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WETTING AND DETERGENT COMPOSITION

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2 Claims. (Cl. 252—161)

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This invention relates to wetting and detergent compositions containing monoalkyl sulfosuccinates together with a solubilizing agent therefor. The invention includes the compositions themselves, in their methods of preparation, and wetting and detergent processes in which these compositions are used.

It is known that the higher monoalkyl sulfosuccinates, when used in the form of their salts with monovalent salt-forming compounds, possess good wetting and particularly good detergent properties. However, the salts of monoesters of sulfosuccinic acid with alcohols of 12 or more carbon atoms, which is the minimum chain length for good detergency, possess relatively low solubility in water, which is a serious disadvantage. It is a principal object of the present invention to provide for this class of compounds a solubilizing agent which improves their water solubility and thus increases their usefulness as wetting agents and detergents. A further object resides in the provision of readily soluble pastes or powders containing the higher monoalkyl sulfosuccinates in a solubilized and therefore water-dispersible condition. Still further objects of the invention will be apparent from the following detailed description of preferred embodiments thereof.

As is noted above the sodium, potassium, ammonium, ethanolamine and similar salts of the higher monoalkyl sulfosuccinates in which the alkyl radical contains 12 or more carbon atoms are only slightly soluble in water. The solubility of the disodium salts, which is given in the following table, is typical of the corresponding salts of potassium and ammonium as well.

Monoalkyl Disodium Sulfosuccinate	Solubility, gms./100 cc. of Solution at 30° C.
Dodecyl	4.5
Tetradecyl	0.05
Hexadecyl	0.2
Octadecyl	0.05

Additional monoalkyl sulfosuccinates which have desirable qualities as wetting agents and detergents comprise the sodium, potassium, ammonium, etc. of salts of mixed esters of sulfosuccinic acid obtained from a mixture of higher aliphatic alcohols that is available commercially and which contains about 40–45% myristyl alcohol (tetradecanol-1), about 30–35% cetyl alcohol (hexadecanol-1), about 3–5% capryl alcohol (octanol-2), about 10–15% of non-alcoholic sub-

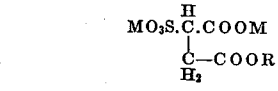
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stances of hydrocarbon nature boiling at 39° C. to 96° C. at 0.3 mm. of mercury pressure and therefore containing about 14–16 carbon atoms, and about 5–8% of higher boiling materials, possibly higher alcohols. This mixture is hereinafter designated as "Ammecol," which is its commercial name.

Attempts have been made to solubilize these and similar compounds by adding water-soluble alcohols, ethers of ethylene glycol and other agents which are known to solubilize the higher dialkyl sulfosuccinates. However, it was found that these alcohols and ethers actually reduce the water solubility of the alkali metal and ammonium salts of higher monoalkyl sulfosuccinates.

I have now discovered that water-soluble lithium salts such as lithium sulfate, lithium chloride, lithium bromide, lithium nitrate, lithium acetate and the like are excellent solubilizing agents for the higher monoalkyl sulfosuccinate salts. This is a remarkable discovery, for the addition of the corresponding salts of other alkali metals such as sodium or potassium sulfate or chloride causes precipitation of the higher monoalkyl sulfosuccinate salts instead of solubilizing them. I have found, however, that I can prepare stable aqueous solutions containing from about 1% to about 30% of the wetting agents, depending on the particular alcohol present in the sulfosuccinate ester, as well as water-soluble concentrated aqueous pastes containing about 30–70% of the wetting agents, and even water-soluble dry powders containing the higher monoalkyl sulfosuccinate salts together with inorganic salts of the type of sodium sulfate, sodium chloride and the like by incorporating therein suitable quantities of lithium salts which ionize in aqueous solution.

The monoalkyl sulfosuccinate salts which are solubilized by lithium salts in accordance with the present invention have the following formula



in which M is a monovalent salt-forming group such as sodium, potassium, ammonium, ethanolamine and the like, and R is an alkyl radical of at least 12 carbon atoms, and preferably 12–18 carbon atoms, which may be saturated or unsaturated, or a mixture of such alkyl radicals. The compounds defined by the above formula are preferably prepared by esterifying maleic anhydride or maleic acid with the dodecyl, tetradecyl, octadecyl, octadecenyl or other alcohols contain-

ing 12 or more carbon atoms at about 100–110° C. to form the monoester, followed by sulfonating this ester to form the sulfosuccinate. Preferably, the sulfonation is carried out by heating the ester with an aqueous solution of sodium sulfite or sodium metabisulfite, or of the corresponding potassium or ammonium compounds. By this method the alkali metal and ammonium salts of the monoalkyl sulfosuccinic acid esters are obtained directly.

