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PROCESS FOR PREPARING A MIXTURE OF SOAP AND FATTY-ACYL-AMINOMETHANE SULFONATE

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This invention relates to an improvement in the preparation of alkali-metal salts of fatty-acyl-aminomethane sulphonates of formula $R \cdot CO \cdot NH \cdot CH_2 \cdot SO_3M$ where $R \cdot CO$ is the acyl radical of a fatty acid and M is sodium or potassium.

Fatty acyl-aminomethane sulphonates have in the past been prepared by the reaction of a fatty amide with between about 0.8 and about 2 molecular proportions of sodium or potassium formaldehyde bisulphite at temperatures of from about 150 to 210° C. The reaction mixture is initially quite fluid at the reaction temperature and may be handled in conventional jacketed vessels with light stirring gear, but, as the reaction proceeds, the mixture becomes more and more viscous and finally so stiff that it becomes incapable of being stirred with such gear and difficult to remove from the reaction vessel. To overcome these difficulties the reaction mixture may be transferred from the first reaction vessel, after the reaction has proceeded to a certain degree but before the mixture has become too viscous to run from the vessel, into a second reaction vessel, of the kneader-mixer type with powerful sigma-shaped stirrers, to complete the reaction, but this process involves more labour, plant and power than is desirable.

It has now been found that when a mixture of free fatty acid and soap in suitable amounts is added to a reaction mixture of fatty amide and not more than about 1.2 molecular proportions of sodium formaldehyde bisulphite, the mixture remains fluid during the reaction, which can therefore be carried out in a simple reaction vessel with a light stirrer, and yields a final product which has a reasonably high conversion of amide to fatty-acyl-aminomethane sulphonate. By simple conversion of the free fatty acids into soap, useful mixtures containing soap and fatty-acyl-aminomethane sulphonate are obtained. For many purposes fatty-acyl-aminomethane sulphonates are required in the form of mixtures with soap.

The present invention provides, therefore, a process for the preparation of mixtures containing fatty-acyl-aminomethane sulphonates and soap in which fatty amides are caused to react with from 0.8 to 1.2 mols of sodium or potassium formaldehyde bisulphite in the presence of free fatty acid and soap, and the free fatty acid is subsequently neutralised.

It is preferred to use a molecular ratio of the formaldehyde bisulphite to amide of about 1:1. The minimum amount of free fatty acid required to maintain the reaction mixture in the fluid state depends to some extent upon the reaction conditions, particularly the actual composition of the reaction mixture, the temperature of the reaction, and the extent to which the escape of water from the reaction mixture is allowed or promoted: a reaction mixture from which the water of reaction is continuously removed by a current of inert gas, for instance, will require more fatty acid to maintain fluidity at a given reaction temperature than one in which some water is retained in the mixture. Generally, the amount of free fatty acid should be not less than about 15% by weight of the amide used. The maximum amount of free fatty acid that can be used is not critical but excessive amounts may reduce the conversion of the amide to fatty-acyl-aminomethane sulphonate and may in any case give final products of restricted usefulness. Normally, therefore, the maximum amount of free fatty acid will be about 40% by weight of the amide.

It has been found that when no or only very small amounts of soap are added to the reaction mixture in addition to free fatty acid, although satisfactory fluidization is obtained, the conversions of the formaldehyde bisulphite and fatty amide to fatty-acyl-aminomethane sulphonate are lower, and the product has an inferior colour and odour. The addition of the proper amount of soap to the reaction has the unexpected advantage of increasing the yield. Preferably, not less than 1% of soap by weight of the amide should be used. The upper limit is also critical since too large a proportion of soap in relation to the free fatty acid may fail to prevent the solidification of the reaction mixture during the course of the reaction. Again, this depends to some extent upon the reaction conditions but, in general, the soap present should not exceed the amount by which the free fatty acid exceeds 14% by weight of the fatty amide. Since the upper limit for free fatty acid is about 40%, the upper limit of soap is about 26%, but normally much less is required.

Thus, for example, using a molar ratio of sodium formaldehyde bisulphite to fatty amide of 1:1, satisfactory amounts of free fatty acid and soap, expressed as percentages by weight of the amide have been found to be as follows:

free fatty acids ---------- percent	17	27	34
soap ---------- do	1.3	10	13

The process will normally be applied to the preparation of mixtures containing sodium fatty-acyl-aminomethane sulphonates derived from acids containing about 10 to 18, preferably 12 to 14, carbon atoms. Mixtures of such amides may be used, particularly mixtures such as may be obtained from the the mixed fatty acids of natural oils such as palm oil, palm kernel oil, coconut-oil, cottonseed-oil, groundnut-oil and bone grease. It is preferred to use amides derived mainly or entirely from the fatty acids of palm kernel oil or coconut oil.

The fatty acids which are added in the process of the invention may also contain from 10 to 18 carbon atoms and may again be mixtures such as those derived from any of the usual natural fats or oils. Commercial fatty amides normally contain a proportion of free fatty acids and the added free fatty acid may be the same as or different from that already present in the amide which, however, must be included when reckoning the amount of free fatty acid present.

