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POLYALKANOLAMINE CONDENSED WITH FATTY ACID MIXTURE

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The present invention is directed to condensation products, more particularly to such products formed by the reaction between fatty acids and certain amines. Products of this type have excellent wetting properties.

It has heretofore been proposed to react a fatty acid or a mixture of fatty acids, usually obtained from vegetable or similar oils, with alkylol amines. Reaction products of this type were produced by reacting 2 mols. of the alkylol amine with 1 mol. of the fatty acid. It was customary in such procedures to provide a mixture of the reactants and heat the same to a temperature as high as 300° C. to cause a reaction to take place therebetween. The resulting products were wetting agents and have found considerable use in industry. As the acid constituent and such substances, single higher fatty acids were used, such as stearic acid or certain mixtures of fatty acids were employed, such as the mixed fatty acids obtained by the hydrolysis of cocoanut oil.

One of the important uses for substances of this type has been as a wetting and cleansing agent for personal use. Solutions of these substances in water have been on the market as hair shampoos and as bubble bath solutions. While substances made in accordance with the prior art were applicable for such uses, they suffered from several disadvantages. For instance, there was a tendency for crystals to be precipitated if the solutions were kept in a cold place, such as 40° to 45° F. for a substantial time. After such precipitation, it was difficult by heating the solution to bring the crystalline material back into solution. This rendered the product unusable in many instances. Also, the sudsing power of the material was not entirely satisfactory. Furthermore, no direct and adequate control of the reaction process was attempted so that various batches of the same reaction product had different wetting properties and different solubilities in water.

The present invention is intended and adapted to overcome the difficulties and disadvantages inherent in the prior art and to provide a method of producing condensation products of the type above set forth, it being among the objects of the present invention to provide an adequate control for the operation which will insure uniform and reproducible results.

It is also among the objects of the present invention to provide a process of forming condensation products of the type described which will result in a product having a maximum and uniform wetting power and which is completely soluble in water.

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It is further among the objects of the present invention to provide a product of the type described which will form clear solutions in great dilutions, which will remain clear without depositing any crystalline material, and which will produce optimum sudsing power in use.

In practicing the present invention, an alkylol amine is heated to a point above its melting point and while at such temperature, there is added thereto a mixture of fatty acids. The proportion is about 2 mols. of alkylol amine to 1 mol. of said fatty acids. The fatty acid used consists of a mixture in which the principal acid is lauric acid with minor amounts of capric and myristic acids.

In the reaction the mixture so formed is heated slowly and gradually for a period of several hours until a temperature of about 140° to 165° C. is reached. Then the temperature is held at said maximum for several hours until the reaction has gone to the desired extent. During the reaction, water is formed which preferably is removed at such rate so as to prevent the formation of substantial amounts of foam. It is advisable that some of the condensed water be returned to the reaction.

The reaction is arrested when the free fatty acid content is below 15% of the total acids present in the reaction mixture. It is essential that some free fatty acid remain in the mixture and usually at least 1% of such acid remains. If the reaction is arrested at this point, then the condensation product is completely soluble in water and forms clear solutions when present in amounts from .1% to 10% of the solution. It has a high wetting speed and meets the standard sudsing test. In standing at low temperatures, no insoluble matter is formed and the solution remains clear.

The proportions of the fatty acid present in the reaction mixture are important and fall within the following limits.

	Percent
Lauric Acid	80–95
Capric Acid	4–10
Myristic Acid	1–10

It has been found that within such limits, wherein the lauric acid is by far the major constituent and the other acids are present in minor amounts, the desirable properties of the condensation product are obtained. The alkylol amines used are preferably the di- or tri-polyalkylol

amines wherein the alkyl group has 2 or 3 carbon atoms. While usually single polyalkylol amines are used, a mixture thereof is also suitable for the purpose.

The operation of the invention is illustrated by the following example:

There is provided a mixture of acids, as follows:

	Percent
Lauric acid	90
Capric acid	8
Myristic acid	2

There is also provided diethanol amine, which is placed in the reaction kettle and heated to between 50° and 70° C. in order to melt the diethanol amine. There is then added to the melted diethanol amine the above mixture of fatty acids, and the mixture is agitated during the addition of the acid, the temperature rising gradually to about 90° C. The ratio of the constituents in the reaction vessel is about 5100 lbs. of diethanol amine and 4900 lbs. of the mixed fatty acids. The kettle is heated to cause a slow and gradual rise in temperature, the rate of heating being such that a temperature of about 150° C. is reached after heating for about four hours. Heating is continued to maintain the temperature at about 150° C. for about three additional hours. By this time, the apparent free fatty acid content of the reaction mass is less than 15. Heating is then discontinued and the reaction is considered to be complete.

The completion of the reaction may be gauged by testing the reaction mixture from time to time. A solution of .1% of the condensation in water is crystal clear. When 50 cc. of this solution is placed in a 4 ounce oil sample bottle and vigorously shaken ten times, it develops from one and one-half to two inches of foam. Said solution when subjected to the Draves test shows a wetting speed of 10 seconds or less at 25° C.

