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TEXTILE WETTING AND
SOFTENING BATH

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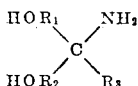
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Our present invention relates to a new group of chemical compounds, which our investigations have shown possess remarkably good properties for use as wetting, detergent, emulsifying, dye-levelling and textile softening agents. It also relates to a treating bath.

It is known that scouring materials can be prepared by condensing higher fatty acids with alkylol amines of the type of mono-, di-, tri-ethanolamines, diethyl ethanolamine, pentanol-amine, alkylolamines of glycerine, sugar and other polyhydric alcohols in various molecular proportions. Other amines also have been condensed with fatty acids or fatty acid derivatives to secure products having utility in the field of detergents and emulsification.

We have found that excellent cleaning, wetting and dispersing agents are obtained by condensing fatty acids or their functional equivalents or derivatives thereof with that type of amino methane which has one or two, much preferably two, alkylol groups attached directly to the methane carbon atom and one alkyl group directly attached to the methane carbon atom. Such compound can be represented by the type formula



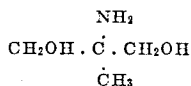
Where R_1OH is an alcoholic group, R_2OH is an alcohol group and R_3 is a hydrocarbon group. R_1 and R_2 can be the same or different and instead of R_1 , R_2 representing an alkyl nucleus, it can represent any other type straight chain or cyclic alcohol. R_3 can be substituted hydrocarbon and the hydrocarbon group can be any kind, preferably the alkyl. The amine group attached to the methane carbon can be a substituted amine. The term fatty acid includes the unsaturated as well as the saturated carboxylic acids having 7 or more carbon atoms, usually above 12. Examples of the kind of amino compounds we use are:

2 amino 2 methyl 1:3 propanediol

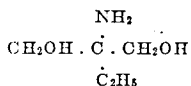
2 amino 2 ethyl 1:3 propanediol

Tris (hydroxy-methyl) amino methane

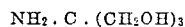
whose structural formulas are represented by



2-amino 2-methyl 1:3 propanediol



2-amino 2-ethyl 1:3 propanediol



Tris (hydroxy-methyl) amino methane

An object of the invention is to condense the above type amino methane compounds having an alcohol group, with fatty acids to form new compounds having excellent properties of the type indicated supra.

A second object is to effect the reaction with the lower molecular weight fatty acids, such as capric, undecylenic, lauric, myristic, or the coconut fatty acids whereby products are secured having very good wetting detergent and emulsifying qualities. It is an added object to improve and enhance their solubility and their technical properties by the sulfonation thereof.

It is another object of our invention to select and use for the condensation reaction instead of the lower molecular carboxylic acids, the higher molecular weight fatty acids such as stearic, palmitic, margaric, oleic and ricinoleic acids, whereby excellent textile softeners are obtained.

Still another object of our invention is to prepare new chemical substances which possess valuable technical properties in neutral, acid or alkaline media.

These objects are accomplished by reacting the fatty acids, functional equivalents or derivatives of these with the type of amino alcohols indicated in the formula and characterized above. The reactants are heated under such conditions as under reflux or in an open vessel at temperatures which can vary from slightly above 100°C ., to as high as 250°C . We have found that for practical purposes the optimum temperature range is between about 150°C . and 180°C . The condensation can be accelerated by a catalyst. Vacuum can be used, thereby proportionately lowering the temperature of the reaction and aiding in carrying off the by-products. Inert gaseous mediums could be used to supplement the heating step and effect a good agitation. The essential thing is not to exceed the temperature of decomposition of the resultant product or reacting substances; the aim is to speed along the reaction while maintaining conditions below the destruction range. The time required for completion of the reaction will vary widely depending on the combination of initial starting materials used, the size of the batch and several other physical or chemico-physical factors. At the lowest temperature ranges this may be as long as thirty hours, whereas at the highest temperatures the period may be as short as thirty minutes.

In addition to the fatty acids mentioned above, the naturally occurring fats and oils such as coconut oil, stearin, palmitin, tallow, castor oil, peanut oil, corn oil, the hydrogenated or other derivatives thereof, may be successfully reacted with these amino hydroxy-alkyl methane compounds.

Other esters of the fatty acids than the glycer-

ides may be used too. Various functional equivalents of the free fatty acids may be employed in fact.

