

Concentrated Aqueous Surfactant

The present invention relates to concentrated aqueous surfactants and in particular to highly concentrated pourable alkyl sulphates having mixed cations.

It has long been known that alkyl sulphates are normally fully dispersible in water to form fluid micellar solutions (the L_1 phase) at concentrations up to about 30% Active (i.e. 30% by weight of surface active matter based on the total weight of solution). At higher concentrations phase changes occur leading to the formation of viscous liquid crystal phases. The majority of alkyl sulphates form an essentially immobile liquid crystal phase (the M_1 phase) at concentrations substantially above about 35 to 40% Active. At higher concentrations further phase changes occur culminating in the formation of a hydrated solid phase at concentrations typically around 80 to 100% Active.

It will be understood that the concentrations at which the foregoing phase changes occur may vary considerably according to the nature of the alkyl groups, and the cations present.

The nature of the phases formed at different concentrations are also profoundly altered by the presence of any substantial amounts of non active material such as electrolytes, solvents and phase modifiers.

Until recently it was assumed that the concentration corresponding to the upper boundary of the L_1 phase (typically about 30-35% Active) represented the maximum concentration at which it was practicable to supply binary mixtures of alkyl sulphate and water, since the immobile M_1 phase and hydrated solid are

difficult and intractable materials to handle, dilute or mix with other ingredients. Higher pourable concentrations could sometimes be obtained by adding substantial quantities of solvents such as ethanol or of phase modifiers such as urea. These greatly increase the cost of the composition, however, and were not always compatible with customers' desired end use formulations.

Accordingly there was generally no alternative but to accept the disadvantages of transporting and storing two parts of water for every part of alkyl sulphate.

In our BPl,488,352 we described how certain alkyl sulphates, namely those of ammonium, amines and mixtures thereof, form a pourable liquid crystal phase (herein referred to as the "G" phase) at ambient temperature and at concentrations above the upper boundary of the M_1 phase but below the lower boundary of the hydrated solid phase (typically between about 65% and about 75% active).

In particular in example 4 of the aforesaid British patent there is described a product which is a highly concentrated, pourable mixture of ammonium, monoethanolamine and triethanolamine alkyl sulphates, an analog of which has proved of commercial value as an ingredient of shampoos and cleaning formulations. The viscosity, on dilution of the aforesaid product according to the Patent Example, is undesirably high, requiring the addition of some inorganic salts such as chloride and/or sulphate to provide the commercial product, which meets desired specifications. This commercial product has a setting point of about 24-26°C which is high enough to cause some inconvenience in handling the product in certain circumstances.

We have now discovered a novel composition which generally matches the aforesaid product in performance, but which has a substantially lower setting point, and which, on dilution, generally provides solutions of satisfactory viscosity with substantially reduced need for extraneous additives. The novel product can be handled commercially at significantly higher Active concentrations than the aforesaid product of Example 4 of our British patent or its commercial analog.

Our invention provides a composition consisting essentially of an aqueous solution of a mixed ammonium, monoethanolamine, isopropanolamine alkyl sulphate wherein the alkyl group has an average of from 10 to 18 carbon atoms, said composition having a concentration such that it is a pourable composition consisting, at least predominantly "G" phase at normal ambient temperature.

Typically the total proportion of base used to prepare the product of the invention is sufficient to provide a pH, measured on an aqueous solution of the product containing 2% solids, of from 4 to 8 preferably 5 to 7, more preferably 5.5 to 6.5 e.g. 5.9 to 6.2.

The proportions of isopropanolamine and of ammonia, measured as concentrated aqueous ammonium hydroxide solution of specific gravity 0.88, are typically each from 2 : 1 to 4 : 1 by weight based on the weight of monoethanolamine, preferably 2.5 : 1 to 3.5 : 1, e.g. 2.8 : 1 to 3.2 : 1.

The product is preferably prepared by neutralising a C¹²⁻¹⁴ alkyl sulphuric acid. Preferably the Active matter in the

composition has a mean molecular weight of from 290 to 350, more preferably 300 to 340, typically 310 to 330 e.g. 315 to 325.

The compositions of our inventions will usually contain small amounts of non surface active matter, such as unsulphated fatty alcohols, which are normally present in commercial alkyl sulphates. We prefer however that the total proportion of non-active non-ionic organic matter be less 10% of the weight of the composition more preferably less than 5% e.g. less than 3.5%. Inorganic materials, and especially inorganic salts such alkali metal and/or, preferably ammonium sulphate or chloride, may be added to the composition in minor amounts, for example to adjust the viscosity of solution formed on dilution. In general we prefer that the total non-surfactant salt content of the composition should not exceed 10% and should preferably be less than 5% by weight, e.g. less than 3%.

In general non-active material is not added to compositions according to our invention in amounts sufficient to suppress altogether the formation, at lower active concentrations of an immobile M_1 phase. We have found that products with particularly satisfactory properties can be prepared by including 1.2 to 2.8% by weight of sulphate and less than 0.1% chloride, without any added organic solvent or phase modifier, other than the residual, unsulphated fatty alcohol in amounts below 3.5% by weight.

Compositions according to our invention may conveniently be adjusted by additions of inorganic salt to provide a viscosity of 35 ± 15 cS at 20°C , on dilution to 16% Active concentration.

