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ENCAPSULATED SYNTHETIC LIQUID DETERGENT AND PROCESS FOR PREPARING THE SAME

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12 Claims

ABSTRACT OF THE DISCLOSURE

Synthetic liquid detergents containing less than 1% water and as little as 0.15% water are encapsulated in gelatin. The detergent composition for encapsulation is obtained by azeotropic distillation of an anionic surface active agent in a solvent followed by addition of a non-ionic surface active agent, heating until a homogeneous solution is obtained and a further azeotropic distillation. The solvent is then removed and the composition encapsulated.

FIELD OF THE INVENTION

This invention relates to an encapsulated liquid detergent. This invention also relates to a process for preparing an encapsulated liquid detergent. This invention also relates to a process for preparing an encapsulated liquid detergent containing a predetermined quantity of active liquid detergent.

DESCRIPTION OF THE PRIOR ART

Present liquid detergents sold commercially have generally 20 to 40%, perhaps even 45% active material, balance water, except for a very minor amount of alcohol. As a consequence, present marketing methods are bulky and use considerable shelf space in retailing outlets. Also, as will be apparent, the mark-up involved at the several stages from production to retail is very uneconomical to the customer because of the large amount of water present.

Moreover, an inherent difficulty facing the customer resides in the problem of measuring the exact quantity of liquid detergent needed for a given purpose.

The possibility of encapsulating liquid detergents in a self-dissolving capsule has been proposed to overcome some of these problems. However, until now, encapsulation of liquid soaps has been successful only in a very specialized field, i.e. for perfumed oil. However, these formulations are totally unacceptable in the field of liquid detergents.

SUMMARY OF THE INVENTION

It is an object of the invention to provide a liquid detergent in capsule form. It is a further object of the invention to provide, in capsules, a highly concentrated liquid detergent composition that is clean, convenient to use and economical, containing a pre-determined amount of active material. It is a further object of the invention to provide a process for preparing said encapsulated liquid detergent.

In accordance with the present invention, an encapsulated liquid detergent is provided, having at least 85% activity and being encapsulated in a water-soluble capsule e.g. gelatin. The capsule obtained usually contains less than 1% moisture.

The product of the invention is obtained by preparing a composition by a process wherein, while the formation of a sodium salt is prevented, an alkylarylsulfonic acid anionic surface active agent is dissolved in an organic solvent comprising a compound forming an azeotrope with water; the mixture is dried; a nonionic surface active

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agent is added, if desired; the mixture is heated under reflux until a homogeneous solution is obtained and the solvent is removed. The resulting composition is then encapsulated in gelatin.

In a specific embodiment, the synthetic detergent composition is obtained as follows:

(1) An anionic surfactant e.g. an alkylarylsulfonic acid is treated to remove as completely as possible any sulfonating agent employed in the production of the anionic surfactant such as acid or SO_3 and water while preventing the formation of the sodium salt and while being dissolved in a solvent such as benzene or toluene, which forms an azeotrope with water. Preferred are alkylarylsulfonic acids wherein the alkyl group contains from 4 to 16 carbon atoms and the aryl group is benzyl, naphthyl or phenyl.

In a practical manner, the alkylarylsulfonic acid is dissolved in e.g. benzene, washing with water and with sodium bicarbonate, removing the water layer and drying. Drying is conveniently effected in two steps, the first one consisting in drying over e.g. calcium chloride, the second one, by azeotropic distillation until all water is removed.

(2) A mixture is made of the composition obtained in (1) with a nonionic surfactant (e.g. alkylaryl polyalkylene glycol) and an alkanolamine e.g. triethanolamine. This mixture is more conveniently made at about 40–45° C. For best results, a foam stabilizer is added (e.g. nonionic fatty acid alkanolamide) as well as small amounts of glycerine (which improves the physical condition of the capsule).

The mixture is then heated under reflux for approximately one hour, that is until

- (1) the pH reaches 7–7.5 or about
- (2) the mixture is homogeneous.

This is followed by a further azeotropic distillation to remove any water contained, e.g., in the nonionic surfactant or in the foam stabilizer. The azeotropic distillation is continued until the product is practically anhydrous.

