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# REACTION PRODUCTS OF PRIMARY ALKYL AMINES AND EPICHLOROHYDRIN

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## ABSTRACT OF THE DISCLOSURE

A water dispersible reaction product of a primary alkyl amine ($C_{12}$–$C_{30}$) and 1.25 to 2 moles of epichlorohydrin per mole of amine, said product being reacted at 80°–200° F. for 30 minutes to 10 hours and then neutralized to an acid value of less than about 30 at a temperature of 60°–150° F.

This application is a continuation-in-part of our copending application, Ser. No. 528,793, filed Feb. 21, 1966, now abandoned, entitled Product and Process for Permanently Attaching Long Alkyl Chain Moieties to Textile Materials. This invention relates to a product and a process which are especially advantageous for permanently attaching long alkyl chain moieties to textile materials.

The reasons for attaching long alkyl chain moieties to textile materials include (a) the improvement of fiber-to-fiber lubricity, (b) softening, and (c) improving water repellency. In order to achieve these results in an economic manner, it is highly desirable that the product can be applied from aqueous milieus. It is also important that the product applied to the textile material does not adversely affect the cloth by, e.g., causing the cloth to yellow or lose strength.

This invention, therefore, has among its objects the provision of a product containing long alkyl chains which can be applied to textile materials to provide a permanent modification of the properties of said textile material.

It is a more specific object of this invention to provide products of the above type which are in convenient forms and/or have particular utility with respect to providing one or more improved textile characteristics.

It is a still further object of this invention to provide new and improved methods for applying long alkyl chain moieties to textile material.

It is a still further object of this invention to provide textile materials having improved properties.

The above objects and other objects as are hereinafter seen can be achieved by providing:

A water dispersible reaction product of a primary alkyl amine having an alkyl chain of from about 12 to about 30 carbon atoms and from about 1.25 to about 2 moles of epichlorohydrin per mole of amine, said reaction product being prepared at a temperature of from about 80° F. to about 200° F., and a reaction time of from about 30 minutes to about 10 hours, and said reaction product being neutralized to an acid value of less than 30, preferably from about 5 to about 15, the neutralization being carried out at a temperature of from about 60° F. to about 150° F.

The above reaction product is critical to achieving the objects of this invention. For example: if a molar ratio of epichlorohydrin to amine of less than 1.25:1 is used, the resulting reaction product is not easily dispersed in water. The reaction product is dispersed in water before it is attached to the textile material as hereinafter described. If, on the other hand, a molar ratio of epichlorohydrin to amine of more than 2:1 is used, the resulting reaction product is not as efficient, e.g., as a softener, on a weight basis and the resulting treated textile material, as hereinafter described, has a poorer color in that the treated textile material tends to discolor (yellow) more. The preferred range of molar ratios of epichlorohydrin to amine based on all performance considerations is from about 1.5:1 to about 1.75:1 and the preferred ratio is 1.5:1.



The primary alkyl amines which can be used in preparing the reaction products of this invention include those wherein the alkyl groups can be derived from naturally occurring glycerides (e.g., tallow, coconut oil, palm oil, soybean oil, lard, etc.), or can be obtained from petroleum sources and/or olefins such as ethylene polymers. The source is not important. However, the preferred alkyl groups are primarily straight chain moieties. Branched chain alkyl groups and alkyl groups containing cycloaliphatic moieties can also be used. The amine group can be attached at any point on the chain, but it is preferred to have the amine group attaached to a terminal carbon atom. Mixtures of amines are preferred. Specific examples of amines include dodecyl, tetradecyl, hexadecyl, octadecyl, pentapropylene, hexapropylene, dodecylcyclohexyl, 12-cyclohexyldodecyl, eicosyl, and docosyl amines.

The preferred reaction temperatures used to form the reaction product is the lowest temperature which will give a liquid reaction mixture. However, if shorter reaction times are desired, a higher reaction temperature is required. Where it is permissible to use organic solvents, temperatures below 125° F., e.g., temperatures down to about 80° F., can be used. Without a solvent, however, temperatures below 125° F. cannot be used since the reaction mixture becomes a solid. Where the solvents have to be removed before use, they are not desirable. Temperatures above about 200° F. give a reaction product which is unacceptable in that the resulting treated textile material is discolored too much.

