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Hewitt et al.

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[54] **AQUEOUS CONCENTRATE DETERGENT COMPONENT**

[75] Inventors: **Thomas Hewitt**, Maryport; **Douglas Edward Mather**, Whitehaven; **Edward Tunstall Messenger**, High Harrington, all of England

[73] Assignee: **Albright & Wilson Limited**, West Midlands, England

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[30] **Foreign Application Priority Data**

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May 26, 1972 United Kingdom..... 25051/72

[52] **U.S. Cl.** **252/551**; 252/156; 252/532; 252/DIG. 13

[51] **Int. Cl.**..... **C11d 1/12**

[58] **Field of Search** ..... 252/532, 551, 156, DIG. 13

[56] **References Cited**

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Primary Examiner—Stephen J. Lechert, Jr.
Attorney, Agent, or Firm—Flynn & Frishauf

[57] **ABSTRACT**

Concentrates of sulphates of alkoxylated alcohols are diluted only with difficulty as a result of a viscosity peak being encountered during the dilution.

This problem may be mitigated if a water soluble salt of an acid of the general formula $A(COOH)_n$ is incorporated in the concentrate. Preferably said acid is one of the following: Glycollic acid, lactic acid, malic acid, glyceric acid, tartaric acid, citric acid, gluconic acid, saccharic acid, formic acid, acetic acid, butyric acid, oxalic acid, maleic acid, succinic acid or itaconic acid.

The formulations may be employed in shampoos.

20 Claims, No Drawings

AQUEOUS CONCENTRATE DETERGENT COMPONENT

The present invention relates to detergent components, in particular it relates to the sulphates of alkoxylated alcohols in particular to sulphates of alkoxylated C_8 to C_{24} alcohols such as sulphated ethoxylated lauryl alcohol or mixtures containing lauryl alcohol derivatives. For example those in which the alkyl derivative has a chain with a carbon number ranging from C_8 to C_{18}. Such a feedstock can readily be obtained from coconut or PK oil, or commercially available Ziegler or oxo alcohols.

Aqueous solutions of these compounds are employed in cosmetic, toiletry and detergent compositions for example shampoos and bubble baths and liquid cleaning compositions. Normally these materials are supplied at 29% active ingredient but in the interests of economy in transport and packaging high concentrations of the order to **50 – 70%** are also commercially available. At these high concentrations they have the texture of a thick paste. In the final formulation they are normally present in an amount less than 30% active in an aqueous solution.

Unfortunately on dilution of these concentrates with water rather than the viscosity diminishing as might be expected it starts to increase. For example the sodium salt of a sulphate of a di-ethoxylated derivative of the commonly used mixture of alcohols of $C_8$to C_{18} with a concentration of 60% active matter the balance being water, has a viscosity of 17,000 centipoises. On dilution to 45% active matter the viscosity increases to greater than 500,000 centipoises. On further dilution the viscosity drops until at the concentration used in shampoos it again becomes liquid.

Although it is possible to employ such concentrates without the incorporation of a viscosity modifier such an increase in the viscosity during dilution naturally contributes substantial problems for the formulator. Such problems include lumps of gel becoming attached to the containers or stirrers employed. Moreover the lumps of gel formed during the dilution are themselves only soluble with difficulty on further dilution.

In order to avoid these problems viscosity modifiers have been incorporated in aqueous concentrates of sulphates of ethoxylated alcohol so as to maintain the viscosity of a solution at high concentrations at a level such that the solutions are reasonably free flowing and are easily diluted to any required concentration. In addition to modifying the viscosity of a solution at concentrations in the range of 25 – 75% active matter it is also necessary that any additive employed should not prevent or hinder the effect of thickeners conventionally used such as sodium chloride or ammonium salts, which it may be desired to incorporate in the finished formulation in order to give it an acceptable consistency.

At present this problem is being reduced by incorporating in the concentrated aqueous sulphate of ethoxylated alcohol, an alcohol of low molecular weight such as isopropanol or ethanol. However, the use of such alcohols is disadvantageous in that having high vapour pressures their odour is detectable in the finished formulation and may constitute a fire hazard.

We have now found that we can prepare aqueous solutions of salts of sulphated alkoxylated alcohols which have viscosity and dilution properties that enable them

to be prepared as concentrates and diluted to normal formulation concentrations without detracting from the properties of such formulations. We can achieve this by incorporating in the aqueous concentrate of the surfactant a salt of a carboxylic acid of the following general formula:

$A - (COOH)_n$

where A is hydrogen or a saturated or unsaturated aliphatic residue having from 1 to 8 carbon atoms, optionally containing hydroxy substituents or may be nothing if n is 2, and n is an integer from 1 to 4.

