

1

2,918,428

FULLING AND SCOURING COMPOSITIONS

Joseph Hunter, Brooklyn, N.Y., and Maurice J. Kelley, Bloomfield, and William Nothum, Chatham, N.J., assignors to Nopco Chemical Company, Harrison, N.J., a corporation of New Jersey

No Drawing. Application July 7, 1955
Serial No. 520,616

10 Claims. (Cl. 252—3.75)

This invention relates to improved surface active agents and more particularly to such agents which are highly useful in the fulling and scouring of woolen and worsted fabrics.

In converting raw wool to a finished fabric a number of processing steps are required. The first step is to subject the raw wool to a scouring procedure to remove the wool grease, dirt and other extraneous foreign matter which may have become attached to the wool fibers. Next the scoured raw wool is subjected to a carding operation which causes the wool fibers to be aligned in a relatively parallel direction. Thereafter the carded wool is put through a spinning operation to convert the fibers to woolen yarn. Then the yarn is woven into cloth. In the carding, spinning and weaving operations various oily materials are usually applied to the wool to reduce or eliminate static electricity and to aid the workability of the fibers and the yarn by providing lubrication for the fibers and the yarn. The cloth as it comes from the weaving operation is comparatively loose, porous and thin. In order to convert the cloth to the form in which it is used in producing woolen clothing and other customer goods the cloth must be subjected to a fulling process, and immediately thereafter it is subjected to a scouring procedure to remove the lubricants employed in the carding, spinning and weaving operations. Frequently, after the fulling and scouring procedures, the cloth is subjected to a carbonizing procedure to remove any cellulosic matter which may not have been removed by the procedure operations. This carbonizing procedure involves wetting the woolen cloth with a dilute solution of an acid such as sulfuric acid or of a salt such as aluminum chloride and then baking the cloth which will result in the sulfuric acid being concentrated through evaporation of the moisture from the dilute solution and which will result in formation by hydrochloric acid from the aqueous solution of aluminum chloride. The concentrated acid material carbonizes any cellulosic material. The carbonized material is then removed by a washing procedure. In many cases the commercial procedure which is now used for the production of woolen cloth involves carbonizing the cloth just prior to the fulling and scouring procedures. In those cases where the cloth has been carbonized prior to the fulling and scouring operations, it has been necessary in the past to neutralize the acid material remaining on the cloth in a separate step and then thoroughly rinse the cloth before carrying out the fulling and scouring operations.

The fulling and scouring procedures involve first passing the thin, loose and porous woolen cloth through a series of rollers which tend to cause the fibers in the cloth to migrate and thereby become entangled with each other thus reducing the thinness, looseness and porosity of the cloth. There are very small scales on the sides of woolen fibers and these scales tend to keep the various fibers in the cloth from pulling away from each other. To aid in the migration of the fibers during the fulling operation, it has been the custom in the past to apply

2

solutions of water-soluble soaps to the cloth prior to the fulling procedure to help lubricate and moisten the fibers. Following the fulling operation the cloth is scoured by immersing it in water and thoroughly agitating the cloth in the water. The soap solution which has been applied to the cloth for the fulling operation is dissolved in the scouring bath and serves as the detergent to wash from the cloth the lubricating oils used in the carding, spinning and weaving operations. Experience has shown that it is advantageous to have present in the fulling and scouring solutions certain amounts of soda ash, i.e. sodium carbonate. The use of the common water-soluble soaps in the fulling and scouring procedures has not been completely satisfactory for a number of reasons. As is well known, ordinary water-soluble soaps have very poor resistance to hard water, which is encountered in many areas. Another reason is that in the scouring of the cloth following the fulling procedure, the soaps do not rinse out of the woolen cloth as efficiently as desired and do not remove from the cloth with the efficiency desired the lubricants used in the carding, spinning and weaving operations. Another disadvantage is the rather high labor cost in the preparation of stock solutions of soap for use in the fulling and scouring procedure. Because of the inherent disadvantages of soaps for fulling and scouring of woolen cloth, there is a considerable demand for improved agents for use in the fulling and scouring procedures and particularly for such agents which will be highly stable in large concentrations of soda ash.

