric acid, before it is applied to the material, or an acid addition salt of the amine base can be employed in forming the dispersion. Likewise, an acid may be added to the bath employed in the scouring operation in amounts sufficient to result in the bath being made neutral or acidic since it is a characteristic of the materials employed in the process of this invention that they possess a greater degree of detergency when in the form of acid addition salts than when in the form of free bases.

The invention will now be illustrated by the following specific examples, in which all parts are by weight unless otherwise indicated:

Example I

To a wool blend consisting of 40% 12-month Texas wool, 20% New Mexican wool, 20% 8-month Texas wool, and 20% fine Lister Noils (noils from a Lister comb), there is applied a 7½% aqueous dispersion of a mixture of amines of the formula:

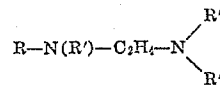


wherein m and n represent integers with the average total of m and n being about 5, and R represents octadecyl, in most instances, with smaller amounts of compounds wherein R represents cetyl, and oleyl also being present. An amount of the dispersion is applied such that about 1.75% of the amine mixture is deposited on the wool and the wool is then picked, carded, spun into a variety of yarn weights and woven into plain weave woolen fabrics. In comparative tests on 20 pieces of fabric thus prepared, four pieces were fulled and scoured with the addition of water only and were thereafter carbonized, rinsed, and dyed; four pieces were "soaped" with an additional 1.25% of the above amine mixture (bringing the total to 3%), fulled, scoured, carbonized, rinsed, and dyed; eight pieces were "soaped" with an additional 1.25% of the amine mixture, fulled, scoured, with the addition of 1% acetic acid, carbonized, rinsed, and dyed; and four pieces were crabbed, carbonized, "soaped" with 1.25% of the amine mixture (total used 3%—total present about 2%), fulled, scoured, and dyed. All 20 pieces were satisfactory, but the fulling operation gave better results when at least about 2% of the amine mixture was present on the fabric.

In this instance comparative tests were also made using, in place of 1.75% of the amine mixture, 5% of a standard and well-accepted mineral oil lubricant (Twitchel 7421 wool oil) for the picking, carding, spinning, and weaving operations. A smaller reduction in average fiber length during carding was experienced when employing the mixture of amines than when employing the standard mineral oil lubricants (the mean reduction in fiber length when employing the amine mixture was 0.1 inch, while the mean reduction in fiber length when employing the mineral oil lubricant was 0.28 inch), and during spinning there were only 107 breaks per thousand spindle hours for 9.15 thousand spindle hours in the case of fibers lubricated with the amine mixture, whereas there were 150 breaks per thousand spindle hours for 11.7 thousand spindle hours in the case of fibers lubricated with the standard mineral oil lubricant.

Example II

To a wool blend containing 45% wool and 55% Nylon 66 fibers there is applied approximately 25% of a 7% aqueous dispersion of a mixture of aliphatic diamines of the formula:



in which R' in each instance represents either hydrogen, hydroxyethyl, or a polyoxyethylene subsituent i.e.

$$—C_2H_4O—C_2H_4OH$$

or

$$—(C_2H_4O)_2—C_2H_4OH$$

with the average total number of oxyethylene groups being 3, and R represents octadecyl in most instances with smaller amounts of compounds also being present in which R represents a cetyl, or an oleyl group. The wool mixture is then picked, carded, spun, woven, and dyed with the fabric being taken directly from the loom to the dye beck. The dyed fabric is substantially free of objectionable shading and is otherwise satisfactory.

The procedure when employing other processing agents according to this invention is the same as in the above examples.

Having thus described my invention, what I desire to claim and secure by Letters Patent is:

1. In the processing of a textile material comprising wool fibers wherein said material is subjected to the processing operations of picking, carding, spinning, weaving, fulling and scouring, the improvement which comprises applying to the textile material prior to picking, as the sole essential processing agent acting as a lubricating agent, fulling agent and scouring agent, a composition selected from the group consisting of amines capable of being represented by the formula:

$$R_1[N(C_2H_4O)_xR_2]_nR_3N[(C_2H_4O)_yR_4]_2$$

wherein R_1 represents a hydrophobic radical having from 10 to 20 carbon atoms and is selected from the group consisting of hydrocarbon radicals and hydroxy substituted hydrocarbon radicals; R_2 and R_4, in each instance, represent a member selected from the group consisting of hydrogen, lower alkyl radicals, and lower fatty acid acyl radicals; R_3 represents a divalent hydrocarbon connecting radical having from 1 to 6 carbon atoms; x and y represent integers of from 0 to 8 in each instance and the total of all x's and all y's is from 3 to 8; and n represents an integer from 0 to 1 inclusive, the hydrophile-lipophile balance of said amine being such that it is readily water dispersible but will not readily form a molecular solution in water; acid addition salts of such amines, and mixtures thereof.