The condensation of the fatty acids with these amino compounds can be effected easily. When these components are warmed they unite and become homogeneous promptly. However with the natural occurring glycerides as the fats, the reaction proceeds very slowly, and in fact in some cases it does not go at all unless a suitable catalyst is used. This fact is visibly evident during the condensation, for when no catalyst is present the amine and the fat form two liquid layers which either do not unite or do so only after many hours of heating. Upon the introduction of a small amount of a catalyst such as a strong, stable, and non-volatile alkali, the condensation proceeds at a much faster rate than otherwise. And in the case of those fats which previously failed to react at all, the condensation now proceeds smoothly and at a rate which approaches or is equal to normal. Examples of suitable catalysts are: sodium hydroxide, potassium hydroxide, sodium carbonate, potassium carbonate, phosphates of the alkali metals such as sodium pyrophosphate and trisodium phosphate, monoethanolamine, diethanolamine, triethanolamine. These may be employed in amounts of from $\frac{1}{4}\%$ to 5%, based upon the weight of the reacting ingredients. The list of basic substances supra is not exhaustive but illustrative.

It is also an object of our invention to increase the speed of the reaction between amino alcohols and fatty acids or their functional equivalents particularly the esters such as the glycerides by the use of strong alkaline catalysts indicated supra. Amino alcohols embrace not only the amino methanes which have one or two alkylol groups but also the alkylolamines such as mono- or diethanolamine.

The condensation products with the lower molecular weight fatty products and amino alcohols have valuable technical properties in themselves without further chemical treatment. However, their scope of usefulness is appreciably broadened by proper sulfation. In like manner the condensates of higher molecular weight fatty substances possess good technical properties, but on addition of various acids, such as acetic, formic, lactic, hydrochloric, etc., water soluble salts are formed which appreciably broaden the technical application of these condensates.

We have found that various molal ratios of these amino compounds to fatty acids may be used, and that the properties of the resulting condensation products which determine in turn the technical utilization thereof depends to a large degree upon the selected molal ratio. Ratios have been varied from somewhat less than 1 mol of amine: 1 mol of fatty acid to as high as 2 mols the amine or somewhat above: 1 mol fatty acid. For some applications a low molal ratio of amine to fatty acid (or fat) is better, while for others a high ratio is preferable. The optimum molal range can be readily arrived at and determined. By increasing the amine content of the product a more water dispersible and improved emulsifying agent results. By decreasing the amine content, a condensate results which is more fat-like, which is admirably adapted for use in textile softening agents. If the amine content is too low, the resulting condensation product will be too poorly soluble in water for practical use in the textile treatments. A person

can be governed by the type of technical product desired as to ratios to be selected. We have also determined that with the employment of an alkaline catalyst in the reaction the resulting condensation product possessed remarkably good emulsifying and detergent qualities, whereas in similar condensations omitting the alkaline catalyst such as the alkali these emulsifying and detergent qualities were either non-existent or much inferior to the corresponding end product where it was present during the reaction.

The following list of examples demonstrate the manner in which our invention may be practiced, and should by no means be considered as exhaustive nor as limiting the scope of our invention.

Example 1

One mol of coconut oil fatty acids is mixed with two mols of 2 amino 2 methyl-1:3 propanediol and heated three to six hours at 150 to 180° C. A viscous red oil is obtained which possesses valuable technical properties.

To 100 parts of H_2SO_4 (100%) is slowly added, with mixing and cooling, 100 parts of the fatty amine condense. The temperature is controlled preferably in the range of 10 to 40° C. throughout the entire procedure of sulfation, and subsequent dilution and neutralization. The sulfated mass is mixed until homogeneous, and then diluted with 60 or more parts of water, depending on the final concentration of active material desired. The diluted mass is now neutralized with an aqueous solution of an alkali such as NH_3 , KOH , NaOH , $(\text{C}_2\text{H}_4\text{OH})\text{NH}_2$, etc., to obtain the final end product. This is a valuable wetting and dye-levelling agent.

The sulfation procedure described is believed to result in the formation of a predominant quantity of the sulfuric esters in which the sulfur atom is joined to the compound by an oxygen linkage and possibly of a small amount of true sulphonic acids in which the sulfur atom is attached to the carbon atoms.

Example 2

One mol of coconut oil fatty acids is condensed with two mols of tris (hydroxy methyl)-amino methane for three to six hours at 150 to 180° C. A light brown wax is obtained which is readily dispersible in water and possesses interesting technical characteristics. By sulfating this wax in a manner similar to that used in Example 1, an excellent wetting, detergent, and dye-levelling agent is obtained.

Example 3

One mol of stearic acid of condensed three to six hours at 150 to 180° C. with $1\frac{1}{2}$ mols of 2 amino 2 ethyl 1:3 propanediol. A dark red oil is obtained which when converted into a salt of the amide by means of an acid such as acetic, formic, lactic, hydrochloric, etc., yields a very good textile softening agent.

