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FATTY ACID POLYGLYCOL-ALIPHATIC
AMINE COMBINATIONS USEFUL AS
TEXTILE SOFTENERS AND PROCESS
FOR PRODUCING THE SAMEAlton A. Cook, Glen Ridge, and Ira Sapers, New-
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9 Claims. (Cl. 252—8.75)

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The present invention relates to textile processing chemicals and methods of making the same, and it particularly relates to fatty acid-polyethylene glycol-aliphatic amine combinations having particular utility as textile softening and finishing agents and having cationic characteristics.

It is among the objects of the present invention to provide novel fatty acid-polyethylene glycol-aliphatic amine combinations useful as finishing and softening agents for textile fabrics.

Still further objects and advantages will appear in the more detailed description set forth below, it being understood, however, that this more detailed description is given by way of illustration and explanation only and not by way of limitation, since various changes may be made therein by those skilled in the art without departing from the scope and spirit of the present invention.

In accomplishing the above objects, it has been found most satisfactory to combine a fatty monocarboxylic acid having 12 to 24 carbon atoms first with a polyethylene glycol of an average molecular weight ranging from 400 to about 1500; then the resulting product is combined with a water-soluble primary or secondary aliphatic amine having at least two amino groups or one amino group and one or more hydroxyl groups.

The first step is preferably carried out at a temperature of 100° C. to 140° C., and desirably at 110° C. to 125° C., for 1 to 4 hours, while the second step is carried out at a temperature of 25° C. to 50° C. or higher, as for example at 140° C. to 180° C., and desirably 150° C. to 165° C., for about one to three hours.

In the first step, 1 mol of fatty acid is combined with the polyethylene glycol in an amount ranging from $\frac{1}{4}$ to $\frac{1}{2}$ of a mol, depending upon the molecular weight of the glycol which may range from 400 to about 1500 or over, in the presence of an acid catalyst, such as an alkylated naphthalene sulfonic acid.

At the end of this step, the polyethylene glycol has become practically completely esterified to form a di-ester with the fatty acid.

In the second step, the amine, preferably a primary or secondary water-soluble alkanolamine, should be present in an amount ranging from $\frac{1}{2}$ of a mol to $1\frac{1}{2}$ mols and desirably from $\frac{3}{4}$ of a mol to $1\frac{1}{4}$ mols for each mol of fatty acid. The second step of the reaction is normally carried out at the preferred range of temperature until the acid number of the mixture ranges from 5 to 20.

The final products are highly complex mixtures

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and include esters, amides and amino-esters. They are usually soluble or dispersible in water and are alkaline in reaction, a 1% solution having a pH of 8 to 9.

If desired, these products may be neutralized or acidified with organic acids, such as acetic, formic or lactic acids, for the purpose of producing finishes having definite substantivity to textile fibers.

They may also be alkylated with dimethyl or diethyl sulfate preferably in alkaline media at about 100° C. to produce more soluble compounds which also act as substantive finishes for textile fibers.

It has been found in the present invention that where these two types of reactions are carried out by a definite succession of steps, as described above, by first combining the fatty acid and polyethylene glycol and then the aliphatic amine, products are obtained which have properties which would not be expected from a physical mixture of an amino fatty acid condensation product and a polyethylene glycol-fatty acid ester as is ordinarily known.

The ingredients used in this invention fall into three classes of compound and at least one compound of each of the following classes is necessary to the manufacturer of the products described herein.

Class 1.—Water-soluble aliphatic saturated polyalcohol having at least four carbon atoms and at least 2 hydroxyl groups and a melting point preferably under 100° C.

Examples.—Polyethylene glycols of various average molecular weights from 200 to 1500 and polypropylene glycols having molecular weights from 400 to 1200. Alkoxy derivatives, such as methoxy- or ethoxy- polyethylene glycols having molecular weights of 350 to 750 or over may be employed.

Class 2.—Mono-carboxylic fatty acids having from 16 to 26 carbon atoms. The saturated fatty acids C_{16} to C_{26} are preferred. This includes mixed fatty acids obtained from splitting natural fats and waxes.

