Patented Apr. 22, 1952

2,593,413

UNITED STATES PATENT OFFICE

2,593,413

FATTY ACID-ALKOXYPOLYGLYCOL ALIPHATIC AMINE COMBINATIONS USEFUL AS TEXTILE SURFACE AGENTS

Alton A. Cook, Glen Ridge, and Ira Sapers, Newark, N. J., assignors to Arkansas Company, Inc., Newark, N. J., a corporation of New York

No Drawing. Application December 20, 1949, Serial No. 134,150

10 Claims. (Cl. 260—404)

1

The present invention relates to surface active agents and process for manufacturing the same, and it particularly relates to such agents having particular utility as wetting agents, detergents and emulsifying agents in the textile assistant field.

It is among the objects of the present invention to provide a novel procedure and novel combinations by such procedure which at low cost will give a high yield of unusually effective detergent, wetting and emulsifying agents particularly useful in the textile field, but also useful in the leather, metal working and other allied fields.

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 the preferred procedure, a high molecular weight fatty acid having 8 to 24 carbon atoms is combined at a temperature below 135° C. and desirably between 100° C.–130° C., first with an alkoxypolyglycol at 100° C.–120° C., and then the combination of fatty acid and alkoxypolyglycol is combined with a primary or a secondary aliphatic amine, preferably either a water soluble alkanolamine or a water soluble polyalkylene polyamine The second step is desirably carried out at a temperature of 10° to 150° C. higher than the temperature employed in the first step, as for example 115° to 130° C.

The resulting product is a highly complex mixture of alkoxypolyglycol esters and amino esters. This complex may be neutralized or acidified, if desired, with lower aliphatic acids, such as formic, acetic or lactic.

The complex may also be treated with alkylating agents, such as diethyl or dimethyl sulfate in alkaline media up to 100° C. with or without the presence of solvents

It has been found in the present invention that where these two types of reactions are carried out by a definite succession of steps under controlled conditions of temperature, products are obtained which have properties which would not be expected from a physical mixture of an alkoxypolyglycol—fatty acid ester and an amino on one hand—fatty acid condensation product on the other hand

The ingredients used in this invention fall into three classes of compounds and at least one compound of each class is necessary for the manufacture of the products described herein.

2

Class 1.—Fatty acids having from 8 to 24 carbon atoms. The saturated fatty acids C_{10} to C_{18} (namely, capric, lauric, myristic, palmitic and stearic) are preferred, but other fatty acids including unsaturated and hydroxylated types may be used. Mixed fatty acids obtained from splitting natural oils and fats, such as coconut oil and palm kernel oil, may also be employed.

Class 2.—Water-soluble aliphatic saturated alkoxypolyglycols having over four carbon atoms, one hydroxy group and a molecular weight of 200 to 1,000 The alkoxy group may have from one to four carbon atoms.

Examples:

Methoxy polyethylene glycol having an average molecular weight of 350.
Methoxy polyethylene glycol having an average molecular weight of 550.
Methoxy polyethylene glycol having an average molecular weight of 750.
Ethoxy polyethylene glycol having an average molecular weight of 560.
Propoxy polyethylene glycol having an average molecular weight of 380.

Class 3.—Water-soluble aliphatic saturated primary or secondary amines with two or more amino groups or one amino group and one or more hydroxy groups.

Examples:

Mono and diethanolamine
Mono and dipropanolamine
2-methyl, 2-amino, 1-propanol;
Aminoethylethanolamine
Triethylene tetramine; and
Tetraethylene pentamine.

In the preferred procedure for producing surface active agents, in the first step a fatty acid (one mol) and an alkoxypolyglycol ($\frac{1}{10}$ to $\frac{1}{2}$ mol) are heated together with an acid catalyst, such as butyl naphthalene sulfonic acid at a temperature of 110° C. to 120° C. for one-half to two hours, or until such time that all the alkoxypolyglycol has been reacted.

In the second step, an aliphatic amine ($\frac{1}{2}$ to $1\frac{1}{2}$ mols) is added slowly with agitation and the temperature is increased to the range of 120° to 130° C. The mixture is then held at this temperature for two to four hours, or until such time as the free fatty acid content of the mixture is from 10% to 20%. During this step, the mixture usually becomes alkaline due to the addition of

the amine. In some cases, the addition of a small amount of caustic soda or other alkali is desirable at the start to insure the neutralization of the acid catalyst used in the first step and to impart a definite degree of alkalinity in the second step. In general, if a higher proportion of the alkoxypolyglycol is used in the first step, a lower proportion of the amine may be used in the second step and vice versa.