The higher reaction temperatures are associated with the shorter reaction times to avoid exposing the reaction mixture to high temperatures too long.

The reaction time varies from about 30 minutes to about 10 hours and preferably from about 3 hours to about 6 hours. The shortest time in which the reaction can be completed is preferred since the reaction product is discolored if exposed to the heat for too long a time and the subsequently treated textile material is also discolored unacceptably.

The solvents which can be used to make the reaction mixtures more fluid are solvents which do not adversely affect the reaction between epichlorohydrin and amine under the reaction conditions. Preferably the solvent is either water soluble or can be removed readily from the reaction mixture. Examples include:

(1) Saturated hydrocarbons containing from about 4 to about 8 carbon atoms, e.g., saturated aliphatic alkyl, aryl, or alkyl aryl groups. Specific examples include hexane, heptane, pentane, benzene, toluene and xylene.

(2) Short chain saturated aliphatic alcohols containing from one to about 8 carbon atoms and from one to about 3 hydroxyl groups. Specific examples include methyl, ethyl, propyl, isopropyl, butyl, and isobutyl alcohols; propylene glycol; and glycerine.

(3) Short chain aliphatic ketones containing from about 3 to about 8 carbon atoms, e.g., acetone, methyl butyl ketone, methyl isobutyl ketone and methyl isobutyl ketone and methyl ethyl ketone.

Choice of a particular solvent is normally dependent upon its water solubility or the ease with which it is

removed from the reaction mixture, and economic considerations. Isopropyl alcohol is a preferred solvent.

The solvent and the reaction mixture are desirably free of moisture since water reacts readily with epichlorohydrin and can interfere with the reaction.

The neutralization of the reaction product to an acid value of less than about 30 is very important. If the reaction product is allowed to remain at the acid value which normally results, e.g., an acid value of about 50 or higher, the treated textile material as hereinafter described tends to discolor unacceptably. Care must be taken not to lower the acid value of the reaction product too close to 0 since the resulting neutralized reaction product is not easily dispersed in water. It is believed that this occurs because of the formation of epoxy groups. As stated hereinbefore, an acid value of from about 5 to about 15 is preferred.

The neutralization of the reaction product to the proper acid value should be done under very mild conditions, i.e., a temperature of from about 60° F. to about 150° F., and vigorous agitation during caustic addition. Failure to observe this limitation will result in the formation of epoxy groups, and it has been found that epoxy groups are not desirable in the reaction product. The reaction product is not attached to the textile material as easily or as completely and the reaction product is not as easily dispersible in water when epoxy groups are present in the reaction product.

The structure of this reaction product is not known. The reaction product appears to be a relatively complex mixture. Similar reaction products which have been disclosed in the prior art, have contained epoxy groups, but it has been shown that epoxy groups adversely affect the performance of the reaction product of this invention.

The above reaction product can be used to prepare a variety of compositions which have utility in the treatment of textile materials. These compositions, which are suitable for imparting durable effects to textile materials, consist essentially of (A) from about 0.01% to about 100% of a reaction product of primary alkyl amine having an alkyl chain of from about 12 to about 30 carbon atoms and from about 1.25 to about 2 moles of epichlorohydrin per mole of amine, said reaction product being prepared at a temperature of from about 125° F. to about 200° F., and a reaction time of from about 30 minutes to about 10 hours, and said reaction product being neutralized to an acid value of less than about 30, the neutralization being carried out at a temperature of from about 60° F. to about 150° F.; (B) as an emulsifier for (A), from about 0% to about 10% by weight of (A) of a nonionic surfactant having a hydrophobic group selected from the group consisting of alkyl chains containing from about 9 to about 18 carbon atoms, and alkyl phenyl groups having alkyl chains containing from about 9 to about 15 carbon atoms and containing from about 10 to about 60 ethylene oxide moieties per molecule of surfactant; (C) from 0% to about 10% water soluble organic solvent, and (D) the balance water.

A preferred composition contains from about 10% to about 35% of the reaction product, and from about 2% to about 5% of the nonionic surfactant based on the weight of the reaction product, and from 0% to about 10% organic water soluble organic solvent (e.g., alcohols of the type described hereinbefore as solvents for the reaction mixture), and the composition is in the form of a dispersion of the reaction product in water. This preferred composition has several advantages. In the first place, this preferred composition, being already an aqueous dispersion, can be used quite readily to prepare more dilute dispersions suitable for application to textile material. Also, since the dust of the dry reaction product can be a health hazard, the aqueous dispersion is much safer to handle.