Example.—Palmitic and stearic acids are preferred. Erucic, montanic, arachidic, behenic, lignoceric and cerotic and mixed fatty acids from tallow, Japan wax and hydrogenated oils (averaging about 20 carbon atoms per molecule) may also be used.

Class 3.—Water-soluble aliphatic saturated amines with two or more amino groups or one amino group and one or more hydroxyl groups, excluding tertiary amines.

Examples.—Mono- and di-ethanolamines;

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mono- and di-propanolamines; butyl-mono- and di-ethanolamine; 2-methyl, 2-amino, 1-propanol; and aminoethylethanolamine. Less preferred are polyalkylene polyamines such as ethylene diamine; diethylene triamine; triethylene tetramine; tetraethylene pentamine.

The following examples illustrate the procedure of preparing the two-step complex condensation products:

Example I

As the first step, 284 parts of stearic acid and 150 parts of polyethylene glycol having a molecular weight of about 600 are heated in the presence of 1 part of butyl naphthalene sulfonic acid at 110° C. to 120° C. for 2 hours or until the polyethylene glycol is completely used up to form a di-ester.

Then in the second step 130 parts by weight of diethanolamine is added and the mixture is heated up to 160° C. for 2 hours until an acid number of about 10 has been achieved.

Example II

	Parts	Mols
First Step:		
Hydrogenated fatty acid (average molecular weight 310) 20 carbon atoms.....	310	1
Polyethylene glycol having a molecular weight of about 1000.....	200	1/2
Monobutyl naphthalene sulfonic acid.....	2.5	
Second Step:		
Diethanolamine.....	115	1.1

Example III

	Parts	Mols
First Step:		
Palmitic acid.....	256	1
Polyethylene glycol having a molecular weight of about 400.....	100	1/4
Sulfuric acid.....	0.2	
Second Step:		
Diethanolamine.....	53	1/2
Monoehtanolamine.....	30	1/2

Example IV

	Parts	Mols
First Step:		
Stearic Acid.....	284	1
Polyethylene glycol having a molecular weight of about 1500.....	300	1/2
Mixed alkane sulfonic acids.....	2.5	
Second Step:		
Aminoethylethanolamine.....	105	1

Example V

	Parts	Mols
First Step:		
Fatty acids from Japan wax.....	265	1
Methoxypolyethylene glycol having a molecular weight of about 550.....	275	1/2
	0.2	
Second Step:		
Diethylene triamine.....	75	1/4

The products made by the processes of Examples I to V are usually soft solids of a waxy consistency and are diluted with water either as produced or after acidification for actual use as textile finishes. In consistency these diluted products are soft to stiff pastes which disperse readily in water at any pH adjustment between 4.0 and 10.0. In concentration these preparations may vary from 10% up to 50% or more active ingredients.

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When used as softeners or finishing agents on woolen, rayon or cotton goods, these compounds in their diluted or acidified form as set forth in Examples VI and VII are employed in amounts of from 1% to 10% on the weight of the material being processed and in aqueous concentrations of from 0.1% up to about 2%. At a pH range of from 4.0 to 6.0 these finishes are markedly substantive and exhaust from the finishing bath like dyestuffs, while at higher pH ranges they are not as highly substantive, but do not have as adverse effects on the light fastness of the dyestuffs as the acidified products.

The following are examples of preparation of the actual finishing compounds from some condensation products of Examples I to V:

Example VI

33 parts of the product of Example I are heated to molten condition and poured with continual stirring into 65 parts of water containing 2 parts of formic acid. The stirring is continued until it forms a thick whitish paste, which is readily soluble in warm water.

Example VII

23 parts of the product of Example III in molten condition are mixed thoroughly with 75 parts of water containing 2% of acetic acid. The stirring is continued until a paste is formed which is readily dispersible in hot water.

The present application is a continuation in part of application Serial No. 747,598 filed May 12, 1947, Patent No. 2,491,478 dated December 20, 1949.