The Active concentration of the compositions of our invention will depend upon the concentration corresponding to the M_1/G phase transition and the concentration of the G phase/hydrated solid phase transition, the composition having a concentration above the former phase transition and below the latter. Preferably the composition has a concentration which corresponds substantially to the viscosity minimum which lies between the aforesaid phase transition concentrations, preferably within $\pm 10\%$ of the minimum more preferably $\pm 5\%$ e.g. $\pm 2\%$.

The actual concentrations at which the viscosity minimum and the two phase transitions fall, will vary according to particular alkyl sulph^{ur}onic acid, the proportions of the bases and the amount of non-active matter. Typically the M_1/G phase transition occurs above 45%, usually above 50% e.g. between 60 and 70% by weight active concentration. The G phase/hydrated solid transition usually occurs above 60% e.g. between 65 and 85% Active concentration. The presence of inorganic electrolytes tends to lower both the maximum and minimum concentrations at which a G phase is formed, the latter being lowered by a greater amount than the former, giving a wider range of concentrations which form the G phase and a narrower range which form the M_1 phase.

We have found that with compositions containing monoethanolamine isopropanolamine and 0.880 s.g. ammonia in proportions of about 1:3:3, by weight, having a mean molar weight of 320 and containing 1.2% sulphate the M_1/G transition occurs at 65% Active and the G/hydrated solid transition at 80% Active concentration. At 2.8% sulphate the M_1/G transition occurs at 59% Active.

The concentrations may readily be determined for any particular sample by preparing a product at say 70% Active concentration and observing the sample through a polarising microscope.

Characteristic textures are observed according to the phase present. These may be identified by comparison, for example with the examples in the paper by Rosevear, JAOCS Vol. 31, P628 (1954) or with J. Colloid and Interfacial Science, Vol. 30, No. 4, P500. Water may be added to, or evaporated from the sample until the desired phase transition is observed.

Preferably compositions according to our invention are manufactured by neutralising the appropriate alkyl sulph^{ur}onic acid with an aqueous mixture of bases containing the appropriate amount of water to form the product at the desired G phase concentration. We do not however exclude sequential neutralisation.

Any additives which are required may be added subsequently or, preferably, may be included in the initial reaction mixture.

The invention is illustrated by the following example:-

Example

The example was prepared using a loop neutraliser as described in our aforesaid British patent. A sulphated mixture of dodecyl and tetradecyl normal primary alcohols was used as the acid feed while the base feed comprised a mixture of 9.3g monoethanolamine, 28.9g isopropanolamine and 26.1g concentrated ammonia solution (S.G. 0.880)

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in 61.7g water. The temperature was maintained at 35°C and the product manufacture rate was 12g/min. The pourable G phase product contained 75.6% of active ingredient having a mean molar weight of 320, 2.7% of free fatty matter and 0.9% of sulphate as SO_4^{--} ion. To this material was added sufficient aqueous ammonium sulphate to affect an adjustment in analysis to 72.0% active ingredient and 1.9% sulphate as SO_4^{--} ion. This final product was a pourable G phase having a setting point of about 12°C, Dilution viscosity was 29 CS at 20°C measured on a 16% Active solution.



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Claims

1. A concentrated aqueous alkyl sulphate surfactant having a C₁₀₋₁₈ alkyl group and having a concentration such that it is present as G phase, wherein the cations comprise a mixture of ammonium, monoethanolammonium and isopropanolammonium.
2. A composition according to claim 1 having a pH, when diluted with water to 2% solids, of from 4 to 8..
3. A composition according to claim 2 having a pH, when diluted with water to 2% solids, of from 5.5 to 6.5.
4. A composition according to any foregoing claim wherein the proportion of isopropanolamine is from 2:1 to 4:1 based on the weight of monoethanolamine.
5. A composition according to any foregoing claim wherein the proportion of ammonia, measured as concentrated aqueous ammonium hydroxide solution of specific gravity 0.88 is from 2:1 to 4:1 based on the weight of monoethanolamine.
6. A composition according to any foregoing claim wherein the alkyl groups of the alkyl sulphate anions have an average of from 12 to 14 carbon atoms.
7. A composition according to any foregoing claim containing less than 5% by weight of non surface active, non-ionic organic matter and less than 5% by weight of non-surface active salts.
8. A composition: consisting essentially of water, and a mixed ammonium, monoethanolamine, isopropanolamine alkyl sulphate; having a pH of from 5 to 7, when diluted to a concentration of 2% by weight solids; said alkyl group having an average of from 10 to 14 carbon atoms; said ammonia, measured as concentrated aqueous ammonium hydroxide of 0.88 specific gravity, and said isopropanolamine, each being present in a proportion of from 2.5:1 to 3.5:1, based on the

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weight of monoethanolamine; said composition containing less than 5% each of non-surfactant, non-ionic organic matter and non-surfactant salts, and having a concentration greater than that corresponding to the M₁ phase/G phase transition, but below that corresponding to the G phase/hydrated solid transition.

9. An aqueous alkyl sulphate salt composition substantially as herein described with reference to the Example.

10. A method for the preparation of a composition according to any foregoing claim which comprises reacting an alkyl sulphuric acid simultaneously, or sequentially in any order, with aqueous ammonia, isopropanolamine and monoethanolamine in the presence of sufficient water to maintain the product in the G phase.