The soap which is used in the process of the invention may again be derived from any fatty acid having from about 10 to 18 carbon atoms in the molecule, including mixtures of acids such as those obtained from natural oils, which may be the same as or different from the free fatty acid also present. Normally it is most convenient to add free fatty acid to the reaction mixture and to convert an appropriate proportion of this to soap in situ. The cation of the soap may be an alkali metal, ammonium or an organic amine such as di-cyclohexylamine. The soap should be water soluble. It is preferred to use the sodium soaps, which are conveniently made by adding the appropriate amount of sodium carbonate to a mixture of the free fatty acid and the other ingredients.

The reaction is carried out in conventional jacketed mixing vessels with paddle-type stirring gear at a temperature of 150° C. to 210° C., preferably 170° C. to 190° C., and is preferably carried out in an inert atmosphere, for example an atmosphere of nitrogen, carbon dioxide or steam. The time during which the reactants

are maintained at the desired temperature depends upon the actual reaction temperature selected and on the proportions of the various ingredients of the reaction mixture. At a temperature of 175° C. to 180° C., for instance, a time of between 1 and 3 hours is usually satisfactory, the conversion having then reached its maximum. At the end of this time the reaction mixture is still sufficiently fluid to be run from the mixing vessel, preferably under an inert gas blanket. After cooling it may be fully or partially neutralised, for example by the addition of sodium carbonate or sodium hydroxide, and the resulting mixture subjected to any desired processing. The presence of unconverted amide and formaldehyde bisulphite and of various by-products of the reaction in the comparatively small proportions in which they remain or arise under these conditions is tolerable for many of the uses to which the reaction product may be put. If desired, the reaction product, after neutralisation, may be purified to some extent from the formaldehyde bisulphite and other water-soluble impurities by dissolving or slurrying it with hot water and graining it out by the addition of sodium chloride or brine in accordance with the normal soap-making technique. Additional soap, if it is required in the final composition, may be added before the mixture is subjected to graining-out. The grained-out mixture may contain a somewhat higher salt content, e.g. 1.5–2.0%, than is normal for a grained-out soap but any tendency for this to cause efflorescence when the mixture is used for solid compositions may be counteracted by adding small amounts, e.g. 3% of sodium toluene sulphonate or sodium xylene sulphonate.

The following example is given solely for purposes of illustration and is not to be considered as limiting the invention to this embodiment. Many modifications will be apparent to those skilled in the art without departing from the spirit or scope of the invention.

Example

A mixture of equimolecular amounts of sodium formaldehyde bisulphite and the fatty amides derived from palm kernel oil together with 34.6%, by weight of the amide, of the free acids derived from palm kernel oil and 9.5%, by weight of the amide, of the sodium soap derived from the same acids, was heated to 180° C. and stirred during 2½ hours in a closed vessel under an atmosphere of carbon dioxide at atmospheric pressure. At the end of the reaction period the mixture was quite fluid and analysis showed that a conversion of 63% of the amide to fatty-acyl-aminomethane sulphonate had taken place. After running from the reaction vessel the mixture was slurried in hot water, additional soap was added and the slurry was carefully neutralised with sodium hydroxide and grained-out by the addition of saturated brine. The grained-out product was eminently suitable for the manufacture of detergent tablets.

What is claimed is:

1. A process for the preparation of a mixture comprising a water soluble soap and an alkali metal salt of a fatty-acyl-aminomethane sulphonate selected from the group consisting of sodium and potassium salts and having from 10 to 18 carbon atoms in the acyl radical, said process comprising reacting a fatty amide having from 10 to 18 carbon atoms with from 0.8 to 1.2 molecular proportions of an alkali metal formaldehyde bisulphite selected from the group consisting of sodium formaldehyde bisulphite and potassium formaldehyde bisulphite, in the presence of: (*a*) from 15 to 40%, by weight of the amide, of a free fatty acid having from 10 to 18 carbon atoms, and (*b*) from about 1 to 26% by weight of the amide of a water soluble alkali metal soap having from 10 to 18 carbon atoms, the reaction being carried out at a temperature of from 150° C. to 210° C.

2. The process of claim 1 in which the reaction is carried out in an inert atmosphere.

3. The process of claim 1 in which subsequent to the reaction the free fatty acid is converted to soap by reaction with a water soluble alkali metal alkali.

4. A process for the preparation of a mixture comprising a water soluble soap and an alkali metal salt of a fatty-acyl-aminomethane sulphonate selected from the group consisting of sodium and potassium salts and having from 10 to 18 carbon atoms in the acyl radical, said process comprising reacting a fatty amide having from 10 to 18 carbon atoms with approximately an equal molecular proportion of an alkali metal formaldehyde bisulphite selected from the group consisting of sodium formaldehyde bisulphite and potassium formaldehyde bisulphite, in the presence of: (*a*) about 35%, by weight of the amide, of a free fatty acid having from 10 to 18 carbon atoms, and (*b*) about 10% by weight of the amide of a water soluble alkali metal soap having from 10 to 18 carbon atoms, the reaction taking place at a temperature of from 170° to 190° C.

5. The process of claim 4 in which the reaction is carried out in an inert atmosphere.

References Cited in the file of this patent

UNITED STATES PATENTS

2,313,695	Yamashito et al.	Mar. 9, 1943
2,366,452	Mack	Jan. 2, 1945
2,857,370	Sundberg	Oct. 21, 1958
2,880,219	Burnette et al.	Mar. 31, 1959