Another preferred composition is a more dilute composition containing from about 0.01% to about 8% of the reaction product, preferably from about .5% to about 2% of the reaction product. This dilute composition is the one which would be applied to the textile material. These dilute dispersions can be prepared from the solid reaction product using water at a temperature of from about 140° F. to about 170° F. and stirring for less than about one-half hour, to form a relatively concentrated aqueous dispersion containing from about 2% up to about 10% of the reaction product. This relatively concentrated aqueous dispersion is then diluted at a temperature of from about 80° F. to about 120° F. as required to provide the desired concentration.

When the preferred composition described hereinbefore (which contains from about 10% to about 35% of the reaction product) is used to form the dilute compositions, the normal procedure is to add the composition to 80° F. to 120° F. water with stirring to give the desired concentration.

Any alkaline material which is added to the dispersion for use as a catalyst as hereinafter described is normally predissolved and added with the water used in the final dilution step.

The concentration of the reaction product in any of the above compositions depends primarily upon the purpose for which the composition is intended. The more concentrated products are most suitable for shipping and the more dilute products are most convenient for preparing the eventual composition which is applied to the textile material. The concentration of the reaction product in the composition which is applied to the textile material will depend upon the purpose for which the reaction product is being applied, as will hereinafter be discussed in more detail, and upon the method of application, which will hereinafter be discussed in more detail.

The amount of nonionic surfactant which is employed in the preferred compositions depends upon the concentration of the reaction product and the presence of other materials. For example, when hard water is used to form the dilute compositions used to apply the reaction product to the textile material, or when an alkaline catalyst is required, more nonionic surfactant is employed in the preferred compositions. Also, when other materials, such as other textile treatment materials, are present, more nonionic surfactant is required to stabilize the preferred dispersion. However, an excess of nonionic surfactant (about 20%) will adversely affect the performance (e.g., softening and improvement of fiber-to-fiber lubricity) of the reaction product and accordingly, no more than about 10% nonionic surfactant should be used.

Specific examples of nonionic surfactants include the condensation products resulting from the condensation of coconut and/or tallow alcohols and/or nonyl, dodecyl, and/or tetradecyl phenol with, e.g., 10, 20, 30, 40, or 50 moles of ethylene oxide. Other nonionic emulsifiers can be used if desired as can mixtures of emulsifiers. The textile material to which the above reaction product is applied can be either filament or staple fibers. It can be raw stock which is to be formed into, e.g., thread or yarn; thread or yarn itself; fabric which is knitted or woven from thread or yarn; and/or fabric which is formed by bonding or other processes employing non-woven techniques.

The textile material can be natural, e.g., cotton, linen (flax) or wool. The preferred natural fibers are cellulosic in nature. Blends of these natural fibers with synthetic fibers are also contemplated.

The textile material also comprises synthetic fibers, for example, rayon, nylon, cellulose esters and chemically-modified cottons such as those treated cottons which have been crosslinked, using, e.g., cyclic ethylene urea and an acidic catalyst. Rayon and nylon are defined in the 1964 Man-Made-Fiber Chart published by Textile World and in the booklet, Textile Fibers and Their Properties, a 1965 publication of Burlington Industries, Inc.

A particular advantage of this invention is the improved stretchability which the reaction product can impart to

knit and stretch fabrics as discussed hereinafter more fully. Stretch fabrics are characterized by their ability to be stretched beyond their original dimensions and then quickly recover to, or almost to, their original dimensions when released. There are both woven and knit stretch fabrics. A woven fabric is made by interlacing warp and filling yarns at right angles to each other. A knitted fabric is made by interlocking a series of loops from one or more yarns. In general, such fabrics may be classified as filling or warp knit. In the former, yarns generally run crosswise, and in the latter, lengthwise. More detailed information on stretch fabrics can be found in: The Textile Dictionary, Calloway; America's Fabrics, Summer 1964, No. 64, pp. X–XI; and America's Fabrics, Winter–Spring 1964, pp. X–XI.