As many changes could be made in the above fatty acid-polyglycol-aliphatic amine combinations useful as textile softeners and process for producing the same, and many widely different embodiments of this invention could be made without departing from the scope of the claims, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

Having now particularly described and ascertained the nature of the invention, and in what manner the same is to be performed, what is claimed is:

1. A process of preparing textile softeners which comprises heating a high molecular weight saturated fatty acid, said fatty acid having 16 to 26 carbon atoms, with a polyalkylene glycol at 100° C. to 140° C. for about 1 to 4 hours, and then heating the reaction mixture with a water soluble aliphatic amine at 140° C. to 180° C. for 1 to 3 hours, said amine being selected from the group consisting of the water soluble primary and secondary alkanolamines and the water soluble polyalkylene polyamines.

2. A process of preparing textile softeners which comprises heating a high molecular weight saturated fatty acid, said fatty acid having 16 to 26 carbon atoms, with a polyalkylene glycol at 100° C. to 140° C. until the polyalkylene glycol has been completely esterified to form a di-ester and then heating the reaction mixture with a water soluble aliphatic amine at 140° C. to 180° C. until the acid number is 5 to 10, said amine being selected from the group consisting of the water soluble primary and secondary alkanolamines and the water soluble polyalkylene polyamines.

3. A process of preparing textile softeners which comprises heating about 1 mol of a high molecular weight saturated fatty acid, said fatty

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acid having 16 to 20 carbon atoms, with about $\frac{1}{20}$ to $\frac{1}{2}$ mol of a polyalkylene glycol at 100° C. to 140° C. for about 1 to 4 hours and until the polyalkylene glycol has been completely esterified to form a di-ester in the presence of an acid catalyst, and then heating the reaction mixture with $\frac{1}{4}$ to $1\frac{1}{4}$ mols of a water soluble aliphatic amine at 140° C. to 180° C. for 1 to 3 hours and until the acid number is 5 to 10, said amine being selected from the group consisting of the water soluble primary and secondary alkanolamines and the water soluble polyalkylene polyamines.

4. A process of preparing textile softeners which comprises heating about 1 mol of a high molecular weight saturated fatty acid, said fatty acid having 16 to 20 carbon atoms with about $\frac{1}{20}$ to $\frac{1}{2}$ mol of a polyethylene glycol 400 to 1600 at 110° C. to 125° C. for about 1 to 4 hours to form a di-ester in the presence of an acid catalyst, and then heating the reaction mixture with $\frac{1}{4}$ to $1\frac{1}{4}$ mols of a water soluble alkanolamine at 150° C. to 165° C. for 1 to 3 hours until the acid number is 5 to 10, said amine being selected from the group consisting of the water soluble primary and secondary alkanolamines and the water soluble polyalkylene polyamines.

5. A process of preparing textile softeners which comprises heating about 1 mol of a high molecular weight saturated fatty acid, said fatty acid having 16 to 20 carbon atoms, with about $\frac{1}{20}$ to $\frac{1}{2}$ mol of a polyethylene glycol 400 to 1600 at 110° C. to 125° C. for about 1 to 4 hours to form a di-ester in the presence of an acid catalyst, and

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then heating the reaction mixture with $\frac{1}{4}$ to $1\frac{1}{4}$ mols of a water soluble alkanolamine at 150° C. to 165° C. for 1 to 3 hours until the acid number is 5 to 10 and then acidifying with a water soluble organic acid, said amine being selected from the group consisting of the water soluble primary and secondary alkanolamines and the water soluble polyalkylene polyamines.

6. A textile softener comprising high molecular weight fatty acid-polyalkylene glycol-water soluble aliphatic amine combination produced according to claim 1.

7. A textile softener comprising the acidified aqueous dispersion of high molecular weight fatty acid-polyalkylene glycol-water soluble aliphatic amine combination produced according to claim 1.

8. A textile softener prepared specifically according to the process of claim 4.

9. A textile softener produced specifically according to the process of claim 5.

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