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3,786,003

LIQUID DETERGENT COMPOSITIONS

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8 Claims

ABSTRACT OF THE DISCLOSURE

Pourable, liquid alcohol ethoxysulfate compositions are prepared from solid alcohol ethoxysulfates by combining with selected amounts of water and a sodium or potassium salt.

BACKGROUND OF THE INVENTION

The present invention is related to the preparation of detergent formulations wherein a detergent active ingredient is an anionic alcohol ethoxysulfate compound which is normally solid at substantially room temperature. In the preparation of detergent formulations, the physical state of the ingredients has an important bearing on the ease with which the formulation process may be carried out. Materials which are solid are more difficult to handle and more difficult to blend uniformly than are liquid materials.

It is known in the art that normally solid anionic alcohol ethoxy sulfates can be converted to pourable liquids by blending therewith selected amounts of aqueous ethanol solutions. However, the presence of ethanol precludes the use of such pourable liquid alcohol ethoxy sulfate formulations in the manufacture of dry particulate detergent compositions which are generally dried by spraying an aqueous slurry of the desired composition into a hot gas stream, heated at a temperature of 350-750° F. The use of ethanol-containing aqueous slurry in such a spray drying operation would create potentially hazardous explosions.

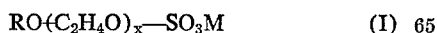
The conversion of normally solid nonionic alcohol ethoxylates into pourable liquids without the use of potentially hazardous volatile organic compounds is disclosed in U.S. Pat. 3,419,500 of Rytter et al.

SUMMARY OF THE INVENTION

It has now been found that certain normally solid anionic alcohol ethoxysulfates can be blended with pre-selected amounts of water and a sodium or potassium salt selected from chloride, bromide, sulfate or mixture thereof to produce a composition which is pourable and liquid at 80° F. and contains about 38 to 42% by weight of the alcohol ethoxysulfate, about 7 to 10% by weight, preferably 8 to 9% by weight, of the sodium or potassium salt and the balance water.

DESCRIPTION OF PREFERRED EMBODIMENTS

The alcohol ethoxysulfates.—The alcohol ethoxysulfate components of the invention comprise sulfuric acid salts of a mixture of ethylene oxide derivatives of at least one alcohol of 11 to 15 carbon atoms wherein the average number of ethylene oxide units per molecule is from about 2 to about 5. More particularly, the alcohol ethoxysulfates are represented as a mixture of compounds of the average Formula I:

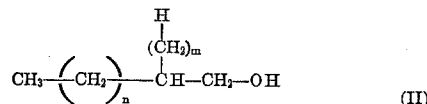


wherein R is primary alkyl of from 11 to 15 carbon atoms, M is an alkali metal, preferably sodium or potassium, x is a number from about 2 to about 5 representing an average of the ethylene oxide units per alcohol ethoxysulfate molecule.

Broadly speaking, the alcohol ethoxysulfates are deriv-

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atives of primary alcohols of carbon numbers from 11 to 15. The alcohol precursors of the alcohol ethoxysulfate are straight-chain alcohols or are of a branched-chain structure but when branched it is preferred that the sole chain-side branching be that of a single alkyl substituent in the beta or 2 position. One class of such alcohols is the class of primary alcohols of the formula ROH where R has the previously stated significance, which class is additionally represented by the Formula II:



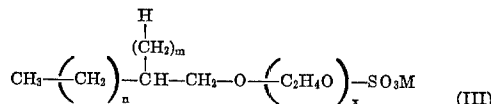
where m is a whole number from 0 to 5 inclusive and n is a whole number from 3 to 12 inclusive selected so that the sum of m and n, i.e., the term (m+n), is a whole number from 8 to 12 inclusive.

The above Formula II provides for alcohols which are straight-chain or which have an alkyl branch which is methyl, ethyl, propyl, butyl or amyl, and in many instances at least a minor proportion of alcohol of each type is present. At least a substantial proportion of the alcohol of each carbon number within the 11 to 15 range is of the straight-chain structure, i.e., the compound of the above Formula II wherein m is 0, and in preferred modifications at least 70%, more preferably at least about 80% of the alcohols are straight-chain in structure. Of any alcohols that are branched, the predominant type is that having a beta-methyl branch and typically about 50% of the branched alcohols are beta-methyl alcohols with lesser amounts of the other branched-chain isomers.