Other fabrics which are also improved remarkably include napped fabrics and dyed fabrics as will be discussed hereinafter in more detail. Napped fabrics and napping are also defined in The Textile Dictionary, Calloway.

The reaction product described hereinbefore is applied to the textile material from an aqueous milieu. There are many advantages of using water as a vehicle for applying the reaction product to textile material. In the first place, water is relatively inexpensive. Also, most businesses which which are engaged in the manufacture of textile materials have equipment for applying aqueous materials to textiles. Accordingly, the process of this invention comprises the steps of applying the reaction product dispersed in water to textile materials under conditions that will permit the textile material to take up from about .1% to about 15% by weight of the material of the reaction product, and thereafter removing the water and heating said textile material and said reaction product to a temperature of from about 80° F. to about 375° F., for a period of time from about 15 seconds to about 600 minutes and preferably no longer than is required to affix the reaction product to the textile material.

The above process can be carried out in many ways. In one way, the textile material is placed in an aqueous dispersion of the reaction product which contains as little as .01% dispersion of the reaction product and the textile material is allowed to remain in said dispersion until a sufficient amount of the reaction product is attached on the material. The textile material is then removed from the aqueous dispersion, excess dispersion removed, dried and subjected to the best treatment hereinbefore described.

The temperature of the aqueous dispersion is preferably from about 60° F. to about 80° F. for the first few minutes, but thereafter the temperature can be raised, i.e., up to about 200° F., although the temperature is preferably less than about 120° F. Exhaustion of the reaction product onto the textile material is sufficiently complete within about 15 minutes or less.

In another way, the textile material is passed through an aqueous dispersion of the reaction product, until the textile material absorbs a sufficient amount of the aqueous dispersion of the reaction product to provide the proper amount of reaction product and the textile material containing the absorbed aqueous dispersion is physically removed from the rest of the aqueous dispersion, the excess dispersion is removed by squeeze rolls, and the material is dried to complete the reaction.

Other methods include circulating the dispersion through the textile material (e.g., package application); spraying the dispersion onto the surface of the textile material; and applying the dispersion to the textile material by means of a kiss roll.

The length of time which the textile material is heated is directly related to the temperature. The higher temperatures should be applied only for very short periods of time since the reaction product is affixed to the textile material quite readily and excessive heating will tend to cause the treated material to yellow. The heating step can be combined with the drying step and this is the normal and preferred procedure.

In a preferred process, the pH of the aqueous dispersion of the reaction product, as applied to the textile material prior to heat treatment, is acidic, e.g., from about pH 3 to about pH 7. It is also possible to carry out the heat treatment when the pH of the aqueous dispersion of the reaction product is alkaline, e.g., from about pH 7 to about pH 13. The alkaline material, e.g., sodium or potassium hydroxides, carbonates, or bicarbonates are added at a temperature of from about 80° F. to about 120° F. immediately before use. The reaction product cannot be stored with alkaline material since this tends to form epoxy groups. Application of the reaction product to textiles under alkaline conditions is also sometimes desirable since the reaction proceeds most quickly under alkaline conditions. However, in general, it is preferred that this application be carried out under acidic conditions since the alkaline conditions tend to promote yellowing of the treated textile material. When the reaction product was applied to nylon, an alkaline catalyst was used.

It is preferred that the aqueous dispersions of the reaction product which are used to treat the textile material have a concentration of reaction product of from about 0.1% to about 8%, preferably from about 0.5% to about 2%.

It is undesirable to have some other textile chemicals present when the reaction product is being applied to the textile material; however, durable softness is still obtained in the presence of many textile chemicals. The reaction product acts as a cationic material and accordingly anionic materials and/or acid catalysts should be used with care in conjunction with the reaction product. The reaction product can be applied with some dyes, but it is preferred to apply the reaction product after the dye is affixed to the textile material. When other textile chemicals are present, a nonionic surfactant is required to maintain the stability of the dispersion of the reaction product.

Once the reaction product is attached to the textile material, the treated textile material can be handled safely without any health hazards.

The textile material that is treated with the reaction product will have different characteristics depending upon the type and amount of reaction product applied and the type of textile material to which the reaction material is applied. One can produce durable fabric softness, lubricity, body, firmness and/or water repellency.