It is the object of this invention to provide improved compositions for use in the fulling and scouring of woolen and worsted fabrics.

A further object of the invention is to provide compositions for the fulling and scouring of woolen and worsted fabrics which will readily dissolve in and which will be highly stable in aqueous solution containing high concentrations of carbonates.

Another object of the invention is to provide compositions useful for fulling and scouring woolen and worsted fabrics which will be highly useful in treating such fabrics immediately after they have been carbonized and which will eliminate the need for separate washing and rinsing steps following the carbonizing procedure when that procedure is carried out prior to the fulling and scouring procedures.

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

The above and other objects of the invention are accomplished by providing compositions which are mixtures of fatty amide condensates of the type described hereinafter and sulfated fatty acids having a relatively high SO₃ content.

The compositions of the invention readily dissolve in and are highly stable in aqueous solutions containing large amounts of carbonates. They have excellent detergent properties and give very excellent results when employed for the fulling and scouring of woolen and worsted fabrics even when the fulling and scouring are carried out immediately following carbonizing of the fabrics. By use of the compositions of our invention it is possible to have sufficient soda ash present in the fulling solution to neutralize the acid material remaining on the carbonized cloth plus the amount of soda ash needed for the fulling and scouring procedures thus eliminating the separate steps of neutralizing and rinsing prior to fulling as is required in the prior art procedures. The compositions of the invention are highly superior to soap for use in the fulling and scouring of woolen and worsted fabrics both from a cost standpoint and because they can be employed under conditions where soap will not give satisfactory results. The ingredients employed

3

in preparing the compositions of the invention exert a synergistic effect on each other since the compositions have a far greater degree of detergency than would be expected from the detergency of the individual components. Because of the excellent detergent characteristics of the compositions they are highly suitable for use for general detergent purposes although they are primarily intended for use in the fulling and scouring of woolen and worsted fabrics.

The fatty amide condensates which are employed in preparing the compositions of the invention are produced by reacting an alkylolamine such as diethanolamine, N-ethylethanolamine, diisopropanolamine, 2-amino-2-methyl-1-propanol, isopropylethanolamine, trimethylolaminomethane, monoisopropanolamine, monoethanolamine, diglycerylamine, and similar alkylolamines or mixtures thereof with a fatty acylating substance containing at least 12 carbon atoms in the acyl group. From 1.5 mols to 3 mols of alkylolamine are employed for each equivalent of the fatty acylating substance. The fatty acylating substance may be a fatty acid or a mixture thereof, an acyl halide or an ester or esters of fatty acids. Thus fatty acids such as lauric, myristic, palmitic, stearic, oleic, ricinoleic and similar acids or acyl halides, alkyl esters or glycerides thereof can be employed if desired. The preferred acylating substance is coconut oil itself or a mixture of coconut oil and tallow and the preferred alkylolamine is diethanolamine. Although at least 1.5 mols of alkylolamine for each equivalent of fatty acylating material is employed, it is preferred to use at least 2 mols of the alkylolamine for each equivalent of acylating agent. Since the structure of the composition which is formed by reacting an alkylolamine with a fatty acylating agent employing an excess of the alkylolamine has not been definitely established, the reaction product obtained is referred to herein merely as a "fatty amide condensate" and such term is used to denote the product or products obtained when the acylating substance combines with the alkylolamine with the splitting off of water or a hydrohalide or an alcohol depending on the type of acylating agent employed.

The sulfated fatty acid which is employed in the compositions of the invention should have a relatively high content of SO_3 , i.e. at least about 10% and in most cases it is preferred that it have an SO_3 content of at least about 13% to 15%. The preferred sulfated fatty acid is sulfated oleic acid. Highly sulfated oleic acid can readily be prepared by sulfating low molecular weight alcohol esters of oleic acid and then saponifying the sulfated esters to give the free sulfated oleic acid. The sulfated group on the sulfated fatty acid is neutralized with an alkali in the usual manner following the sulfation. Such a sulfation procedure is well known in the art and, therefore, details will not be set forth herein. If desired, some of the sulfated oleic acid may be left in the form of its low molecular weight alcohol ester. However, in all cases the ester of the sulfated acid should be saponified to such an extent that the product has an acid value of at least 100. If desired, mixtures of highly sulfated fatty acid and sulfated fatty acid having an SO_3 content of less than 10% may be employed provided the final mixture in all cases has an average SO_3 content of 10% or greater. Likewise mixtures of sulfated fatty acids and sulfated fatty esters having an acid value below 100 may be employed with mixtures of sulfated fatty acids and sulfated fatty esters having acid values above 100 provided the acid value of the final mixture is at least 100. Fatty acids such as ricinoleic acid can be used in place of or together with oleic acid in preparing the highly sulfated fatty acid used for the products of the invention.