The reaction product is capable of dilution with water to an extremely low concentration without producing cloudiness. It has a high sudsing power and has excellent wetting and cleansing properties. The procedure can be repeated time after time with the resulting condensation product having uniform and highly desirable wetting and cleansing properties. The control of the operation is relatively simple and no difficulties develop in carrying out and in repeating the procedure.

Although the invention has been described setting forth a single specific embodiment thereof, said specific example is intended to illustrate the invention and not to limit the same. Various changes in the details of the procedure may be made without departing from the spirit of the invention. For instance, although in the first step of the operation the alkylol amine is heated to above the melting point thereof, before the addition of the fatty acid mixture, it is possible to heat the same to a temperature higher than that set forth in the specific example, but below the principal reaction temperature, say up to 90° or 100° C. before the addition of the fatty acid mixture. The water formed in the reaction may be removed continuously by subjecting the reaction kettle to a partial vacuum whereby most of the water is removed during the reaction. It is desirable that the water be removed but the presence of some water in the reaction mixture is not detrimental. The removal of the excess water may be accomplished by different methods than the above mentioned vacuum.

The course of the reaction is dependent upon both the temperature and the time involved. When the higher temperatures are used, the reaction proceeds more vigorously and therefore the time of reaction is correspondingly decreased. Also when a vacuum is used during the reaction, the same is accelerated and this may be compensated for by either reducing the time of reaction or lowering the temperature thereof, or both. In place of the acids specifically set forth, other fatty acids having the same numbers of carbon atoms, which may be considered isomeric with the acids named herein, may be used in place thereof, either in whole or in part. The mixture of fatty acids is essential to the operation as the properties obtained in the eventual condensation product are believed to have the desirable properties due to the mixed compounds produced.

These and other changes in the details of the invention may be made within the scope thereof and the invention is therefore to be broadly construed and not to be limited except by the character of the claims appended hereto.

I claim:

1. A condensation product of about 2 mols. of a polyalkylolamine having from 2 to 3 carbon atoms in the alkyl groups with about 1 mol. of a mixture of lauric, capric and myristic acids, the lauric acid constituting at least 80% of the acids present, said product having a free fatty acid content of 1% to 15% of the total acid present formed by heating the reaction mixture to gradually raise the temperature to about 140°–155° C. and maintaining said temperature until the free fatty acids are 1% to 15% of the acids present.

2. A condensation product of about 2 mols. of a polyalkylolamine having from 2 to 3 carbon atoms in the alkyl groups with about 1 mol. of a mixture of lauric, capric and myristic acids in the following proportions:

	Percent
Lauric acid	80–95
Capric acid	4–10
Myristic acid	1–10

having a free fatty acid content of 1% to 15% of the total acids present formed by heating the reaction mixture to gradually raise the temperature to about 140°–155° C. and maintaining said temperature until the free fatty acids are 1% to 15% of the acids present.

3. A condensation product of about 2 mols. of diethanolamine with about 1 mol. of a mixture of lauric, capric and myristic acids, the lauric acid constituting at least 80% of the acids present, said product having a free fatty acid content of 1% to 15% of the total acid present formed by heating the reaction mixture to gradually raise the temperature to about 140°–155° C. and maintaining said temperature until the free fatty acids are 1% to 15% of the acids present.

4. A method of making condensation products which comprises heating 2 mols. of a polyalkylolamine having from 2 to 3 carbon atoms in the alkyl groups to above the melting point thereof, mixing therewith at said point about 1 mol. of a mixture of lauric, capric and myristic acids of which lauric acid constitutes at least 80% of the acids present, heating the reaction mixture to gradually raise the temperature to about 140°–155° C., and maintaining said temperature until the free fatty acids remaining in the condensation product are 1 to 15% of the acids present.

5. A method of making condensation products which comprises heating 2 mols. of a polyalkylolamine having from 2 to 3 carbon atoms in the alkyl groups to above the melting point thereof, mixing therewith at said point about 1 mol. of a mixture of lauric, capric and myristic acids of which lauric acid constitutes at least 80% of the acids present, heating the reaction mixture to gradually raise the temperature to about 140°–155° C., and maintaining said temperature until the free fatty acids remaining in the condensation product are 1 to 15% of the acids present, while removing in the vapor state water formed in the reaction.

6. A method of making condensation products which comprises heating 2 mols. of diethanolamine to above the melting point thereof, mixing therewith at said point about 1 mol. of a mixture of lauric, capric and myristic acids of which lauric acid constitutes at least 80% of the acids present, heating the reaction mixture to gradually raise the temperature to about 140°–155° C., and maintaining said temperature until the free fatty acids remaining in the condensation product are 1 to 15% of the acids present.

7. A method of making condensation products which comprises heating 2 mols. of a polyalkylolamine having from 2 to 3 carbon atoms in the alkyl groups to above the melting point thereof, mixing therewith at said point about 1 mol. of a mixture of lauric, capric and myristic acids of which lauric acid constitutes at least 80% of the acids present, heating the reaction mixture to gradually raise the temperature to about 140°–155° C., and maintaining said temperature until the free fatty acids remaining in the condensation product are 1 to 15% of the acids present, while removing in the vapor state water formed in the reaction, and returning a part of the water to the reaction.

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