If superior softening effects and improved fiber-to-fiber lubricity are desired, the alkyl chain of the reaction product should contain from about 16 to about 20 carbon atoms. Furthermore, for such characteristics, the amount of reaction product which is applied to the textile material should vary from about .5% to about 2%.

Higher levels of reaction product tend to give water repellency characteristics to the textile material. If water repellency is desired, reaction products having longer chain lengths should also be used, e.g., from about 18 to about 30 carbon atoms. For water repellency from about 3% to about 15% of the reaction product should be applied to the textile material.

Because the reaction product gives excellent fiber-to-fiber lubrication, cloth prepared from textile material which has been treated with the reaction product so as to give softening will also have an improved appearance when the cloth is washed and tumble-dried, i.e., there will be less wrinkle in the cloth.

This improvement in fiber-to-fiber lubricity is especially important with respect to knit and stretch fabrics, i.e., knitted fabrics and woven stretch fabrics.

When the reaction product of this invention is applied to knit and stretch fabrics, it requires less energy to stretch the fabrics and the fabrics recover to more nearly their original dimensions when they are released (elastic

recovery). This improvement in stretchability is manifested both before and after several cycles of usage and washing, with, e.g., a commercial heavy duty detergent product.

As stated hereinbefore, it is believed that the improvement in stretchability results from improved fiber-to-fiber lubricity which permits the fibers to move with respect to each other with the application of less force. It is believed that this improved fiber-to-fiber lubricity also (1) permits the fibers to recover their original alignment more easily and/or (2) diminishes the permanent deformation and/or damage to the individual fibers. It is believed that this accounts for the improved stretchability, improved elastic recovery and improved bursting strength observed in the fabrics treated with the reaction product of this invention as opposed to untreated fabrics or fabrics treated with a conventional non-permanent finish.

Improved fiber-to-fiber lubricity apparently is responsible for the excellent results obtained when the reaction product is applied to napped fabrics. There is less "matting" and "pilling" when these fabrics are treated with the reaction product of this invention. This means that after wearing and/or washing the fabric maintains more of its original uniformly-napped appearance. It is believed that the ability of the fibers to slip with respect to each other enables the fibers to separate when they become entangled.

The advantages which are obtained with knit, stretch and napped fabrics are so remarkable that reaction products having acid values from 0 to the acid value which naturally occurs from the reaction can be used for this purpose. Although the reaction product described hereinbefore is still preferred, it is recognized that the advantages in these special areas would outweigh the disadvantages attendant upon using reaction products outside the scope of the description of the preferred reaction product. This is also true with respect to colored fabrics, especially where the coloring will help mask any yellowing.

Corduroy and other pile fabrics also benefit from the application of the reaction product.

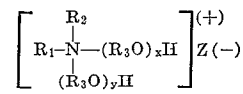
The reaction product increases the tear strength of the textile material to which it is applied. This is a very important factor in improving the acceptability of textiles.

The reaction product also improves the wet and dry crease angles of the textile material when it is in the form of cloth. This is very important with the newer fabrics which are designed to have wash-wear characteristics. It has been noted that this improved crease angle is apparent even with chemically modified (treated) cottons which have been cross linked. The reaction product improves the crease angle approximately 10° to 25° whether the reaction product is applied to textile materials which have, or have not, been cross linked.

It has been noticed that the reaction product, when applied to colored (dyed) textile material tends to prevent the dye from bleeding. This is a special advantage of the products and processes of this invention.

It is, of course, a primary advantage of this invention that the reaction product is attached so strongly to the textile material that the reaction product is not removed even after a large number, e.g., 10–20 cycles of home washing with a conventional heavy-duty built detergent. The improved characteristics which the reaction product gives to the textile material are accordingly maintained for a relatively long time.