In preparing the compositions of the invention the fatty amide condensate and the sulfated fatty acid are employed in a ratio of from 5 parts of fatty amide condensate to 1 part of sulfated fatty acid to a ratio of 1

4

fatty acid. The preferred ratio is approximately 2 parts of fatty amide condensate to each part of sulfated fatty acid.

The products as prepared for shipment to the customer may be substantially moisture-free although in most cases in the preparation of the sulfated fatty acid some moisture will be introduced into the sulfated fatty acid product during the process of its manufacture. In most cases it is preferred that the moisture content of the compositions as shipped to the customer is not greater than about 70% for the obvious reasons of savings on freight, storage charges and container costs. If desired, the materials can be completely anhydrous although as a practical matter it is not economical to produce them completely free of water. Also in most cases the anhydrous products would be pastes instead of liquid materials. Such pastes would be more difficult for the textile mill to use than the liquid products.

In the fulling and scouring of woolen and worsted fabrics the compositions of the invention are used to replace the water-soluble soaps employed in the conventional fulling and scouring operations. The compositions of the invention are considerably superior to soap since they are stable under all conditions of use, i.e. both in hard and soft water and in alkaline and acid solutions. Considerable savings in labor are realized by the use of the compositions of the invention since the stock solutions may be quickly prepared whereas a considerable length of time is often required for the preparation of stock solutions of soap. The actual quantities of the compositions of the invention which are employed will vary depending upon each mill's particular requirements. In most cases it will be found that a concentration of from 0.5% to 2% (calculated on a moisture-free basis) of the compositions in the fulling and scouring solutions is sufficient to give very excellent results. The amount employed does not depend upon the quantity of sodium carbonates or sodium bicarbonate which is added to the fulling and scouring solutions. Consequently, if the woolen or worsted fabric is carbonized prior to fulling and scouring, it is not necessary to employ larger amounts of the compositions of the invention than when the carbonizing is done after the fulling and scouring even though sufficient additional soda ash to neutralize all the acid material remaining on the carbonized cloth is added to the fulling solution.

It has not been definitely determined just why the compositions of the invention give results which are superior to the use of soaps and why these compositions are exceedingly stable under both acid and alkaline conditions. It is very possible that the sulfated fatty acid and the fatty amide condensate form a stable salt or adduct which is not affected by either acid or alkaline conditions. Regardless of the reason for the excellent results obtained with the compositions of the invention, both laboratory and mill tests demonstrate quite clearly the outstanding utility of the compositions of the invention.

In some cases pine oil is added to fulling and scouring solutions for a number of purposes such as aiding the solutions to spread rapidly through the cloth, to prevent redeposition on the cloth of any dyes that may be removed from the cloth during the fulling and scouring, and to aid in removing from the cloth the lubricants applied during the carding, spinning and weaving operations. The compositions of our invention are completely compatible with pine oil and may be used freely with it.

For a fuller understanding of the nature and objects of the invention, reference may be had to the following examples which are given merely as further illustrations of the invention and are not to be construed in a limiting sense. Unless otherwise specified, all parts are given by weight.

Example I

A fatty amide condensate was prepared by reacting

5

2.2 mols of diethanolamine with one equivalent of a fatty material composed of a mixture of 75% coconut oil and 25% tallow. To 55 parts of this fatty amide condensate there were added 45 parts of a sulfated oleic acid having an SO_3 content of 13.5% and an acid value of 150. This sulfated oleic acid had been prepared by sulfating methyl oleate and then hydrolyzing the sulfated ester to give a sulfated fatty acid product having the desired acid value. The sulfated fatty acid contained 40% water. The composition prepared by mixing the fatty amide condensate with the sulfated oleic acid was highly stable in solutions containing relatively large amounts of carbonates. In addition the product was a highly effective foaming agent and detergent.