Normally the residue A will have no more than 5 hydroxy substituents. Examples of such acids are glycollic acid (HO CH_2COOH), lactic acid ($CH_3CH(OH)$ (COOH) CH_2COOH), Malic acid (H OOC $CH_2CH(OH)COOH$), glyceric acid (HO $CH_2CH(OH)$-COOH), tartaric acid ($HOOC(CHOH)_2COOH$) citric acid (HOOC CH_2 C(OH)(COOH) CH_2COOH), gluconic acid (HO $CH_2(CHOH)_4$ COOH), saccharic acid ($HOOC(CHOH)_4COOH$), formic acid (HCOOH) acetic acid (CH_3COOH), butyric acid (C_3H_7COOH), oxalic acid (HOOCCOOH), maleic acid (HOOC CH=CH COOH), succinic acid (HOOC CH_2CH_2COOH) and itaconic acid (HOOC C(=CH_2) CH_2COOH)

Salts of some of these acids, such as those of citric acid have been employed previously in detergent formulations as buffers. However, the amounts of the acids or salts required to produce the viscosity modifying effect of the present invention are greater than those previously employed for such buffering action.

Accordingly the present invention provides an aqueous concentrate comprising at least 30% preferably 50% by weight of a sulphate of an alkoxylated alcohol and from 1 – 10% preferably 2 – 4% by weight of a salt of an aliphatic carboxylic acids hereinbefore defined.

Concentrates which are particularly suitable for modification according to the present invention are normally commercially available materials such as the sodium salt of the sulphate of an ethoxylated commercial lauryl alcohol having nominally 1 – 4 oxyethylene groups. Commercial lauryl alcohol is normally a mixture of compounds having an average chain length of about 12 carbon atoms. Other compounds such as the equivalent propoxylated derivatives or mixed ethoylated propoxylated derivatives or the derivatives of other $C_8 - C_{18}$ alcohols may also be employed. Such compounds are frequently in the form of their sodium salts although concentrates according to the invention may also be of use for salts such as potassium, magnesium, lithium, ammonium or alkylolamine salts. The normally available commercial materials sold as the salts of sulphated alkoxylated alcohols frequently contain electrolytes such as sodium chloride and sodium sulphate and free alkoxylated alcohol. These materials in themselves have an effect on the viscosity of the product, which may in some cases by synergistic with the added modifiers. However, it is not normally desirable to add excessive amounts of inorganic electrolyte (eg greater than 10% based on the sulphate material). Excessive amounts of alkoxylated alcohol may tend to separate out of solution and thereby detract from its properties.

Other components which are to be employed in the final formulation may of course also be present in the concentrate. For example there may also be present other surface active agents such as monoalkylolamides,

detergent sulphosuccinates, sulphosuccinamates, alkyl sulphate salts or nonionic surface active agents such as polyoxy alkylene ethers and esters.

The concentrate according to the invention may be diluted by the formulator at any convenient temperature, preferably in the range 15°– 70°C, to the final concentration required for the formulation. After such dilution other ingredients required for the final product may also be added as desired. From a second aspect therefore the present invention comprises making an aqueous solution of a sulphated ethoxylated alcohol as previously described of a concentration below 30% by weight by the addition of water to an aqueous concentrate having a content of at least **30%** 30% weight of the sulphated ethoxylated alcohol and from 1 – 10% by weight of a salt of an aliphatic hydroxylic carboxylic acid.

Compositions according to the invention are illustrated by the following examples.

EXAMPLE 1

Aqueous solutions of commercial sodium lauryl ether (2Eto) sulphate having concentrations of 50%, 40%) and 30% of the above compound were 2% containing 2% trisodium citrate based on total solution at each concentration. The viscosities of these solutions were compared with solutions equal concentrations of sodium lauryl ether sulphate containing no citrate. The viscosity data given below were obtained using a Brookfield viscometer (spindle LV 3 at 3 RPM) at ambient temperature.

% ether sulphate	% citrate	Viscosity (cp)
50	0	100,000
50	2	9,200
40	0	400,000
40	2	8,600
30	0	28,500
30	2	5,400

A consequence of the reduced viscosity of those samples containing citrate is that such samples dilute more readily than samples without added citrate.

EXAMPLE 2

Solutions were prepared as in example 1 but containing 2.5% disodium tartrate instead of 2% citrate. The viscosity data given below were obtained under the same conditions as in Example 1.

% ether sulphate	% tartrate	Viscosity (cp)
50	2.5	7,000
40	2.5	3,200
30	2.5	4,800

EXAMPLE 3

The following compounds disodium oxalate, disodium succinate, disodium maleate, disodium itaconate have been found to reduce the viscosities of 40% solutions of commercial sodium lauryl ether sulphate from greater than 400,000 cp to below 25,000 cp. when added at 5% concentration.

EXAMPLE 4

Commercial sodium lauryl ether sulphate was prepared at a concentration of 50% by neutralising lauryl ether sulphuric acid with aqueous sodium hydroxide in a continuous mixer. The product was a gel having a viscosity of 200,000 cp.

The same procedure was followed in a second run in which sufficient trisodium citrate was dissolved in the aqueous base to give a neutralised product containing 2.5% trisodium citrate.

The product was a free flowing mobile liquid crystalline system having a viscosity of 10,000 cp when measured by the procedure given in Example 1.