2. The improvement of claim 1 wherein the processing agent is applied in an amount from about 0.01 to about 5%, based on the weight of the textile material.

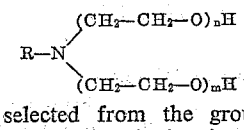
3. The improvement of claim 2 wherein the amount of said processing agent applied is from 1% to 2%.

4. The improvement of claim 2 wherein an additional quantity of said processing agent is applied immediately prior to fulling to assist in the fulling and scouring operations.

5. The improvement of claim 4 wherein the amount of said processing agent applied immediately prior to fulling is from 0.5% to 6%, based on the dry weight of said textile material.

6. The improvement of claim 4 wherein said processing agent comprises an aliphatic mono-amine in which the amino group has at least one polyoxyethylene hydrophilic substituent, the total mean number of oxyethylene groups in said amine being from 3 to 8 inclusive.

7. The improvement of claim 6 wherein said aliphatic mono-amine has the formula



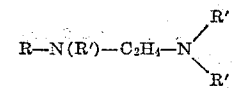
wherein R is selected from the group consisting of octadecyl, cetyl and oleyl and wherein m and n represent integers with the average of m and n being about 5.

8. The improvement of claim 6 wherein said amine has an octadecyl substituent.

9. The improvement of claim 8 wherein the total mean number of oxyethylene groups in said amine is 5.

10. The improvement of claim 4 wherein said processing agent comprises an ethylene-diamine in which one of the amino groups is substituted with a hydrophobic aliphatic radical and the compound contains at least one hydrophilic oxyethylene substituent, the total means number of oxyethylene groups in said amine being from 2 to 5 inclusive.

11. The improvement of claim 10 wherein said processing agent comprises an ethylene-diamine having the formula



wherein R is selected from the group consisting of octadecyl, cetyl and oleyl and wherein R' is selected from the group consisting of hydrogen, hydroxyethylene, $—C_2H_4O—C_2H_4OH$ and $—(C_2H_4O)_2—C_2H_4OH$, with the average number of the oxyethylene groups present being 3.

12. The improvement of claim 10 wherein said hydrophobic radical is an octadecyl radical and the total means number of oxyethylene groups in said amine is 3.

13. A method for processing a textile material composed of a member selected from the group consisting of blends of wool with synthetic fibers, halogenated wool fibers, and mixtures thereof, which comprises applying to said textile material from about 0.1% to 5% of a processing agent consisting essentially of a member selected from the group consisting of amines capable of being represented by the formula:

$$R_1[N(C_2H_4O)_xR_2]_nR_3N[(C_2H_4O)_yR_4]_2$$

wherein R_1 represents a hydrophobic radical having from 10 to 20 carbon atoms and is selected from the group consisting of hydrocarbon radicals and hydroxy substituted hydrocarbon radicals; R_2 and R_4, in each instance, represents a member selected from the group consisting of hydrogen, lower alkyl radicals, and lower fatty acid acyl radicals; R_3 represents a divalent hydrocarbon connecting radical having from 1 to 6 carbon atoms; x and y represent integers of from 0 to 8 in each instance and the total of all x's and all y's is from 3 to 8; and n represents an integer from 0 to 1 inclusive, the hydrophile-lipophile balance of said amine being such that it is readily water dispersible but will not readily form a molecular solution in water; acid addition salts of such amines, and mixtures thereof, and thereafter subjecting said textile material to the operations of picking, carding, spinning, weaving, and dyeing without an intermediate scouring operation.

14. A process according to claim 13 wherein said processing agent comprises an alkyl amine having at least one hydrophilic oxyethylene substituent, the total mean number of oxyethylene groups being from 3 to 8 inclusive.

15. The improvement of claim 14 wherein the alkyl group in said amine is an octadecyl radical.

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