An improved variation of this invention involves adding to the aqueous dispersion of the reaction product the following textile lubricant in an amount sufficient to give from about ⅛ to about 1½ parts of textile lubricant for each part of reaction product; said textile lubricant consisting essentially of a homogeneous water suspension of an oxidized Fischer-Tropsch wax having a chain length of from about 40 to about 55 carbon atoms, a melting point ranging from about 200° F. to about 220° F., an acid value ranging from about 10 to about 35 and a penetration ranging from about 1 to about 6, emulsified by an emulsifier selected from the group consisting of (a) a cationic quaternary ammonium compound having the formula:



wherein $R_1$ is an alkyl group containing 14 to 20 carbon atoms; $R_2$ is selected from the group consisting of alkyls containing 1 to 3 carbon atoms, phenyl, naphthyl, and $C_1$–$C_3$ alkyl substituted phenyls, $R_3$ is an alkylene group containing 2 to 4 carbon atoms, Z represents an anion, and $x$ plus $y$ ranges from 1 to 6 and $x$ ranges from 1 to 6, and (b) mixtures of said ammonium compound and the nonionic surfactant hereinbefore described wherein the nonionic surfactant content ranges from about 0 to 7 parts for every 3 parts of cationic emulsifier, and the ratio of oxidized Fischer-Tropsch wax to total of emulsifier and nonionic surfactant ranges from about 10:1 to 3:7.

A more complete description of the above textile lubricant can be found in the copending application of Arthur W. Lanner, Ser. No. 550,890, filed May 18, 1966.

The above textile lubricant is an especially valuable additive to the compositions of this invention where improved "sewability" is desired. The textile lubricant is also a very valuable additive where the reaction product is to be applied to a chemically modified (treated) cross-linked cotton fiber.

All parts, ratios, and percentages herein are by weight unless otherwise specified.

The following examples illustrate the practicality of this invention:

## EXAMPLE I

Tallow alkyl amine (1040 g.) and epichlorohydrin (556 g.), in a molar ratio of epichlorohydrin to amine of about 1.5:1, were reacted together at a temperature of about 125° F. (the temperature was kept below 150° F.) for a period of about six hours. 400 g. of the reaction product was then neutralized to an acid value of about 9 with 25 g. of 50% NaOH in 400 g. of 95% denatured ethyl alcohol. The temperature of the neutralization reaction was kept below about 100° F.

The above neutralized reaction product was mixed with the condensation product of an alcohol derived from a fraction of coconut oil fatty acids (2% $C_{10}$, 66% $C_{12}$, 23% $C_{14}$, and 9% $C_{16}$) and about 45 moles of ethylene oxide. The mixture contained about 97% of the neutralized reaction product and about 3% of the condensation product of the alcohol and the ethylene oxide. 40 g. of this mixture was dissolved in about 500 ml. of soft water to give an approximately 8% dispersion of the neutralized reaction product. This intermediate dispersion was then diluted with water at about room temperature (80° F.) to form the approximately 1% dispersion of the reaction product in the application bath. The NaOH which was added to show the effect of pH was added to the intermediate dispersion with the water of dilution.

White cotton crash toweling material was passed through the application bath which contained about 1% by weight of the reaction product. The toweling material picked up about 70% of its own weight of the application bath and the amount of the reaction product absorbed on the toweling material (cloth) was accordingly about .7% by weight of the cloth.

The cloth was then air dried and heated to about 260° F. for about 4 minutes.

The treated cloth was tested for "hand" by at least two qualified judges who compared the cloths on a

scale where 1 is very harsh, 10 is very soft, 2 is the value of the blank and 8 is the softness value achieved by a conventional softener, ditallowyldimethylammonium chloride, at a level of about 1% by weight of the cloth.

The results of the tests were as follows:

| | Blank (no softener) | Percent of NaOH in the application bath | | | | |
|---|---|---|---|---|---|---|
| | | 0.00 | 0.015 | 0.075 | 0.15 | 0.16 |
| pH of application bath | | 3.6 | 3.9 | | 11.3 | |
| Hand: | | | | | | |
| Before washing | 2 | 8 | 8 | 8 | 8 | 8. |
| After 5 "Tide" washes* | 2 | 6 | 6 | 8 | 8 | 8. |
| Color: | | | | | | |
| Before ageing | White | White | White | White | Very slightly yellow. | Very slightly yellow. |
| After ageing for 40 hrs. at 180° F. | do | do | do | Slightly yellow | Slightly yellow | Slightly yellow. |

*"Tide" is a commercially available, heavy duty laundry detergent composition. The washing concentration was at the recommended levels.