We claim:

1. An aqueous concentrate comprising 30–75% by weight of a sulphate of an alkoxylated aliphatic C_8-C_{24} alcohol and form 2 to 10% by weight of a water soluble salt of a carboxylic acid of the general formula

$$A(COOH)_n$$

wherein n is an integer of 1 to 4, and A is selected from the group consisting of hydrogen, a saturated aliphatic residue having from 1 to 8 carbon atoms, an unsaturated aliphatic residue of 2 to 8 carbon atoms, and when n is 2 the said formula also may be $(COOH)_2$.

2. A concentrate according to claim **1** wherein said salt is present in an amount of 2 to 4% by weight.

3. A concentrate according to claim **2** wherein the A group contains from 1 to 5 hydroxy groups.

4. A concentrate according to claim **3** wherein said salt is a salt of an acid selected from the group consisting of glycollic acid, lactic acid, malic acid, glyceric acid, tartaric acid, citric acid, gluconic acid or saccharic acid.

5. A concentrate according to claim **1** wherein said salt is a salt of an acid selected from the group consisting of formic acid, acetic acid, butyric acid, oxalic acid, maleic acid, succinic acid or itaconic acid.

6. A concentrate according to claim **3** wherein the sulphate of the alkoxylated alcohol is the sulphate of the reaction product of from 1 to 4 mols of ethylene oxide per mole of commercial lauryl alcohol.

7. A concentrate according to claim **5** wherein the sulphate of the alkoxylated alcohol is the sulphate of the reaction product of from 1 to 4 mols of ethylene oxide per mole of commercial lauryl alcohol.

8. A concentrate according to claim **2** which also contains sodium chloride or sodium sulphate in an amount of up to 10 % by weight of the alkyl ether sulphate.

9. A concentrate according to claim **3** which contains free alkoxylated alcohol.

10. A concentrate according to claim **5** which contains free alkoxylated alcohol.

11. A concentrate according to claim **1** which contains at least 50% by weight of sulphate of alkoxylated alcohol.

12. A concentrate according to claim **2** wherein said water-soluble salt of a carboxylic acid is a salt of tartaric acid; wherein said sulphate of the alkoxylated alcohol is the sulphate of the reaction product of from 1 to 4 moles of ethylene oxide per mole of commercial lauryl alcohol; and wherein said concentrate contains at least 50% by weight of said sulphate of alkoxylated alcohol.

13. A concentrate according to claim **2** wherein said water-soluble salt of a carboxylic acid is a salt of citric

acid; wherein said sulphate of the alkoxylated alcohol is the sulphate of the reaction product of from 1 to 4 moles of ethylene oxide per mole of commercial lauryl alcohol; and werein said concentrate contains at least 50% by weight of said sulphate of alkoxylated alcohol.

14. A concentrate according to claim **1** wherein said salt of said carboxylic acid is present in an amount of about 5% by weight.

15. A concentrate according to claim **14** wherein said water-soluble salt of a carboxylic acid is a salt of tartaric acid; wherein said sulphate of the alkoxylated alcohol is the sulphate of the reaction product of from 1 to 4 moles of ethylene oxide per mole of commercial lauryl alcohol; and wherein said concentrate contains at least 50% by weight of said sulphate of alkoxylated alcohol.

16. A concentrate according to claim **14** wherein said water-soluble salt of a carboxylic acid is a salt of citric acid; wherein said sulphate of the alkoxylated alcohol is the sulphate of the reaction product of from 1 to 4 moles of ethylene oxide per mole of commercial lauryl alcohol; and wherein said concentrate contains at least 50% by weight of said sulphate of alkoxylated alcohol.

17. A concentrate according to claim **1** which does not contain isopropyl alcohol or ethanol in amounts sufficient to render said concentrate free-flowing in the absence of said salt.

18. A free-flowing aqueous concentrate comprising an admixture of

i. between 2 and 10% by weight of a water soluble salt of a carboxylic acid of general formula $A(-COOH)_n$,

wherein n is an integer of 1 to 4, and A is selected from the group consisting of hydrogen, a saturated aliphatic residue of 1 to 8 carbon atoms, an unsaturated aliphatic residue of 2 to 8 carbon atoms, and when n is 2, the said formula also may be $(COOH)_2$; and

ii. an aqueous concentrate comprising between 30 and 75by weight of a sulphate of an alkoxylated aliphatic C_8-C_{24} alcohol which aqueous concentrate would be a thick paste or gel in the absence of admixture with component (i).

19. A concentrate according to claim **18** wherein said salt is present in an amount of from 2–4% by weight.

20. A concentrate according to claim **18** wherein said salt is present in an amount of about 5% by weight.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,893,955 Dated July 8, 1975

Inventor(s) THOMAS HEWITT et al

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 47, "C_8 - $_{c18}$" should be --C_8 - C_{18}--.

Column 3, line 14, "30%" should appear only once.

Column 3, line 24, replace "2%" with --prepared--.

Column 3, line 27, after "solutions" insert --of--.

Column 6, line 15, after "75" insert --%--.

Signed and Sealed this

sixteenth Day of *March 1976*

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks

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