To show the synergistic effect which the ingredients have on each other with respect to foaming properties, the well known Ross-Miles foam test was carried out on a 0.5% active solution of each of the ingredients and of the blended composition in tap water at 25° C. The initial foam of the fatty amide condensate was 180 mm. After five minutes the foam was 150 mm. The initial foam of the sulfated acid was 175 mm. but after five minutes it was 0 mm. The blend of the fatty amide condensate and the sulfated acid had an initial foam of 255 mm. and after five minutes had a foam of 215 mm. thus unequivocally demonstrating that the ingredients had a highly synergistic effect on each other with respect to their foam producing properties.

A similar test with respect to the synergistic effect of the ingredients on each other as far as detergency is concerned was carried out. This test showed that there is a very high degree of synergism with respect to the detergent properties. This test was carried out in an Atlas Launder-O-Meter using a 0.15% active solution of the ingredients and of the blended composition in each case in tap water at 120° F. and using standard soiled woolen cloth supplied by the United States Testing Company. The fatty amide condensate had a detergency of 53% whereas the sulfated fatty acid had a detergency of 0%. The blend of the two ingredients, however, had a detergency of 54%.

The effectiveness of the blended composition as a fulling and scouring agent was tested in a textile mill on various types of woolen fabrics ranging from a fine worsted containing very little oil (1%) to a cheaper construction, i.e. 13½ ounce worsted with considerable oil on it. In the tests 27 pounds of the blended composition were mixed with 60 pounds of soda ash and sufficient water to give a total of 375 gallons. The resulting fulling solution was metered directly on the fabric using a weight of the fulling solution approximately equal to the weight of the fabric being treated. Approximately the same amount of time was employed for the fulling process as with conventional fulling agents. Following the fulling procedure the fabrics were then washed in the usual manner except that the conventional detergents employed in the scouring bath were replaced with equivalent amounts of the compositions of this example. The resulting fabrics had a very excellent hand, appearance and odor and a very low content of material extractable with ethyl ether and ethyl alcohol.

Example II

Another composition of the invention was prepared in the same manner as the product of Example I except that the fatty amide condensate was prepared from 2.2 equivalents of diethanolamine and one equivalent of coconut oil. When this fatty amide condensate was blended with the sulfated fatty acid employed in Example I using the same proportions as in Example I a highly satisfactory fulling and scouring composition was obtained.

Example III

Another excellent product was prepared employing 75

6

as the fatty amide condensate a product prepared by reacting 2.2 mols of diethanolamine with one mol of coconut fatty acids.

Example IV

Another product of the invention was prepared in the same manner as in Example I except that the fatty amide condensate was prepared from 2 mols of diethanolamine and one mol of lauric acid.

Example V

The product of this example was similar to that of the previous examples except that the fatty amide condensate was prepared from 2 mols of diethanolamine and one mol of myristic acid.

Example VI

Another product of the invention was prepared as in the previous examples except that the fatty amide condensate was prepared from 2 mols of diethanolamine and one mol of palmitic acid.

Example VII

A product wherein the fatty amide condensate was prepared from a mixture of alkylolamines was prepared by reacting one equivalent of lauric acid with a mixture of 1½ mols of diethanolamine and one-half mol of diisopropanolamine. This fatty amide condensate was blended with the sulfated fatty acid employed in Example I using the same ratios as in Example I to give a highly effective fulling and scouring composition.

Example VIII

In this example the fatty amide condensate was prepared by reacting one equivalent of coconut fatty acids with a mixture of 1½ mols of diethanolamine and one-half mol of monoisopropanolamine. The blended compositions prepared from this fatty amide condensate and the sulfated oleic acid of Example I using the same ratios as in Example I gave a very excellent fulling and scouring composition.