When, in the above example, the following alkaline salts are substituted, either wholly or in part, for the sodium hydroxide in the neutralization and/or in the application bath on an equivalent basis, substantially equivalent results are obtained in that the reaction product has the proper acid value and the reaction product is attached to the textile material; sodium and potassium carbonates, hydroxides, carbonates, and/or bicarbonates and their mixtures in, e.g., 1:1, 2:1, and 3:1 ratios.

When, in the above example, the following nonionic surfactants are substituted, either wholly or in part, for the condensation product of the alcohol and the ethylene oxide, substantially equivalent results are obtained in that the emulsion is stabilized and the reaction product is attached to the textile material: the condensation products of coconut and tallow alcohols with 20 or 60 moles of ethylene oxide; the condensation product of nonyl, dodecyl, or tridecyl phenol with 15, 25, 30, 45, or 60 moles of ethylene oxide; the condensation product of 2-hexadecanol with 40 moles of ethylene oxide; the condensation of dodecanol, tetradecanol, hexadecanol, or octadecanol with 20, 30, 45 or 60 moles of ethylene oxide, and mixtures of the above in, e.g., a 1:1 ratio.

## EXAMPLE II

Tallow alkyl amine (64.6 lbs.) was reacted with epichlorohydrin (35.4 lbs.) in a molar ratio of epichlorohydrin to amine of about 1.5. The reaction was carried out at a temperature of about 140° F. for a period of about 5 hours. The resulting product was neutralized to an acid value of about 7.5 by reacting the product with 6.7 pounds of 50% NaOH in 50 pounds of 95% ethyl alcohol at a temperature of about 100° F.

An approximately 3.2% dispersion, on a 100% active basis, of the above neutralized reaction product was made by dispersing the neutralized reaction product first in approximately 50 gallons of water at 160° F. in about 35 minutes with constant stirring and then diluting with cold water to the required final volume.

The cloth that was treated was of the following two types.

(a) Flat knit T-shirting material made from 22 single combed yarn, knit 16 cut (knitting needles/inch of machine width), and 32 stitches (length). This material contained Pontamine White BT as an optical whitener.

(b) Yellow, direct cotton dyed children's sleeper fabric, which was then napped before compacting.

The above cloths were passed through the 3.2% dispersion to pick up about 40% by weight of the fabric of the dispersion. This gave approximately 1.3% by weight of reaction product on the fabric. The (a) fabric was then dryed at about 310° F. for about 25 seconds to affix the reaction product to the fabric. The (b) fabric was dryed at about 250° F. for about 25 seconds to affix the reaction product to the fabric. After ten "Tide" washes both cloths were very much softer than similar cloth which had not been treated.

Cloth (a), in addition to being soft, also had improved stretchability, i.e., it required less energy to stretch the treated cloth and the cloth recovered more closely to its original dimensions after stretching. This cloth was tested against a similar cloth which had been treated with a conventional non-durable softener (hardened tallow monoglyceride). The cloth was stretched 33% and 66% in the width direction using 2-inch jaws to clamp the cloth which had a width of 4 inches. The jaws were initially 3 inches apart and were moved further apart at 12 inches/minute to achieve the desired stretch, i.e., the jaws were moved 1 inch for 33% stretch and 2 inches for 66% stretch.

The cloth was allowed one minute to recover its original dimension and the percent difference in dimensions was noted.

| | 33% stretch | | 66% stretch | |
|---|---|---|---|---|
| | Pounds to stretch | Percent unrecovered stretch after 1 min. | Pounds to stretch | Percent unrecovered stretch after 1 min. |
| Before washing: | | | | |
| Reaction product | 1.0 | 16 | 5.1 | 22 |
| Conventional softener | 1.1 | 19 | 5.0 | 27 |
| After 10 "Tide" washes: | | | | |
| Reaction product | 0.45 | 7 | 1.5 | 11 |
| Conventional softener | 1.1 | 12 | 4.2 | 20 |

This cloth was also tested for Mullen bursting strength using two thicknesses of cloth. The pounds per square inch required to burst the cloth treated with the reaction product was 182 whereas it only took 175 pounds per square inch to burst the cloth treated with the conventional softener. After 10 "Tide" washes the difference was even more remarkable. The reaction product treated cloth had a bursting strength of 182 pounds/inch$^2$ versus a bursting strength of only 167 pounds/inch$^2$ for the cloth treated with the conventional softener.