(3) The solvent is then removed from the product thus obtained. This is conveniently done by applying vacuum in the case of benzene.

(4) The product thus obtained is encapsulated in a water-soluble capsule e.g. in gelatin.

As mentioned hereinbefore, formation of the sodium salt of the anionic surfactant must be prevented as otherwise the surfactant would precipitate. The sodium salt is formed at approximately pH 8. To retain the pH below this limit, agents such as alkylamines or alkanolamines can be used. Addition of alkanolamines also lowers the hardness of the surface active agent. Preferred are alkanolamines or alkylamines having a hydrocarbon radical containing from 2 to 6 carbon atoms such as ethanolamine, diethanolamine, triethanolamine, dimethylamine or trimethylamine.

DESCRIPTION OF PREFERRED EMBODIMENTS

The invention will now be described in further detail, with reference to the following non-limiting examples, wherein parts are by weight unless otherwise indicated.

Example I

45 gr. dodecylbenzenesulfonic acid are added to 200 cc. benzene and the solution is washed twice with 50 cc. water, then with 50 cc. of a 5% solution of sodium bicarbonate, then again with 50 cc. water. The water layer is removed, dried over sodium sulfate or calcium chloride and subjected to an azeotropic distillation. The benzene is then removed in vacuum and replaced with an equal amount of fresh benzene. The solution is heated to 40° C.–45° C. and a solution of 37.5 gr. nonionic sur-

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factant sold under the trademark Tergitol NPX (a non-ionic nonyl phenol polyethylene glycol having 10.5 mol of ethyl oxide, a cloud point of 60–65° C., specific gravity 20/20° C.: 1.06, solidification temperature: 5–7° C. and surface tension—0.10% aqueous solution at 25° C.: 32.3 dynes/cm.) is added slowly with 1.0 gr. triethanolamine, stirred for 15 minutes and 2 gr. laurylamide and 1.5% glycerine are added. The mixture is refluxed for 1 hr., then, is subjected to an azeotropic distillation. The solvent is evaporated in vacuum at 45° C., then 3% alcohol is added and the solution is stirred for 30 minutes at 45° C.–50° C.

This synthetic detergent is ready for encapsulation.

Example II

Part I.—37.5 gr. of a nonionic surface active agent sold under the trademark Triton X-100 (octyl phenoxy polyethoxy ethanol by inserting 9–10 moles of ethylene oxide) is heated to 40° C.–45° C. and 22.0 gr. of triethanolamine is added.

Part II.—To 45 gr. dodecylbenzenesulfonic acid is added 150 cc. benzene, the solution is washed with water and then with a 5% solution of sodium bicarbonate. This solution is then removed and the balance is washed again with water. The water layer is then discarded and the remainder is dried over sodium sulfate or calcium chloride. The thus dried solution is then subjected to azeotropic distillation, and, then, the benzene is replaced by the same amount of fresh benzene. At 45° C.–50° C., under good agitation, Part II is added to Part I. After this introduction is terminated, the mixture is refluxed for 1 hr. and then, the solvent is distilled off at normal pressure. The resulting solution is subjected to vacuum for ½ hr. at 45° C., and at this temperature, a solution of alcohol, alkanolamide and glycerine is added and stirred in for ½ hr.

This synthetic detergent is ready for encapsulation.

Example III

A solution of 330 g. of tert-butylphenylsulfonic acid in 600 ml. of benzene is washed with a 5% sodium carbonate solution and several times with water in order to bring the pH to 5–6. The solution is dried over sodium sulfate for 12 hrs. The solvent is removed under reduced pressure and replaced with dry benzene. A solution of 235 g. of triethylamine in 200 ml. of benzene is added under efficient agitation in 30 minutes at a temperature of 35° C.–40° C. The resulting solution is stirred for 30 minutes and treated in one portion, with a solution of 72 g. of nonylphenylpolyethylene glycol in 100 ml. of benzene and 22 g. of glycerol. After refluxing for 1 hr. the solvent is dried by an azeotropic distillation and the solvent removed at reduced pressure. Perfume (0.2%) and absolute alcohol (15 ml.) is then added. The pH of the solution (0.1%) is approximately 7.5.