Example IX

18.7 parts of the fatty amide condensate employed in Example I were mixed with 9.7 parts of a sulfated oleic acid having an acid value of 150 and an SO_3 content of approximately 9%, 9.1 parts of a sulfated oleic acid having an acid value of 70 and an SO_3 content of 14%, 2.9 parts of pine oil and 59.6 parts of water. The sulfated oleic acid having the SO_3 content of approximately 9% contained about 30% water and the sulfated oleic acid having an SO_3 content of about 14% contained about 44% water. Thus the final composition had a water content of approximately 67%. This product had very excellent stability in solutions containing relatively large amounts of carbonates and had excellent fulling and scouring properties when employed in full scale plant tests. The product of the example was employed in a fulling solution at a concentration of 5 ounces per gallon, i.e. approximately 1.7 ounces per gallon on an active basis. The fulling solution also contained 1½ ounces of soda ash, 1½ ounces of sodium bicarbonate and ¼ ounce of a commercial sequestering agent. Very excellent results were obtained in the fulling of the woolen fabrics with this solution using the same fulling time as when soap was employed as the fulling detergent. The sudsing qualities of the fulling solution were far superior to the fulling solutions prepared using soap as the detergent and in addition there was a great saving in labor in the preparation of the fulling solution as compared to the preparation of the solution when soap is used as the detergent. It was found that the fabric after it came out of the washing bath was completely free of any carbon tetrachloride-soluble material whereas when soap was employed as the detergent in fulling and scouring it

was not possible to obtain a fabric completely free of carbon tetrachloride-soluble material.

Example X

Another fulling and scouring composition of the invention was prepared by mixing 16.8 parts of the fatty amide condensate of Example I with 12.8 parts of the sulfated oleic acid of Example IX which contained 9% SO_3 , 8.0 parts of the sulfated oleic acid of Example IX which contained 14% SO_3 , 2.9 parts of pine oil and 59.5 parts of water. The resulting composition was a very excellent fulling and scouring composition and had the same excellent qualities as the products of the previous examples.

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

1. A composition consisting essentially of (a) a fatty amide condensate prepared by reacting from one and one-half mols to three mols of an alkylolamine with one equivalent of a fatty acylating substance selected from the group consisting of fatty acids, acyl halides, esters of fatty acids and mixtures thereof having a fatty chain length of at least twelve carbon atoms and (b) a sulfated fatty acid having an acid value of at least 100 and an SO_3 content of at least 10%, said fatty amide condensate and said sulfated fatty acid being present in a ratio of from about five parts of fatty amide condensate to one part of sulfated fatty acid to about one part of fatty amide condensate to two parts of sulfated fatty acid.

2. The composition of claim 1 wherein the fatty acylating substance is a mixture of coconut oil and tallow.

3. The composition of claim 1 wherein the fatty amide condensate is prepared from one equivalent of the fatty

acylating substance and about two equivalents of alkylolamine.

4. The composition of claim 3 wherein the fatty acylating substance is a mixture of coconut oil and tallow.

5. The composition of claim 3 wherein the alkylolamine is diethanolamine.

6. The composition of claim 3 wherein the sulfated fatty acid material is a mixture of sulfated methyl oleate and sulfated oleic acid having an acid value of at least about 150 and an SO_3 content of at least about 13%.

7. The composition of claim 4 wherein the alkylolamine is diethanolamine.

8. The composition of claim 4 wherein the sulfated fatty acid material is a mixture of sulfated methyl oleate and sulfated oleic acid having an acid value of at least about 150 and an SO_3 content of at least about 13%.

9. The composition of claim 5 wherein the sulfated fatty acid material is a mixture of sulfated methyl oleate and sulfated oleic acid having an acid value of at least about 150 and an SO_3 content of at least about 13%.

10. The composition of claim 7 wherein the sulfated fatty acid material is a mixture of sulfated methyl oleate and sulfated oleic acid having an acid value of at least about 150 and an SO_3 content of at least about 13%.

References Cited in the file of this patent

UNITED STATES PATENTS

2,245,412	Pilgrim	June 10, 1941
2,483,253	Young	Sept. 27, 1949

FOREIGN PATENTS

665,475	Great Britain	Jan. 23, 1952
---------	---------------	---------------