Cloth (b) also showed improved softness and stretchability. Cloth (b) also showed that its color was much brighter after 10 "Tide" washes when it was treated with the reaction product than when it was not treated. The reaction product apparently locked the dyestuff on to the fabric. Also, the napping, after 10 "Tide" washes, was much more even and showed less "matting" and "pilling" than a similar piece of cloth which had not been treated.

When, in the above examples, the following amines are substituted, either wholly or in part, for the tallow alkyl amine, on a mole-for-mole basis, in the preparation of the reaction products, substantially equivalent results are obtained in that the textile materials are sofened: dodecylcyclohexyl, hexapropylene, pentapropylene, 12-cyclohexyldodecyl, dodecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, eicosyl, docosyl, tricosyl, pentacosyl, hexacosyl, and octacosyl amines.

When, in the above examples, the following textile materials are substituted, either wholly or in part, for the cotton textile materials, substantially equivalent results are obtained in that these textile materials are also softened: nylon 6,6; nylon 6; viscose rayon; wool; fiber 40; fiber HM; fiber W–63; couprammonium rayon; cellulose acetate; cotton which has been crosslinked using cyclic ethylene urea and an acidic catalyst; linen; cellulose triacetate; ethylene glycol terephthalate polyesters; and, e.g., 1:1, 2:1 and 3:1 blends of the above textile materials.

When, in the above examples, a 1:1 mixture of eicosyl and docosyl amines is substituted, either wholly or in part, for the tallow alkyl amine and the reaction product is applied to the textile material at a level of about 5% by weight of the textile material, substantially equivalent results are obtained in that the reaction product is attached to the textile material. However, the textile material is waterproofed to a significant degree.

When, in the above examples, the dispersions of the reaction products are placed on the textile material by spraying, a kiss roll, and/or soaking (including, e.g., when different concentrations, 0.5% and/or 2% of the reaction product are used) substantially equivalent results are obtained in that the reaction products are attached to, and improve the characteristics of, the textile materials.

## EXAMPLE III

Sodium tetraphosphate, sodium hexametaphosphate and other phosphate systems having a nearly neutral pH, e.g., buffered phosphate systems, can be added in amounts from about 5% to about 20% by weight of the reaction product to the compositions and dilute dispersions of this invention to provide additional benefits, especially when optically whitened textile materials are treated. These phosphate additives improve the color of the finished treated textile materials. Lower amounts are not as effective and higher amounts begin to adversely affect lubricity.

Pontamine White BT, Calcofluor EDS, and Tinopal SV liquid are attached to white bleached cotton cloth by passing cloths through 0.13% solution of these optical whiteners to achieve a 70% pickup of the solutions and thereafter drying the cloths. These optically whitened cloths are then treated with the reaction product of tallow alkyl amine (64.6 lbs.) with epichlorohydrin (35.4 lbs.) in a molar ratio of epichlorohydrin to amine of about 1.5. (The reaction is at about 140° F. for a period of about 5 hours and the resulting product is neutralized at a temperature of about 120° F. to an acid value of about 12.5 with sodium hydroxide.) An approximately 1% dispersion of this reaction product is made with about 0.02% of a condensation product of coconut alcohol and about 45 moles of ethylene oxide. When this dispersion also contains 0.1% of sodium tetraphosphate; the cloth is allowed to pick up about 70% of its own weight of the dispersion; the cloth is held in a wet condition for about four hours; and the cloth is allowed to dry naturally, the cloth has a much better (whiter) color than when the sodium tetraphosphate is not present. Similar results, i.e., whiter cloths, are achieved when sodium hexametaphosphate is substituted for the sodium tetraphosphate.

What is claimed is:

**1.** A water dispersible reaction product of a primary alkyl amine having an alkyl chain of from about 12 to about 30 carbon atoms and from about 1.25 to about 2 moles of epichlorohydrin per mole of amine, said reaction product being prepared at a temperature of from about 80° F. to about 200° F., and a reaction time of from about 30 minutes to about 10 hours, and said reaction product being neutralized to an acid value of less than about 30, the neutralization being carried out at a temperature of from about 60° F. to about 150° F.

**2.** The reaction product of claim **1** having an epichlorohydrin to amine ratio of about 1.5:1.

**3.** The reaction product of claim **1** wherein the acid value of the reaction product is from about 5 to about 15.

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