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2,993,071

PURIFICATION OF SALTS OF DETERGENT AMINO ACIDS

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The present invention relates to the purification of salts of certain detergent amino acids and more particularly to the removal from salts of certain detergent amino acids organic color, odor and cloudiness forming impurities.

The salts of the detergent amino acids to which the present invention is directed are those having the following formula: $\text{RNH}_m(\text{CHR}'\text{CH}_2\text{COOMe})_n$ in which R is an aliphatic hydrocarbon group containing 8–22 carbon atoms and is derived from a detergent forming fatty acid, R' is H or CH_3 , Me is a salt forming group, m is 0 or 1, n is 1 or 2 but 1 when R' is CH_3 and the sum of m and n is 2.

The present commercial method of preparing these salts of detergent amino acids is by the condensation of a primary fatty amine and an acrylic acid ester as disclosed in the Isbell Patent No. 2,468,012.

While single primary fatty amines may be used in the preparation of these detergent amino acids, the mixtures of amines obtained by the amination of the fatty acids or selected fraction thereof present in common fats and oils such as tallow, coconut oil and the like are readily available and therefore particularly well suited for this purpose.

The production of detergent amino acid salts suitable for use in the manufacture of cosmetic grade products has heretofore required as a starting material the use of expensive, high quality, distilled amines. The presence of color, odor, and cloudiness forming impurities in the less expensive and more readily available lower quality distilled and undistilled primary fatty amines have made salts of detergent amino acids made with them heretofore less desirable for this use.

It is therefore an object of the present invention to provide a novel process of purifying the salts of detergent amino acids which were made with low quality distilled or undistilled fatty amines so that they may be used in the manufacture of cosmetic grade products.

It is also an object of the present invention to provide a novel process by which impure detergent amino acid salts may be upgraded in quality.

It has now been discovered that the salt of a detergent amino acid may be purified by neutralizing it with a non-oxidizing acid to form the zwitterion as described in the Andersen Patent No. 2,816,920, adding ethylene dichloride to the zwitterion phase to dissolve the color, cloudiness, and odor-causing impurities, and then removing the ethylene dichloride solution containing these impurities to leave a purified zwitterion which may then be reconstituted to the original salt of a detergent amino acid by addition of suitable alkali.

In the preferred practice of the invention, the salt of the detergent amino acid is dissolved in sufficient water to form 10–30% aqueous solution. The solution is then neutralized by means of sulphuric acid to a pH within the range of 3.5–5.5, at which stage a two phase separation occurs. The upper phase which contains the zwitterion is then removed and mixed with ethylene dichloride, the mixture is then heated until the zwitterion dissolves. After the solution has cooled to room temperature, a phase separation occurs and the ethylene dichloride layer is decanted. Further washings with ethylene dichloride may be required if a particularly low grade product is being purified. The original detergent

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amino acid salt is then formed by reconstituting the zwitterion with suitable alkali.

While sulphuric acid is the preferred acid in that it is less expensive, less corrosive, and requires less accurate control, other known nonoxidizing mineral acids such as hydrochloric and phosphoric may also be used with essentially the same results.

The choice of alkali to be used in the reconstitution step will be determined by the salt desired. For instance if a sodium salt is desired, NaOH will be used, whereas if a potassium or ammonium salt is desired KOH or NH_4OH will obviously be used.

The following examples further illustrate the practice of the present invention.

Example I

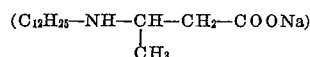
One thousand grams of flaked sodium β dodecylaminopropionate ($\text{C}_{12}\text{H}_{25}\text{NHC}_2\text{H}_4\text{COONa}$) was dissolved in 4,000 grams of water, sulphuric acid was added to adjust the pH to 3.5 whereupon phase separation occurred. The upper phase contained the zwitterion while the other aqueous phase contained the sodium sulphate. The zwitterion phase was then separated and washed twice with water. To the zwitterion phase was added 2,000 grams of ethylene dichloride and the mixture heated until the zwitterion dissolved. The solution was then cooled with stirring to room temperature at which point an ethylene dichloride layer separated out and was decanted. The zwitterion layer was washed by stirring with another 2,000 grams of ethylene dichloride and decanted. The zwitterion phase was then reconstituted with sodium hydroxide to a pH of seven and adjusted to a 40% solid solution. The final product was noticeably improved in color and odor over a similar product prepared without an ethylene dichloride treatment.

