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2,974,154

PROCESS OF PREPARING SALT-FREE N-ACYL TAURINES

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This invention relates to an improved process for preparing salt-free N-acyl taurines and more particularly, to an improved process for separating such taurines from aqueous slurries containing both water soluble salts and such taurines.

The reaction of higher fatty acids, fatty acid chlorides and fatty acid esters with 2-aminoalkane sulfonic acids (taurines) and the alkali metal salts thereof to yield anionic surface active materials useful as wetting, cleansing, softening and dispersing agents is well known. In U.S. Patent No. 1,932,180, several processes are described for the preparation of such surface active materials, but the only process which has up to the present been employed commercially is that wherein an acid chloride is reacted in aqueous medium with a 2-aminoalkane sulfonic acid in the presence of an acid neutralizer such as caustic soda. This inherently produces, in addition to the N-higher acyl taurine, a considerable quantity of salt, e.g. sodium chloride (or potassium chloride if caustic potash is used as acid neutralizer) as a by-product.

The presence of such salts with the N-higher acyl taurine is highly undesirable and disadvantageous for a number of reasons. Not only do such salts have a substantial corrosive effect in the packaging and handling of detergents and other surface active compositions containing such acyl taurines, but they also exert a strong adverse effect on the lathering, detergent and other surface active properties of mixtures of such acyl taurines with soap. The acyl taurines are very good lime soap dispersants and the combination of minor amounts thereof with ordinary fatty acid soaps in detergent and built soap formulations results in highly effective detergent properties, particularly in hard water. However, for such combinations to have optimum properties, the N-higher acyl taurine present therein should have a low salt content.

The presence of this salt in a number of commercially available N-acyl taurines, has been identified as a major reason for the unduly high hygroscopicity of such products. The presence of such salt is also undesirable when the N-acyl taurine is to be employed in certain emulsion polymerization reactions and in other uses.

A number of proposals have been made in the prior art for the production of salt-free N-higher acyl taurines, some of which involve the use of a different method of reaction or of different reactants in the production of such taurines, and others of which involve expensive and difficult separation methods.

It is an object of this invention to provide a process for producing a salt-free N-higher acyl taurine which will not be subject to the above disadvantages. Another object of this invention is the provision of a process for producing a salt-free N-higher acyl taurine from an aqueous slurry containing such taurine in combination with a water soluble salt. Other objects and advantages will appear as the description proceeds.

The attainment of the above objects is made possible by the present invention which includes a process com-

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prising adding to an aqueous slurry containing (A) an N-higher acyl taurine of the formula



- 5 wherein one R is selected from the group consisting of H and methyl and the other R is H, R' is selected from the group consisting of H and hydrocarbon radicals of 1 to 6 carbon atoms, R' is selected from the group consisting of aliphatic, alicyclic and aromatic hydrocarbon radicals of 7 to 23 carbon atoms, and M is selected from the group consisting of H, alkali metal, alkaline earth metal, ammonium and amino, and (B) a water soluble salt selected from the group consisting of the alkali metal chlorides and sulfates, a water insoluble compound
- 10 C selected from the group consisting of aliphatic and alicyclic monocarboxylic acids of 8 to 24 carbon atoms and their esters in an amount sufficient to form a compatible solvent system with said taurine (A), heating the resulting mixture to distill off the water, and then separating the crystallized salt B from the mixture at a temperature above the solidification point of the A and C solvent system.

An outstanding advantage of the process of this invention is that it is operable on the commercially available N-higher acyl taurines produced by the Schotten-Baumann reaction between a higher fatty acid chloride and a taurine, and may be carried out by soap manufacturers themselves in readily available equipment. The product produced by the above process is a mixture of the acyl taurine with the higher molecular weight carboxylic acid or ester which may be employed as such in certain cosmetic and other uses, or may be treated with an inorganic or organic base such as the alkali metals or amines such as triethanolamine or the like to saponify the carboxylic acid and produce its soap in situ. Such soap compositions are useful directly as toilet soaps and other cosmetic applications in liquid or solid form and can readily be formulated into bars or cakes. If desired, the resulting combination of soap and N-higher acyl taurine may be spray dried or drain dried to produce a flake or powder detergent and may be combined before or after such drying with common builders, stabilizers and the like for the production of heavy duty built detergent compositions. Common components of such built compositions include alkali metal phosphates and polyphosphates, silicates, carbonates, and sodium carboxymethyl cellulose.