To give some typical examples of the preferred combinations and procedure involving the reaction of the fatty acid and the alkoxypolyglycol in the first step followed by further reacting this product with a water soluble aliphatic saturated primary and secondary amine (all parts being by weight):

Example 1

200 parts (1 mol) of lauric acid and 175 parts (½ mol) of a methoxy polyethylene glycol having an average molecular weight of 350 are heated in the presence of an alkylated aromatic sulfonic acid (one to two parts by weight) at 110° to 120° C. for about one hour or until the alkoxypolyglycol has been completely reacted. Then the temperature is raised to 120° to 130° C. and 115 parts (1.1 mols) of diethanolamine are added slowly and the temperature maintained for about two hours or until the free fatty acid is reduced to about 10%. The resulting composition, when cooled to room temperature, consists of an amber viscous liquid which is useful as a wetting agent and detergent in textile wet processing operations.

In the following examples, the proportions are given below and the procedure in all cases is similar to that described above:

Example 2

First step:
- Myristic acid, 228 parts (1 mol)
- Methoxy polyethylene glycol having an average molecular weight of 550, 185 parts (approx. ⅓ of a mol)

Second step:
- Aminoethylethanolamine, 70 parts (⅔ of a mol)

Example 3

First step:
- Coconut fatty acids, 210 parts (approx. one mol)
- Ethoxy polyethylene glycol having an average molecular weight of 560, 280 parts (½ of a mol)

Second step:
- Diethanolamine, 35 parts (⅓ of a mol)
- Monoethanolamine, 61 parts (1 mol)

Example 4

First step:
- Capric acid, 172 parts (1 mol)
- Propoxy polyethylene glycol having an average molecular weight of 380, 195 parts (½ of a mol)

Second step:
- Triethylene tetramine, 73 parts (½ of a mol)

Example 5

First step:
- Palmitic acid, 256 parts (approx. 1 mol)
- Methoxy polyethylene glycol having an average molecular weight of 750, 200 parts (approx. ¼ of a mol)

Second step:
- Diethanolamine, 150 parts (approx. 1.4 mols)

Since certain changes may be made in the above textile assistants and process for manufacturing the same and different embodiments of the invention could be made without departing from the scope thereof, 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 making a surface active agent which comprises successively combining a high molecular weight fatty acid having 10 to 18 carbon atoms with an alkoxypolyglycol and with an aliphatic saturated amine selected from the group consisting of primary and secondary water-soluble aliphatic amines at a temperature under 135° C.

2. A process of making a surface active agent which comprises successively combining a high molecular weight fatty acid having 10 to 18 carbon atoms with an alkoxypolyglycol at a temperature in the range of 110° C. to 120° C. and with an aliphatic saturated amine selected from the group consisting of primary and secondary water-soluble aliphatic amines at a higher temperature in the range of 120° C. to 130° C.

3. A process of making a surface active agent which comprises successively combining a high molecular weight fatty acid having 10 to 18 carbon atoms with an alkoxylpolyglycol having a molecular weight of 200 to 1,000 and with an aliphatic saturated amine selected from the group consisting of primary and secondary water-soluble aliphatic amines.

4. A process of making a surface active agent which comprises successively combining one mol of a high molecular weight fatty acid having 10 to 18 carbon atoms with $\frac{1}{10}$ to ½ mol of an alkoxypolyglycol and then with ½ to 1½ mols of an aliphatic saturated amine selected from the group consisting of primary and secondary water-soluble aliphatic amines.

5. A process of making a surface active agent which comprises successively combining a high molecular weight fatty acid having 10 to 18 carbon atoms with an alkoxypolyglycol at 110° C. to 120° C. for ½ to 2 hours and then with an aliphatic saturated amine selected from the group consisting of primary and secondary water-soluble aliphatic amines at 120° C. to 130° C. for 2 to 4 hours.

6. A process of making a surface active agent which comprises successively combining a high molecular weight fatty acid having 10 to 18 carbon atoms with an alkoxypolyglycol and then with an ethanolamine selected from the group consisting of mon-ethanolamine and di-ethanolamine.

7. A process of making a surface active agent which comprises successively combining a high molecular weight fatty acid having 10 to 18 carbon atoms with a methoxy polyethylene glycol and then with a polyalkylene polyamine.

8. An emulsifying and wetting agent and detergent consisting of the complex reaction products of a combination of a high molecular weight fatty acid having 10 to 18 carbon atoms, an alkoxypolyglycol and an aliphatic saturated amine selected from the group consisting of primary and secondary water-soluble aliphatic amines.

9. An emulsifying and wetting agent and detergent consisting of the complex reaction products of a combination of a high molecular weight fatty acid having 10 to 18 carbon atoms and a

methoxy polyethylene glycol with an aliphatic saturated amine selected from the group consisting of primary and secondary water-soluble aliphatic amines.

10. A process of preparing surface active agents which comprises heating about 1 mol of a fatty acid having 8 to 24 carbon atoms with ½ mol of an alkoxypolyethylene glycol having a molecular weight of about 200 to 1000 at 110° C. to 120° C. for about one hour and then reacting the combination with about 1.1 mol of an alkanolamine at 115° C. to 130° C. for about two hours.

ALTON A. COOK.
IRA SAPERS.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,491,478	Cook et al.	Dec. 20, 1949