The corresponding salts of ethanolamine and other bases are preferably produced by first acidifying an alcoholic solution of the alkali metal or ammonium sulfosuccinates, whereby the free sulfosuccinic acid monoester is obtained, filtering off the inorganic salt of the acid used and reacting the sulfosuccinic acid ester with the desired base. Salts of other metals such as calcium, barium, zinc, chromium, tin, lead and the like are prepared by adding stoichiometric quantities of their oxides or hydroxides to the alcoholic sulfosuccinic acid monoester followed by stirring until the salt formation is complete. Salts of organic bases such as methylamine, ethylamine, ethanolamine and the like may also be prepared by the same method.

For many purposes it is desirable to prepare the higher monoalkyl sulfosuccinates in the form of dry, water-dispersible powders. Heretofore, this has not been possible, for upon evaporation of their solutions in water or organic solvents the compounds form a white pasty mass similar in texture to ordinary soap. The compounds are precipitated from their aqueous solutions by the addition of ammonium, sodium, or potassium salts, and therefore the addition of sodium or potassium sulfate or chloride to form a dry, free-flowing powder could not be resorted to.

Another important feature of the present invention is the discovery that the solubilizing action of water-soluble lithium salts persists even in the presence of sodium sulfate, sodium chloride, sodium carbonate and other salts which would ordinarily precipitate the higher monoalkyl sulfosuccinate salts from their water solutions. This discovery has enabled me to prepare dry, free-flowing powders containing the higher monoalkyl sulfosuccinate salts in admixture with water-soluble alkali metal compounds other than lithium compounds, or in admixture with water-soluble ammonium compounds, which powders are readily soluble in cold water. No material change in the ratio of lithium salt to monoalkyl sulfosuccinate salt need be made when other water-soluble salts are added, i. e., the solubilizing action of the lithium salts is the same in the presence of sodium sulfate, sodium chloride, sodium carbonate and the like as it is in the absence of these salts. However, it has been found that while the upper limit of diluent which may be employed depends entirely on the desired concentration of wetting agent in the dry mixture, the lower limit of diluent is not substantially less than 40% of the mixture. For example, when a drix mix is prepared containing more than about 60% of the monoalkyl sulfosuccinate, the material is not easily soluble in cold water; for this reason dry mixtures containing in excess of about 60% of monoalkyl sulfosuccinate are not included within the purview of this invention.

In practicing the invention, the amounts of lithium salts to be employed vary according to the original water solubility of the various monoalkyl sulfosuccinate salts. It will be readily apparent that dodecyl disodium sulfosuccinate, having a water solubility of 4.5 grams per 100 cc., will require the addition of less lithium salt than will disodium hexadecyl sulfosuccinate, for example, having a water solubility of 0.2 gram per 100 cc., in the preparation of aqueous solutions of 10% wetting agent content. The following table sets forth the increase in aqueous solubility of various sulfosuccinate salts induced by optimum molar addition of lithium salts. Mol, as expressed in the description of the present invention, may be defined as the gram ionic equivalent of the lithium salts employed.

	Per Cent Solubility, gms./100 cc. at 30° C. No Lithium Salt Addn.	Per Cent Solubility, gms./100 cc. at 30° C., $Li_2SO_4.H_2O$	Molar Ratio of Lithium to Succinate Salt
Dodecyl	4.5	25	1:1
Dodecyl	4.5	7	0.25:1
Tetradecyl	0.05	5	3:1
Hexadecyl	0.2	10	2:1
Mixture [1]	0.2	10	2:1
Octadecyl	0.65	1-2	3:1

[1] The sulfosuccinate mixture above referred to is that prepared from Ammecol.

As is common in the case of the solubility of salts, higher solution temperatures result in an increase of the solubility of the various salts. In the case of the present invention, such variations in solution temperatures permit the use of smaller amounts of the solubilizing lithium salts. For this reason, the lower limits of lithium salt addition may in some cases and for some purposes be within the range of from 0.25 mol to 0.5 mol per mol of sulfosuccinate salt employed. On the other hand, while there is no absolute upper limit of lithium salt addition since the solubilizing agent does not interfere with the wetting and detergent properties of the monoalkyl sulfosuccinates, for most commercial applications not more than about 7 to 10 mols of lithium salt should be employed for each mol of sulfosuccinate. Moreover, I have found that the most preferable quantity of lithium salt to be employed is from 0.25 mol to 3.0 mols for each mol of the sulfosuccinate depending on its water solubility.

Conversely, in preparing aqueous pastes and dry powders containing water-soluble inorganic salts of monovalent metals such as sodium and potassium, larger amounts of lithium salts may of course be used, although amounts substantially larger than those given in the above table are not usually necessary.

It is evident, therefore, that the invention in its broader aspects is not limited to the use of large quantities of the lithium salts but that relatively small proportions of these salts may be used if desired.

The invention will be illustrated in greater detail by the following specific examples. It should be understood, however, that although these examples may describe in detail certain specific features of the invention, they are given primarily for purposes of illustration and the invention in its broader aspects is not limited thereto. All parts are by weight.