The straight-chain alcohols of Formula II (where m is 0) are derived from fats, e.g., coconut oils, or are synthetic. Synthetic straight-chain alcohols are produced, for example, by the oxidation of aluminum trialkyls and the hydrolysis of the resulting aluminum trialkoxides by an aqueous reagent, as disclosed in U.S. Pat. 3,391,175 of Davis.

The alcohols of Formula II containing branching in the beta or 2-position, or the aldehydes corresponding thereto, are produced by the hydroformylation of straight-chain olefins of 10 to 14 carbon atoms as described in, for example, U.S. Pats. 3,420,898 and 3,440,291. In the hydroformylation processes, both straight-chain and branched products are formed. The product mixtures within the desired range of proportions are useful as such or alternatively the individual components are separated by conventional procedures and recombined to provide a mixture of any desired proportion of alcohol types.

In terms of the above Formulas I and II, the preferred derivatives are represented by the Formula III:



wherein m, n, M and x have the previously stated significance. It should be appreciated that there will be present ethoxysulfate derivatives of at least one alcohol of a carbon number of 11, 12, 13, 14 or 15, that is, ethoxysulfate derivatives of one or more C₁₁ to C₁₅ alcohols. In the case of mixtures of derivatives of alcohols of differing carbon number, the relative proportions of derivatives of alcohol of each carbon number are not critical. Preferred mixed compositions comprise no more than about 75% by weight of derivatives of any one alcohol within the C₁₁ to C₁₅ range, more preferably, from about 10% to about 40% by weight on the same basis.

The alcohol ethoxysulfates are produced in part by more or less conventional methods, i.e., the alcohols are reacted with 2 to 5, preferably 3, molar proportions of

ethylene oxide and the resulting ethoxylate mixture is sulfated and neutralized.

Suitable ethoxylation results are obtained by adding to the alcohol or mixture of alcohols to be ethoxylated a calculated amount, e.g., from about 0.1% by weight to about 0.6% by weight, preferably from about 0.1% to about 0.4%, based on total alcohol, of a strong base, typically an alkali metal hydroxide such as sodium hydroxide or potassium hydroxide, which serves as catalyst for the ethoxylation. The resulting mixture is dried, as by vapor phase removal of any water present, and an amount of ethylene oxide calculated to provide from about 2 to about 5 moles of ethylene oxide per mole of alcohol is then introduced and the resulting mixture is allowed to react until the ethylene oxide is consumed, the course of the reaction being followed by the decrease in reaction pressure.

The ethoxylation is conducted at an elevated temperature and pressure. Suitable reaction temperatures are from about 120° C. to about 220° C. with the range of from about 140° C. to about 160° C. being preferred. A suitable reaction pressure is achieved by introducing to the reaction vessel the required amount of ethylene oxide which has a high vapor pressure at the desired reaction temperature. The pressure serves as a measure of the degree of reaction and the reaction is considered to be substantially complete when the pressure no longer decreases with time.

It should be understood that the ethoxylation procedure serves to introduce a desired average number of ethylene oxide units per alcohol ethoxylate molecule. For example, treatment of an alcohol mixture with 3 moles of ethylene oxide per mole of alcohol serves to effect the ethoxylation of each alcohol molecule with an average of 3 ethylene oxide moieties per mole alcohol moiety, although a substantial proportion of alcohol moieties will become combined with more than 3 ethylene oxide moieties and an approximately equal proportion will have become combined with less than 3. In a typical ethoxylation product mixture, there is also a minor proportion of unreacted alcohol.

To prepare the alcohol ethoxysulfate component of the invention, the alcohol ethoxylate products are sulfated with a suitable sulfating reagent such as sulfur trioxide, and the resulting sulfated product mixture is neutralized with an alkali metal solution, e.g., sodium or potassium hydroxide. Suitable sulfation procedures are disclosed in U.S. Pats. 3,259,645 and 3,350,428.

The sodium or potassium salt.—Suitable sodium or potassium salts are NaCl, NaBr, KCl, KBr, Na₂SO₄, K₂SO₄ and mixtures thereof. Preferred salts are sodium chloride and sodium sulfate.