Since the present process is performed at moderate temperatures, no discoloration of product or reactants is experienced and the original purity of the intermediates is representative of the final product, excluding those impurities that are insoluble in the dehydrated solution and thereby removed during the processing. Highly specialized equipment required by the prior art to maintain the necessary reaction conditions for the production of the desired salt-free N-higher acyl taurines is no longer necessary. The operability of the present process is surprising and unexpected in view of the anticipated foaming which would normally be expected to occur in a fatty acid-synthetic detergent-soap and water formulation of the type involved in the present process.

If a final product is desired which must be not only salt-free but also substantially pure, the higher carboxylic acid employed to form a compatible solvent system with the acyl taurine may be conveniently removed by distillation.

The process of this invention is applicable for the treatment of any aqueous slurry containing the N-higher acyl taurine and a water soluble salt, regardless of its manner of production. The N-higher acyl taurines which are produced in salt-free form in accordance with this invention may in general be ascribed the formula given

above. Such acrylated taurines may be prepared by reaction of a taurine salt containing at least one N-bonded hydrogen atom with a higher molecular weight acylating agent of the formula $R'COOH$ or $R'COCl$. Those acylating agents particularly preferred are the higher free fatty acids of 12 to 18 carbon atoms (R' is 7 to 17 carbon atoms). As generally representative of higher aliphatic and alicyclic carboxylic acids operative in the instant invention, there may be mentioned caprylic acid, pelargonic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, oleic acid, ricinoleic acid, linoleic acid, undecylenic acid, tall oil acid, acid mixtures from various natural plant and animal oils such as olive, tallow, castor, peanut, coconut, soybean, cotton seed, ucahuba, linseed, cod, herring, menhaden, neatfoot, sperm, palm, corn, butter, babassu, kapok, hempseed, mustard, rubberseed, rape, sunflower, sesame, acids from the oxidation fractions of petroleum, and from oxo-aldehydes, naphthenic acids, abietic acids, and the hydrogenated derivatives of such acids and acid mixtures. Other acids which may be employed include alkyl benzoic acids such as dodecyl benzoic acid, nonyl benzoic acid, octyl benzoic acid, alkyl naphthoic acids such as nonyl naphthoic acid, and the like.

The above described carboxylic acid acylating agents are accordingly representative of the aliphatic, alicyclic and aromatic hydrocarbon radicals of 7 to 23 carbon atoms which may be employed as R' in the above formula. Such carboxylic acid acylating agents (e.g. $R'COCl$) are reacted with taurines of the formula



to produce the acylated taurines treated in accordance with the present process. In this formula, R'' may represent hydrogen, methyl, ethyl, isopropyl, butyl, hexyl, cyclohexyl, phenyl and the like, and M may represent hydrogen or a salt-forming cation such as an alkali metal, e.g. sodium, potassium or lithium, an alkaline earth metal such as calcium, magnesium or barium, ammonium, or an amine such as mono-, di- or triethanolamine, cyclohexylamine, guanidine or the like. Thus, by way of example only, the following specific 2-aminoalkane sulfonic acids may be employed as such or in the form of their salts for reaction with the above defined carboxylic acid acylating agents: taurine, N-methyl taurine, N-ethyl taurine, N-propyl taurine, N-isopropyl taurine, N-butyl taurine, N-amyl taurine, N-hexyl taurine, N-cyclohexyl taurine, N-phenyl taurine, N-methyl-2-methyl taurine, N-methyl-1-methyl taurine, and the like.