Example IV

To a solution of 65 g. of octylphenylsulfonic acid in 200 ml. of ethylenedichloride previously washed according to Example I or II is added in 30 minutes a mixture of 12 g. of laurylethoxylate in 50 ml. of ethylenedichloride. Then 23 g. of diethanolamine are added in 30 minutes at a temperature of 30° C.–35° C. The solution is then refluxed for 1 hr., then, the solvent is removed under reduced pressure. 200 ml. of benzene are added to the viscous solution and the solvent is dried by an azeotropic distillation and then removed at reduced pressure. 10 ml. of abs. alcohol, 8 g. of glycerol and 0.2% of perfume are then added to the mixture which is stirred vigorously for 30 minutes at 40° C.–45° C. The pH of the resultant solution is approximately 7.7.

Example V

Through a solution of 200 ml. of toluene previously cooled to 5° C. is bubbled 75 g. of trimethylamine. Then

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a solution of 42 g. of nonylphenyl-sulfonic acid in 100 ml. of toluene is slowly added under vigorous agitation during a period of 1 hr. The temperature is maintained at between 10° C.–15° C. The resulting solution is stirred for 30 minutes and the temperature is raised to 25° C., then treated in one portion with a solution of 10 g. of nonylphenylpolyethylene glycol, 5 g. of glycerol and 75 ml. of toluene. After refluxing for 1 hr., the solvent is dried by an azeotropic distillation and the solvent removed at reduced pressure. 10 m. of abs. alcohol and 0.2% of perfume are added and stirred until the viscous solution is homogeneous. The pH of the resultant solution is approximately 7.6.

The product of the invention has the following characteristics:

Activity—95% or more

Color—Amber

Odor—None or slight perfume

Cloud point—15° C.

Texture—Viscous slurry

pH (1% aq. sol.)—Between 6.5 and 8.0—usually about 7.5

Stability in hard water—Good

Stability in sea water—Good

Effect of change of temperature—When cooled to 0° C. and warmed to 40° C. for 24 hrs. the product remains homogeneous and stable.

Foaming ability—20–22 ml.

The product, for encapsulation, must contain less than 1% water, preferably less than 0.5%. By the process described hereinabove, it was found possible to obtain as little as 0.15% water in the product.

Alcohol and/or glycerine are preferably added to the synthetic detergent before encapsulation. Glycerine is a softening agent which also acts as a preservative or barrier for the gelatin capsule. Alcohol traditionally acts as a binder between anionic and nonionic surfactants and is also, to a slight degree, a dehydrating agent. Both optional ingredients are recommended since they facilitate the production of a homogeneous mixture and/or the encapsulation.

The encapsulation in gelatin of the products obtained as hereinbefore described can be made in accordance with known methods. According to a preferred method, gelatin and, if desired, glycerine are first blended. Dyes can also be added if desired. The mass is then melted under a high vacuum to eliminate entrapped air. The liquid melted mass is then formed into two continuous ribbons of gelatin which are then shaped and filled with the liquid detergent described. The resulting filled halves of the capsule are then sealed under temperatures not higher than 37° C. and the sealed capsules are washed with alcohol and dried.

I claim:

1. An encapsulated synthetic liquid detergent composition at least 85% active, the synthetic liquid detergent component of said composition consisting essentially of, by weight,

(a) 33–65% alkylarylsulfonic acid wherein the alkyl group contains from 4 to 16 carbon atoms and the aryl group is benzyl, naphthyl or phenyl,

(b) 8–36.5% nonionic surface active agent, and

(c) 20–59% alkanolamine or alkylamine wherein the alkyl or alkanol group contains from 2 to 6 carbon atoms, said composition containing less than 1% water, encapsulated in gelatin.

2. An encapsulated synthetic liquid detergent composition at least 85% active, the synthetic liquid detergent component of said composition consisting essentially of, by weight,

(a) 33–65% alkylarylsulfonic acid wherein the alkyl group contains from 4 to 16 carbon atoms and the aryl group is benzyl, naphthyl or phenyl,

(b) 8–36.5% nonionic surface active agent, and

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(c) 20-59% alkanolamine or alkylamine wherein the alkyl or alkanol group contains from 2 to 6 carbon atoms, said composition containing less than 0.5% water, encapsulated in gelatin.