Example II

To 331 grams of an aqueous solution which contained 100 grams of sodium β,β' -dodecylaminobispropionate ($\text{C}_{12}\text{H}_{25}\text{N}(\text{C}_2\text{H}_4\text{COONa})_2$) was added 169 grams of water. The mixture was heated to 60° C. and sufficient 35% sulphuric acid (37.3 cc.) was slowly added while continuing agitation to adjust the pH of the mixture to 3.5. Phase separation occurred and upon discontinuing agitation, the organic phase floated to the top of the aqueous phase and the latter was separated by decantation. Then 250 grams of ethylene dichloride were added to the organic phase which was heated to 80° C. to melt the zwitterion; the resulting mixture was then heated to boiling on a steam cone. The mixture was then cooled to room temperature with stirring whereupon the zwitterion precipitated. The mixture was filtered, and 10 grams of the filtrate evaporated to dryness on a steam cone. A residue of 0.0235 gram or 0.6% color, odor and cloudiness forming impurities was recovered. The zwitterion was reconstituted with sodium hydroxide to a pH of 7 and adjusted to a 40% solids solution. The final product was noticeably improved in color and odor over a similar product prepared without the ethylene dichloride treatment.

Example III

The process of Example I is repeated with sodium β dodecylaminocrotonate



with essentially the same results as Example I.

It is to be understood that the present invention is intended to cover all changes and modifications of the examples of the invention herein chosen for the purpose

of illustration, which do not constitute departure from the spirit and scope of the invention.

I claim:

1. The process of purifying the salt of a detergent amino acid of the formula $\text{RNH}_m(\text{CHR}'\text{CH}_2\text{COOMe})_n$ 5 where R is an aliphatic hydrocarbon group containing 8-22 carbon atoms, R' is selected from a group consisting of hydrogen and methyl, Me is a cation selected from the class consisting of sodium, potassium, and ammonium ions, m is not greater than 1, n is at least 1 and not more than 2 but 1 when R' is methyl and the sum of n and m is 2, which method comprises forming the zwitterion, separating said zwitterion, adding ethylene dichloride to said zwitterion, dissolving the zwitterion in the ethylene dichloride, cooling to cause separation of an ethylene dichloride phase, separating the ethylene dichloride and recovering the zwitterion. 10 15

2. The process of claim 1, where the zwitterion is formed by dissolving the salt of the detergent amino acid in water and adding sufficient nonoxidizing mineral acid to adjust the pH to 3.5-5.5. 20

3. The process of claim 2, where the nonoxidizing acid is sulphuric acid.

4. The process of purifying the salt of the detergent amino acid of the formula $\text{RNH}_m(\text{CHR}'\text{CH}_2\text{COOMe})_n$ 25

where R is an aliphatic hydrocarbon group containing from 8-22 carbon atoms, R' is selected from a group consisting of hydrogen and methyl, Me is a cation selected from the class consisting of sodium, potassium, and ammonium ions, m is not greater than 1, n is at least 1 and not more than 2 but 1 when R' is methyl and the sum of m and n is 2, which method comprises preparing a solution of the salt of the detergent amino acid, adjusting said solution to a pH within the range of 3.5-5.5 by use of a nonoxidizing mineral acid to form a two phase system, one of which contains the zwitterion of said amino acid, separating the zwitterion-containing phase, adding ethylene dichloride to said phase, dissolving the zwitterion in the ethylene dichloride, then cooling to cause separation of the zwitterion, separating the ethylene dichloride and finally reconstituting the zwitterion to a salt of the detergent amino acid.

5. The program of claim 4, where the nonoxidizing acid is sulphuric acid.

References Cited in the file of this patent

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