The aqueous slurries treated in accordance with the present process, should in general be sufficiently fluid to be readily workable. In general, in such slurries, the acylated taurine would be present in concentrations of about 15 to 40% by weight, 35% slurries being those most readily available on the market. In such slurries, the water soluble salts usually present as a result of the method manufacturing the acylated taurine may be present in proportions as high as 30 to 35% of the weight of the acylated taurine, 20 to 25% being common. It will of course be understood that the present process is also operative for removing smaller proportions of salt, e.g. as little as 5% or less or higher proportions, e.g. up to 50% or more, of the weight of the acylated taurine. The salt most commonly present in such slurries is sodium chloride, usually accompanied by smaller proportions of sodium sulfate. However, potassium chloride and potassium sulfate may also be present for other reasons or because potash was employed in the reaction for producing the acylated taurine. The acylated taurine slurries commonly available normally have a pH of about 7 to 8, but the process of this invention is operative not only on these slurries but on those of a much higher acidity or alkalinity.

As stated above, the process of this invention requires the addition to an aqueous slurry as above described of

a water insoluble compound (component C) selected from the group consisting of aliphatic and alicyclic monocarboxylic acids of 8 to 24 carbon atoms and their esters in an amount sufficient to form a compatible solvent system with the acylated taurine (component A).

The preferred component C for use in the present process is a free higher fatty acid of 12 to 18 carbon atoms which may be saturated or unsaturated, branched but preferably straight. Other aliphatic and alicyclic monocarboxylic acids which may be employed as component C in the present process include those disclosed above as operative acylating agents ($R'COOH$) with the exception of the alkyl benzoic and alkyl naphthoic acids since component C and component A (acylated taurine) must be mutually soluble to form a compatible solvent system melting below the boiling point of water and boiling above the boiling point of water under the conditions of the distillation required in the present process. The methyl, ethyl, propyl or glyceryl esters of such acids may also be employed. It will be generally found that the amount of component C necessary to form a compatible solvent system with component A at the temperature of separation of the crystallized salt from the mixture above the solidification point of such system will be at least twice the weight of said component A (acylated taurine), and will in general range from about 2 to 6 parts per part by weight of component A. Higher proportions may be employed but are uneconomical except where a larger proportion of soap is desired in the final product as obtained by saponification of component C in situ following removal of the water and salt in accordance with the present process.

After addition of the required amount of component C to the aqueous slurry containing the acylated taurine and water soluble salt, the slurry is heated to the boiling point to distill off the water. The distillation may be carried out at super- or sub-atmospheric pressure but it is preferred, in the interests of simplicity and economy to operate at atmospheric pressure. Surprisingly, the boiling proceeds without the anticipated foaming. At normal atmospheric pressures, temperatures of about 95 to 135° C. are preferred for optimum simplicity, economy and rate of dehydration. A small flow of air or inert gas over the surface of the boiling slurry is desirable but not essential to aid in removal of water vapor. An inert gas such as nitrogen or carbon dioxide is in such case preferred to prevent possible discoloration of the acylated taurine. Lower temperatures of boiling may be employed at sub-atmospheric pressures.

The distillation is continued until substantially no more water is removed, as indicated by substantial cessation of loss of weight of the distillation vessel. The duration of the distillation process will of course in any particular instance depend upon the amount of slurry being treated, the temperatures and pressures employed, and the like. Under usual conditions and batch sizes, a duration of about 1 to 3 hours is sufficient. At the completion of this distillation step, substantially all of the salt has been precipitated in the distillation vessel which may still contain a small amount of occluded water.

During the boiling operation and as the water is removed from the aqueous slurry, the water soluble salts crystallize out and may, after completion of the distillation step, be readily separated from the remaining mixture at a temperature above the solidification point of the compatible solvent system of components A and C contained in the mixture. Stated otherwise, such solvent system is maintained in liquid form to permit removal of the crystallized water soluble salt originally dissolved in the aqueous slurry. The separation of the salt may be carried out in any desired manner as by filtration, centrifugation, decantation, or the like. In practice, it would be desirable to filter or centrifuge the mixture immediately after all the water has been removed and while it is still hot and in liquid condition. Generally, the separa-

tion would be carried out at about 130° C. although any temperature would be suitable provided it is above the solidification point of the system throughout the separating operation.