The pourable liquid compositions of the invention are prepared by mixing preselected amounts of alcohol ethoxysulfate, sodium or potassium salt and water to produce a composition having the following weight composition: about 38 to 42% alcohol ethoxysulfate, about 7 to 10%, preferably 8 to 9%, sodium or potassium salt and the balance water.

In a preferred modification, the liquid pourable compositions are prepared by neutralizing the corresponding alcohol ethoxysulfuric acid with a solution containing preselected amounts of an alkali metal hydroxide (e.g., sodium hydroxide), as well as the sodium or potassium salt and water to produce a pourable liquid composition containing the amounts of alcohol ethoxysulfate, sodium or potassium salt and water defined above.

In preparing the pourable compositions potassium or sodium salts having water of crystallizations, e.g., Na₂SO₄·10H₂O, are suitably employed.

EXAMPLE I

A mixture of alcohol ethoxysulfuric acid was prepared by sulfation of a commercial alcohol ethoxylate mixture (marketed by the Shell Chemical Company as Neodol®

25-3 Alcohol Ethoxylate). The alcohol ethoxysulfate mixture has an average of 3 ethylene oxide units per molecule and is derived from a mixture of C₁₂, C₁₃, C₁₄ and C₁₅ primary alcohols, in substantially equal proportions by weight, wherein approximately 80% of the alcohol of each carbon number was straight-chain, approximately 10% of the alcohol of each carbon number was beta-methyl branched and the remainder of the alcohol of each carbon number comprised alcohols of beta-ethyl, beta-propyl, beta-butyl or beta-amyl branching. The sulfation was conducted by treating the alcohol ethoxylate mixture in a continuous reactor (19 inches long) with a sulfur trioxide/nitrogen gas mixture under the following conditions:

Reaction temperature ° C.	50
SO ₃ , gram/min.	2.5
SO ₃ /alcohol ethoxylate mole ratio	1.04
N ₂ /SO ₃ volume ratio	40/1

EXAMPLE II

A 765.5 g. sample of the free ethoxysulfuric acid prepared as described in Example I was added to a mixture of 72 g. sodium hydroxide, 147.4 g. sodium sulfate and 862.1 g. water at a temperature of 50-60° C. The resulting mixture was a moderately viscous liquid which could be stirred efficiently throughout the entire addition period. The ingredients were contacted in proportions calculated to produce a blend having 40% w. alcohol ethoxysulfate, 8% w. sodium sulfate and 52% w. water. Analysis of the blend showed 40.3% w. alcohol ethoxylate. The blend had a Klett (5%) color of 23 and was a pourable liquid at a temperature of 80-85° F.

EXAMPLE III

A 745.7 g. sample of the free alcohol ethoxysulfuric acid prepared as described in Example I was neutralized by adding it to a mixture of 62 g. sodium hydroxide, 147.4 g. of sodium sulfate and 862.1 g. of water. The amounts of reactants was calculated to produce a blend of 42% w. alcohol ethoxysulfate, 8% w. sodium sulfate and 50% w. water. Analysis of the blend showed 41.7% w. alcohol ethoxysulfate. The blend had a pH of 8.5, a Klett (5%) color of 25 and was a pourable moderately viscous liquid at a temperature of 80-85° F.

EXAMPLE IV

A 5.07 g. sample of a soft aqueous gel consisting of 78.9% w. of the sodium salt of the ethoxysulfuric acid prepared in Example I and 21.1% w. water was mixed by hand with 0.8 g. of sodium sulfate and 4.13 g. of distilled water. The ingredients were mixed in proportions calculated to produce a blend with the composition: 40% w. alcohol ethoxysulfate, 8% w. sodium sulfate and 52% w. water. By a similar procedure a variety of compositions were prepared. The weight concentration of alcohol ethoxysulfate and sodium sulfate and the physical state thereof at 80° F. are tabulated in Table I.

TABLE I

Na ₂ SO ₄ , percent w.	Alcohol ethoxysulfate, percent w.			
	29	38	40	42
0..... (1)				Non-pourable stiff gel
6.....				Barely pourable viscous liquid
8.....				Pourable moderately viscous liquid
10.....				Pourable moderately viscous liquid
12.....				Hazy, non-homogeneous two-phase viscous liquid

¹ Pourable liquid.