Example 4

One mol of stearic acid is condensed for three to six hours at 150 to 180° C. with $1\frac{1}{2}$ mols of 2 amino 2 methyl 1:3 propanediol. A light yellow wax is obtained which when converted into the salt of the amide by means of an acid such as acetic, formic, lactic, hydrochloric, etc., yields an excellent textile softening agent.

Example 5

One mol of stearic acid is condensed with

1½ mols of tris (hydroxy methyl) amino methane for three to six hours at 150 to 180° C. A water dispersible light brown wax is obtained which possesses excellent technical properties. Conversion to the salt with an acid, as mentioned in Example 4, yields a product having remarkably good textile softening powers.

Example 6

One mol of stearic acid is condensed with 2 mols of 2 amino 2 methyl 1:3 propanediol for three to six hours at 150 to 180° C. A light yellow wax is obtained which is dispersible in water, and which yields very good textile softening agents when converted into the salt of the amide by means of any of the acids mentioned in Example 4.

Example 7

One mol of stearic acid is condensed as shown in previous examples with two mols of tris (hydroxy methyl) amino methane for three to six hours at 150 to 180° C. A light yellowish brown wax resulted which had excellent technical properties, and which upon treatment with one of the acids mentioned in Example 4 yielded a very good textile softener.

Example 8

One mol of oleic acid is condensed with 1½ mols of 2 amino 2 methyl 1:3 propanediol for three to six hours at 150 to 180° C. A clear reddish oil is obtained, which yields a good textile softener when acidified with one of the acids mentioned in Example 4.

Example 9

668 parts of coconut oil is reacted with 726 parts of tris (hydroxy methyl) amino methane and 28 parts of an aqueous 50% potassium hydroxide solution at 150 to 180° C. for two to six hours. A light yellowish brown wax results, which in itself has very good technical properties, and which upon sulfating with an equal weight of 100% sulfuric acid, and subsequent neutralization yields an exceptionally high quality wetting, detergent, and dye-leveling agent.

Example 10

884 parts of olive oil is condensed with 420 parts of 2 amino 2 methyl 1:3 propanediol and 26 parts of an aqueous 50% sodium hydroxide solution for two to six hours at 150 to 180° C. A light brown grease is obtained, which upon solubilizing by acidification with one of the acids mentioned in Example 4 yields a good textile softening agent.

When an alkali catalyst is employed as in this example, the condensate has very good emulsifying and detergent properties. When this condensation is carried out in the absence of this catalyst, the resulting condensates have no emulsifying or detergent qualities.

Example 11

890 parts of tallow is heated with 420 parts of 2 amino 2 methyl 1:3 propanediol for three to ten hours at 150 to 180° C. A light brown grease is obtained, which yields an excellent textile softening agent when rendered water soluble by treatment with one of the acids mentioned in Example 4.

Example 12

807 parts of palm oil are condensed for three to ten hours at 130 to 170° C. with 476 parts

of 2 amino 2 ethyl 1:3 propanediol. A dark red oil is obtained which upon solubilizing with one of the acids mentioned in Example 4 yields a very readily soluble product giving perfectly clear water solutions, and possessing good properties as a textile softener.

Example 13

210 parts of lanolin are heated with 105 parts of 2 amino 2 methyl 1:3 propanediol for three to ten hours at 130 to 170° C. A brown grease is obtained which when treated with one of the acids mentioned in Example 4 yields a good textile softening agent.

Example 14

932 parts of castor oil is condensed with 420 parts of 2 amino 2 methyl 1:3 propanediol for three to ten hours at 150 to 180° C. A clear light yellow oil is obtained which yields a very good textile softener on treatment with one of the acids mentioned in Example 4.

Example 15

One mol of capric acid is heated with two mols of tris (hydroxy methyl) amino methane for three to six hours at 150 to 180° C. The condensate is a light brown wax having valuable technical properties in itself. However, by sulfating same with an equal weight of 100% sulfuric acid, and neutralizing the sulfate product, there is obtained an excellent agent for use as a detergent, wetting or dye-leveling purposes.

Example 16

668 parts of coconut oil is condensed with 201 parts of monoethanolamine and 18 parts of an aqueous 50% potassium hydroxide solution for two to six hours, at 150 to 180° C. A light yellowish brown hard wax is obtained which upon sulfating and subsequently neutralizing yields an excellent detergent, wetting, and dye-leveling agent.

Example 17

807 parts of palm oil are condensed for two to six hours at 130 to 170° C. with 420 parts of 2 amino 2 methyl 1:3 propanediol and 24 parts of a 50% aqueous potassium hydroxide solution. A soft brown wax is obtained which upon solubilizing with one of the acids mentioned in Example 4 yields a readily soluble product possessing good properties as a textile softener.

When an alkali catalyst is employed as in this example, the condensate has very good emulsifying and detergent properties. When this condensation is carried out in the absence of this catalyst, the resulting condensates have no emulsifying or detergent qualities.