By the present process, there is thus obtained from the original aqueous slurry a substantially homogeneous mixture or solution of component A and component C. This product is advantageously subjected to saponification with caustic soda or potash to produce the soap of component C in situ together with the acylated taurine. However, if desired, component C may be separated from the acylated taurine by distillation or the like.

The following examples, in which parts are by weight, unless otherwise indicated, are only illustrative of the instant invention and are not to be regarded as limitative. In these examples, all parts and proportions of fatty acid amides are determined by the methylene blue analytical method described in Nature, 160, 759 (1947) and Trans. Faraday Soc. 44, 226-239 (1948). The tallow fatty acid contains, by weight, approximately 10% myristic, 43% palmitic, 9% stearic, 30% oleic, and 8% linoleic acids, and the coconut fatty acid contains, approximately, 8% caprylic, 7% capric, 49% lauric, 17% myristic, 9% palmitic, 2% stearic, 6% oleic, and 2% linoleic acids.

Example 1

Twelve hundred grams of an aqueous commercial slurry containing 300 g. of sodium N-methyl-N-"coconut fatty acid" taurate and 69 g. sodium chloride is admixed with 900 g. commercial coconut fatty acid and gradually heated to 130° C. with agitation in an open vessel with a loss of 685 g. water, after which crystallized salts are immediately removed by filtration. The filtrate contains by analysis 300 g. of said taurate, 0.63 g. sodium chloride, and 17.3 g. water, in addition to the fatty acid.

Upon saponifying the fatty acid with caustic to convert same to the sodium soap in situ, the resultant detergent-soap mixture can be converted to bars with excellent detergent properties.

Example 2

One hundred fifty grams of an aqueous commercial slurry containing 52.5 g. sodium N-methyl-N-oleoyl taurate and 10.2 g. sodium chloride is admixed with 100 g. coconut fatty acid and heated to 135° C. to remove 71 g. water by atmospheric distillation, after which the inorganic suspension is immediately removed by filtration, and the filtrate analyzed as containing 52.0 g. of said taurate, 0.1 g. sodium chloride, and 3.0 g. water in addition to the fatty acid.

Example 3

One hundred fifty grams of an aqueous commercial slurry containing 52.5 g. of sodium N-methyl-N-oleoyl taurate and 10.2 g. sodium chloride is admixed with 100 g. coconut fatty acid and dehydrated at 55° C. by heating under reduced pressure of about 150 mm. of Hg until no more water distills over. The crystallized salts are then immediately removed by centrifuging, and the clear oil effluent found by analysis to contain in addition to said fatty acid 52.5 g. of said taurate, and substantially no sodium chloride or water.

Example 4

Two hundred forty grams of an aqueous commercial slurry containing 24.6% by weight sodium-N-methyl-N-"coconut fatty acid" taurate and 5.75% by weight sodium chloride is added to 160 g. tallow fatty acid and the mixture dehydrated by heating to 140° C. under a stream of nitrogen until substantially no more water distills over. The inorganic suspension is then immediately removed by filtration, and the product, 235 g. analyzed as containing, in addition to the tallow fatty acid and some water, 25.1% by weight of said taurate and 0.291% by weight sodium chloride.

Example 5

Four hundred parts by weight of an aqueous commercial slurry containing 25% by weight of sodium-N-methyl-N-"tallow fatty acid" taurate and 6.82% by weight sodium chloride are admixed at 60° C. with 200 parts by weight of commercial tallow fatty acid. The resultant slurry is fed into a continuous evaporator operating at 125 to 135° C., at a rate allowing substantially complete dehydration and the essentially anhydrous fluid effluent immediately freed of suspended inorganic salts by continuous filtration to yield a product containing 100 parts by weight of said taurate, 1 part by weight of sodium chloride and 19 parts by weight of water, in addition to the tallow fatty acid.