3. An encapsulated synthetic liquid detergent composition at least 85% active, the synthetic liquid detergent component of said composition consisting essentially of, by weight,

(a) 33-65% alkylarylsulfonic acid wherein the alkyl group contains from 4 to 16 carbon atoms and the aryl group is benzyl, naphthyl or phenyl,

(b) 8-36.5% nonionic surface active agent, and

(c) 20-59% alkanolamine or alkylamine wherein the alkyl or alkanol group contains from 2 to 6 carbon atoms, said composition containing less than 1% water and a small but significant amount up to 7.5% of anhydrous glycerol, encapsulated in gelatin.

4. An encapsulated synthetic liquid detergent composition at least 85% active, the synthetic liquid detergent component of said composition consisting essentially of, by weight,

(a) 33-65% alkylarylsulfonic acid wherein the alkyl group contains from 4 to 16 carbon atoms and the alkyl or alkanol group contains from 2 to 6 carbon

(b) 8-36.5% nonionic surface active agent, and

(c) 20-59% alkanolamine or alkylamine wherein the alkyl or alkanol group contains from 2 to 6 carbon atoms, said composition containing 0.15-0.5% water and a small but significant amount up to 7.5% of anhydrous glycerol, encapsulated in gelatin.

5. An encapsulated synthetic liquid detergent as claimed in claim 1, wherein said nonionic surface active agent is an alkylaryl polyalkylene glycol.

6. An encapsulated synthetic liquid detergent composition at least 85% active, the synthetic liquid detergent component of said composition consisting essentially of, by weight,

(a) 33-65% alkylarylsulfonic acid wherein the alkyl group contains from 4 to 16 carbon atoms and the aryl group is benzyl, naphthyl or phenyl,

(b) 8-36.5% nonionic surface active agent selected from the group consisting of alkylaryl polyalkylene glycol laurylamide and lauryl ethoxylate, and

(c) 20-59% alkanolamine or alkylamine wherein the alkyl or alkanol group contains from 2 to 6 carbon atoms, said composition containing less than 1% water and a small but significant amount up to 7.5% of anhydrous glycerol, encapsulated in gelatin.

7. Process for preparing an encapsulated synthetic liquid detergent composition comprising dissolving an alkylarylsulfonic acid free from free acid wherein the alkyl group contains from 4 to 16 carbon atoms and the aryl group is benzyl, naphthyl or phenyl, in benzene or tolu-

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ene, adding a liquid nonionic surface active agent, heating the mixture under reflux until a homogeneous solution is obtained, subjecting the solution to azeotropic distillation to remove water to a maximum of 1% by weight of the composition, removing the solvent by distillation under reduced pressure, and encapsulating the resulting composition in gelatin.

8. Process for preparing an encapsulated synthetic liquid detergent composition comprising dissolving an alkylarylsulfonic acid wherein the alkyl group contains from 4 to 16 carbon atoms and the aryl group is benzyl, naphthyl or phenyl, in benzene or toluene, treating said acid to remove free acid, heating the resulting solution under reflux until a homogeneous solution is obtained, subjecting the homogeneous solution to azeotropic distillation to remove water to a maximum of 1% by weight of the composition, removing the solvent by distillation under reduced pressure and encapsulating the resulting composition in gelatin.

9. Process as defined in claim 8, wherein said acid is treated to remove free acid by adding an alkylamine or an alkanolamine containing 2 to 6 carbon atoms in the alkyl or alkanol group and allowing the products to react until a pH of 6.5 to 7.5 is obtained.

10. Process as defined in claim 9, which comprises adding a liquid nonionic surface active agent previous to heating under reflux.

11. Process as claimed in claim 10, which further comprises adding anhydrous glycerin before encapsulation.

12. An encapsulated synthetic liquid detergent as claimed in claim 1, wherein said nonionic surface active agent is a member selected from the group consisting of laurylamide and lauryl ethoxylate.

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