Example 18

668 parts of coconut oil is condensed with 636 parts of diethanolamine and 26 parts of an aqueous 50% potassium hydroxide solution for two to six hours at 150 to 180° C. A light orange colored viscous oil is obtained possessing valuable characteristics as a detergent and wetting agent. By sulfating this oil with an equal weight of 100% sulfuric acid, and subsequently neutralizing, there is obtained a very good wetting, detergent and dye-leveling agent.

The condensate obtained when the alkali catalyst is not employed has good emulsifying and detergent qualities, but by using the catalyst a condensate having far superior emulsifying and detergent properties is obtained.

In preparing the aqueous treating baths for the treatment of textile fabrics, a small amount of the products is added. A 0.25% aqueous solution of a 20% aqueous paste of the sulfated condensate produced by the method in Example 2 is employed for scouring wool, cotton, silk or rayon fabrics. As little as a 0.05% aqueous solution of the non-sulfated fatty amide condensate of Example 18 is sufficient to yield an excellent detergent action on wool, cotton, silk or rayon.

An aqueous solution containing 0.05% to 0.5% by weight of a 30% aqueous acid paste of the condensate obtained according to Example 4 marked improves the appearance of textile fabrics composed wholly or in part of rayon, silk, cotton or wool. The goods following the operations of wetting the fabric and drying are remarkably soft and smooth to the touch. The same is true in operating with the other softeners described herein.

From the above examples, it is obvious that we may synthesize many different types of textile treating substances. Mixtures of the fatty acids or their functional equivalents such as the fats and other derivatives, and/or mixtures of the amines can be used, yielding products with somewhat different properties from those in the specific examples, yet valuable in the fields set out herein. Not a lone compound may result from the condensation, and this is particularly true if mixtures of retactants are employed.

We do not intend to limit ourselves to the specific embodiments except as indicated in the claims, since these embodiments and examples serve the purpose of clarity and preference. Where the term fatty acid is used in the claims it is intended to embrace the acids per se and their functional equivalents and that the term fatty acid includes the unsaturated and saturated carboxylic acids of not less than 6 carbon atoms.

A divisional application to Weisberg & Corman, Serial Number 505,996, filed October 12, 1943, is filed covering the subject matter of original claims required to be divided out of this application.

We claim as our invention:

1. A textile wetting bath comprising an aqueous solution of a small amount of an amide condensation product of saturated fatty acids, containing at least seven carbon atoms with tris (hydroxy-lower alkyl) amino methane.

2. A textile wetting bath comprising an aqueous solution of a small amount of an amide con-

densation product of saturated fatty acids, containing at least seven carbon atoms with tris (hydroxy-methyl) amino methane.

3. A textile wetting bath comprising the aqueous solution of a small amount of an amide condensation product of coconut oil fatty acids with tris (hydroxy-methyl) amino methane.

4. A textile wetting bath comprising an aqueous solution of a small amount of an amide condensation product of a saturated fatty acid, containing at least seven carbon atoms with an amino methane which has at least two alkylol groups of not over three carbon atoms each directly attached to the methane carbon atom wherein the minimum molar ratio of the amine to the fatty acid is 1.2:1 and the maximum about 2:1.

5. A textile wetting bath comprising an aqueous solution of a small amount of an amide condensation product of stearic acid with 2-amino 2-ethyl 1:3 propanediol wherein the minimum molar ratio of the amine to the fatty acid is 1.2:1 and the maximum about 2:1.

6. A textile wetting bath comprising an aqueous solution of a small amount of an amide condensation product of stearic acid with 2-amino 2-methyl 1:3 propanediol, wherein the minimum molar ratio of the amine to the fatty acid is 1.2:1 and the maximum about 2:1.

7. A textile softening bath comprising an aqueous solution of a small amount of a salt of an amide condensation product of fatty acids, containing at least seven carbon atoms with tris (hydroxy-lower alkyl) amino methane.

8. A textile softening bath comprising an aqueous solution of a small amount of a salt of an amide condensation product of a saturated fatty acid containing at least seven carbon atoms with an amino methane which has at least two alkylol groups of not over three carbon atoms each directly attached to the methane carbon atom wherein the minimum molar ratio of the amine to the fatty acid is 1.2:1 and the maximum about 2:1.

9. A textile softening bath comprising an aqueous solution of a small amount of a salt of an amide condensation product of stearic acid with tris (hydroxy-methyl) amino methane each of the carbon atoms of the hydroxy methyl groups being directly attached to the methane carbon wherein the minimum molar ratio of the amine to the fatty acid is 1.2:1 and the maximum about 2:1.

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