Example 6

One hundred parts by weight of a commercial aqueous slurry containing 18.8% by weight of sodium-N-cyclohexyl-N-palmitoyl taurate and 4.51% by weight of sodium chloride is admixed with 100 parts by weight of palmitic acid and processed in the manner disclosed in Example 2. The resultant product contains 18.8 parts by weight of said taurate, 0.01 parts by weight of sodium chloride and 0.3 parts by weight of water, in addition to the palmitic acid.

Example 7

The process of Example 2 is repeated, except that the initial slurry contains the corresponding potassium taurate salt and potassium chloride. Similar results are obtained.

Example 8

Six hundred parts by weight of an aqueous commercial slurry containing by weight 25% of sodium-N-methyl-N-"coconut fatty acid" taurate and 5.75% by weight of sodium chloride is admixed with 450 parts by weight of coconut fatty acid and heated to 135° C. to distill out substantially all the water originally contained in the slurry, after which the precipitated salts are immediately removed by filtration, and the coconut fatty acid is removed from the filtrate by distillation in vacuo to leave a residue analyzing 145 parts by weight of said taurate and 0.3 parts by weight of sodium chloride.

Example 9

The process of Example 6 is repeated except that the taurate in the initial slurry is sodium-N-butyl-N-palmitoyl taurate. Similar results are obtained.

This invention has been disclosed with respect to certain preferred embodiments, and various modifications and variations thereof will become obvious to the person skilled in the art. It is to be understood that such modifications and variations are to be included within the spirit and purview of this application and the scope of the appended claims.

I claim:

1. A process comprising adding to an aqueous slurry containing (A) an N-higher acyl taurine of the formula



wherein one R is selected from the group consisting of H and methyl and the other R is H, R' is selected from the group consisting of H, phenyl, alkyl radicals of 1 to 6 carbon atoms, and cyclohexyl, R' is selected from the group consisting of aliphatic and alicyclic and hydrocarbon radicals of 7 to 23 carbon atoms, and M is selected from the group consisting of H, alkali metal, alkaline earth metal, ammonium and amino, and (B) a water soluble salt selected from the group consisting of the alkali metal chlorides and sulfates, a water insoluble compound C selected from the group consisting of aliphatic and alicyclic monocarboxylic acids of 8 to 24 carbon atoms and their esters in an amount sufficient to form a compatible solvent system with said taurine A, heating the resulting mixture to distill off the water, and

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then separating the crystallized salt B from the mixture at a temperature above the solidification point of the A and C solvent system.

2. A process as defined in claim 1 wherein component A is sodium-N-methyl-N-"coconut fatty acid" taurate. 5

3. A process as defined in claim 1 wherein component A is sodium-N-methyl-N-"tallow fatty acid" taurate.

4. A process as defined in claim 1 wherein component A is sodium-N-oleoyl taurate.

5. A process as defined in claim 1 wherein component A is sodium-N-cyclohexyl-N-palmitoyl taurate. 10

6. A process as defined in claim 1 wherein component C is a free fatty acid of 12 to 18 carbon atoms.

7. A process as defined in claim 1 wherein said component C is added to said slurry in an amount at least twice the weight of said component A. 15

8. A process comprising adding to an aqueous slurry containing an N-higher fatty acid taurine compound and an alkali metal chloride, an amount of higher fatty acid 20

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of 12 to 18 carbon atoms sufficient to form a compatible solvent system with the said taurine compound, heating the resulting mixture to distill off the water, and then separating the crystallized alkali metal chloride from the mixture at a temperature above the solidification point of the solvent system containing the said taurine compound and the higher fatty acid.

9. A process as defined in claim 8 wherein said higher fatty acid is coconut fatty acid.

10. A process as defined in claim 8 wherein said higher fatty acid is tallow fatty acid.

11. A process as defined in claim 8 wherein said higher fatty acid is palmitic acid.

12. A process as defined in claim 8 wherein said higher fatty acid is added to said slurry in an amount ranging from about 2 to 6 parts by weight per part of the said taurine compound in the slurry.

No references cited.