EXAMPLE V

A 5.32 g. sample of a soft aqueous gel consisting of 78.9% w. of the sodium salt of the ethoxysulfuric acid prepared in Example I and 21.1% water was mixed with 0.8 g. sodium chloride and 3.88 g. of water. The resulting composition was a pourable, slightly hazy, moderately viscous liquid (80° F.) with the composition (calcu-

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lated): 42% w. alcohol ethoxysulfate, 8% sodium chloride and 50% w. water. By a similar procedure a variety of compositions were prepared. The weight concentration of alcohol ethoxysulfate and sodium chloride and the physical state of the compositions at 80° F. are tabulated in Table II.

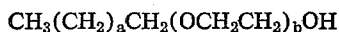
TABLE II

NaCl, percent w.	Alcohol ethoxysulfate, percent w.			
	29	38	40	42
0..... (1)---				Non-pourable stiff gel
6.....				Barely pourable viscous liquid
8.....				Pourable moderately viscous liquid
10.....				Pourable moderately viscous liquid
12.....				Hazy, non-homogeneous two-phase viscous liquid

¹ Pourable liquid.

EXAMPLE VI

By a procedure similar to that of Example I a mixture of alcohol ethoxysulfuric acid was prepared by sulfation of a commercial alcohol ethoxylate mixture (marketed by Continental Oil Company as Alfonic® 1412-40 Ethoxylate) having the structural formula



wherein the approximate value of a is 11 and b is 3.

A 3.39 g. sample of a soft aqueous gel, consisting of 59% of the sodium salt of the above ethoxysulfuric acid and 41% water, was mixed with 0.4 g. sodium sulfate and 1.21 g. water. The resulting pourable, clear moderately viscous liquid (at 80° F.) has a composition consisting of 40% w. alcohol ethoxysulfate, 8% w. sodium sulfate and 52% w. water.

EXAMPLE VII

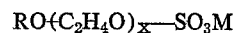
A 2.58 g. sample of a soft aqueous gel, consisting of 77.5% of the sodium salt of the ethoxysulfuric acid prepared in Example I and 22.5% water, was mixed with 0.4 g. of potassium bromide and 2.02 g. distilled water. The resulting blend, consisting of 40% w. alcohol ethoxysulfate, 8% w. potassium bromide and 52% w. water, was a pourable, moderately viscous liquid (at 80° F.).

I claim as my invention:

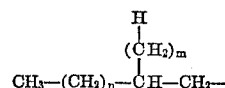
1. A composition, pourable and liquid at a temperature of about 80° F., consisting essentially of (1) about 38 to 42% by weight, based on total composition, of a nonionic alcohol ethoxysulfate (2) about 7 to 10% by weight, based on total composition, of a salt selected from NaCl, KBr, Na_2SO_4 or a mixture thereof and (3) the balance water, said alcohol ethoxysulfate being a

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solid at a temperature of about 80° F. and being represented by the formula



wherein M is sodium or potassium, X is a number from 2 to 5 representing an average of the ethylene oxide units per molecule and R is alkyl of from 11 to 15 carbon atoms of the formula



wherein m is a whole number from 0 to 5 inclusive and n is a whole number from 3 to 12 inclusive so that the sum of $m+n$ is a whole number from 8 to 12 inclusive.

2. The composition of claim 1 wherein the salt is a sodium salt.

3. The composition of claim 2 wherein m is 0 in about 80% of said alkyls and no more than 75% by weight of R is alkyl of any one number of carbon atoms.

4. The composition of claim 3 wherein M is sodium and x is 3.

5. The composition of claim 4 wherein the percentage by weight of salt is about 8 to 9% and the percentage by weight of alcohol ethoxysulfate is about 40%.

6. The composition of claim 5 wherein R is alkyl of from C_{12} to C_{15} .

7. The composition of claim 2 wherein m is 0 in essentially 100% of said alkyls, x is 3 and M is sodium.

8. The composition of claim 7 wherein the percentage by weight of salt is about 8 to 9% and the percentage of alcohol ethoxysulfate is about 40%.

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U.S. Cl. X.R.

252-550