Example 1

A paste was prepared by mixing together 92 parts of a 30% aqueous gel of a disodium monoalkyl sulfosuccinate prepared from Ammecol and 8 parts of $Li_2SO_4.H_2O$. The paste was used to prepare an aqueous solution containing 10% of the salt of the sulfosuccinate mixture. The mate-

rial was completely soluble and remained clear at temperatures as low as 6°–10° C., whereas the same sulfosuccinate salt is normally only 0.2% soluble at temperatures of or less than 35° C.

Example 2

A dry, free-flowing powder was obtained by drying to 100 parts an aqueous mixture consisting of 116.6 parts of a 30% gel of the monoalkyl sulfosuccinate mixture employed in Example 1, 9.5 parts of $Li_2SO_4.H_2O$, and 55 parts of Na_2SO_4. The dried powder formed clear solutions with water when used in concentrations up to 10%.

Example 3

To 100 cc. of a 2% aqueous dispersion of dipotassium cetyl sulfosuccinate was added 3.6 cc. of a 10% aqueous LiCl solution (1:2 molar ratio). The cloudy dispersion immediately became clear, indicating that a true solution of the sulfosuccinate had been obtained.

Other lithium halides such as lithium bromide and lithium iodide have also been found to give comparable results.

Example 4

To 100 cc. of a 2% aqueous dispersion of diammonium cetyl sulfosuccinate was added 5.9 cc. of a 10% aqueous $LiNO_3$ solution (1:2 molar ratio). The cloudy dispersion immediately became clear, indicating that a true solution of the sulfosuccinate had been obtained.

Example 5

To 100 cc. of a 2% aqueous dispersion of disodium cetyl sulfosuccinate was added 8.8 cc. of a 10% $Li(OOCCH_3)_2.2H_2O$ solution (1:2 molar ratio). The initially cloudy dispersion immediately became clear, indicating that a true solution of the sulfosuccinate had been obtained.

Additional organic lithium salts which increase the solubility of higher monoalkyl sulfosuccinates in a manner comparable to that set forth above, are lithium salicylate, lithium citrate, lithium formate, and lithium benzoate.

Example 6

To 100 cc. of a 5% aqueous dispersion of disodium oleyl sulfosuccinate was added 8.8 cc. of a 10% LiCl solution (1:2 molar ratio). The initially cloudy dispersion immediately became clear, indicating that a true solution of the sulfosuccinate had been obtained.

Example 7

A dry, free-flowing powder was obtained by drying to 100 parts an aqueous mixture consisting of 116.6 parts of a 30% gel of a disodium monoalkyl sulfosuccinate prepared from Ammecol 9.5 parts of $Li_2SO_4.H_2O$, and 55.5 parts of NaCl. The dried powder formed clear solutions with water when used in concentrations up to 10%.

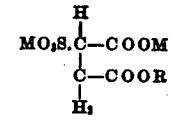
Example 8

A dry, free-flowing powder was prepared by drying to 100 parts an aqueous mixture consisting of 233.2 parts of a 30% gel of the monoalkyl sulfosuccinate mixture employed in Example 7, 9.5 parts of $Li_2SO_4.H_2O$, and 20.5 parts of Na_2CO_3. The dried powder formed clear solutions with water when used in concentrations up to 10%.

This is a continuation-in-part of my co-pending application Serial No. 626,576, filed November 3, 1945, and which is now abandoned.

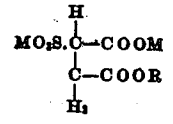
I claim:

1. A wetting and detergent composition comprising essentially a surface active material which is a monoalkyl sulfosuccinate having the formula



in which M is a member of the group consisting of alkali metal and ammonium radicals and both M's are the same, and R is an alkyl radical of from 12 to 18 carbon atoms, and a water-soluble lithium salt capable of increasing the water-solubility of the monalkyl sulfosuccinate, said water-soluble lithium salt being present in an amount within a minimum range of from 0.25 mol to 3.0 mols and a maximum amount of 10 mols for each mol of the sulfosuccinate.

2. A wetting and detergent composition comprising essentially a surface active material which is a mixture of monalkyl sulfosuccinates having the formula



in which M is a member of the group consisting of alkali metal and ammonium radicals and both M's are the same, and R is an alkyl radical of from 12 to 18 carbon atoms, and a water-soluble lithium salt capable of increasing the water solubility of the monalkyl sulfosuccinates, said water-soluble lithium salt being present in an amount within a minimum range of from 0.25 mol to 3.0 mols and a maximum amount of 10 mols for each mol of the sulfosuccinates.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
Re. 19,719	Hall	Apr. 24, 1924
2,316,234	Flett	Apr. 13, 1943
2,181,087	Caryl et al.	Nov. 21, 1939

OTHER REFERENCES

Aerosol Wetting Agents, Pub. of Amer. Cyanamide and Chem. Corp., N. Y., (1941), pp. 8–13.