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CAKE DETERGENT AND METHOD FOR ITS PRODUCTION

Emil A. Vitalis, Springdale, Conn., assignor to American Cyanamid Company, New York, N. Y., a corporation of Maine

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This invention relates to the production of a pressure-hardened synthetic detergent in cake form suitable for use as a toilet soap and other household purposes and to the product so obtained. Its principal object is the provision of a particular type of dialkylsulfosuccinate detergent in the form of a pressure-hardened cake that will last many times longer than a cake of ordinary toilet soap of the same size and that will possess a number of other advantages over soap as will hereinafter be described in greater detail.

The excellent detergency of the higher dialkylsulfosuccinates (the preparation of which is described in U. S. Patent No. 2,028,091) has long been recognized, and a number of household detergent compositions containing these materials have been proposed. These compounds, however, are soft and gelatinous at atmospheric temperatures and, in their normal condition, they form soft gels upon soaking in water. For this reason they have only been proposed for use in admixture with diluents such as sodium sulfate in powdered form or, in one instance, with equal or greater quantities of powdered boric acid in the form of a detergent cake. In no case has it been considered possible to produce a suitable cake detergent composed substantially completely of the detergent material itself.

I have now discovered that certain of these higher dialkylsulfosuccinate detergents, which are normally soft and plastic in character, can be converted into hardened masses suitable for use in cake form by compacting them under pressure. I have also found that the resulting hardened masses possess all of the most desirable attributes of a cake detergent; they preserve their cake form upon extended soaking in water, have the correct solubility to release a small but effective amount of detergent into the water, and are not sticky or otherwise unpleasant to handle. In addition to these desirable physical properties, I have also found that the higher dialkylsulfosuccinates in question are non-dermatitic; i. e., they have absolutely no poisoning or irritating action on the skin. They are neutral in reaction (the pH of a 1% solution is between 6.0 and 7.0) and are colorless, tasteless, practically odorless, and non-poisonous. They produce copious foam in water even in dilutions as high as 1 to 30,000 and they foam equally well in soft or hard water. They possess an unusually good calcium tolerance and therefore will form no scum even in very hard water, and they leave no residue on dishes, glassware or other objects washed therewith. Sample cakes have been tested extensively by a number of families for dish washing, for toilet, bath and shower, as hair shampoos and for light laundering, and all of the reports of those using them have been favorable. These tests have also shown that a compacted sulfosuccinate detergent cake will last from ten to about fifteen times as long under average conditions of home use as will a cake of "Ivory" soap of the same size.

The dialkylsulfosuccinates which I have found to possess the above-described properties are the diesters of sulfosuccinic acid with straight-chain primary aliphatic alcohols of from 8 to 10 carbon atoms in the form of their

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sodium, potassium or other alkali metals salts. They are produced by esterifying 1 mol of maleic or fumaric acid with 2 mols of n-octanol-1, n-nonanol-1, or n-decanol-1 and reacting the resulting diester with an aqueous solution of sodium, potassium or other alkali metal bisulfite or metabisulfite using the procedures described in U. S. Patent No. 2,028,091. The resulting dialkylsulfosuccinate solutions are then evaporated to dryness. The dried product is a substantially pure diester of sulfosuccinic acid and is a soft, odorless wax that disperses to a cloudy gel upon soaking in water.

The three dialkylsulfosuccinates used in practicing the invention are quite similar in physical properties and in foaming power and detergency. They differ somewhat in water solubility, the di-n-decyl compound being somewhat less soluble in water than the other two, but exhibiting a slightly higher degree of detergency due to its greater alkyl chain length. Any one of the three compounds can therefore be used in practicing the invention or any two or more may be used in admixture; the di-n-decyl sodium or potassium sulfosuccinate is especially well adapted for use in admixture with either or both of the other two esters as its higher detergency will improve the detergent properties of the mixture while the greater water solubility of the lower esters will make the detergent cake easier to use. Thus, for example, a compacted mixture containing 20–40% of di-n-decyl sodium sulfosuccinate and 80–60% of di-n-octyl sodium sulfosuccinate or di-n-nonyl sodium sulfosuccinate combines the best properties of both types of compounds.

The dialkylsulfosuccinates and sulfosuccinate mixtures described above are converted into the hardened detergent mass of the present invention by compacting under high pressure. A number of compacting procedures may be used effectively; thus, the gel may be filled into an extrusion press and extruded into a rod of pressure-compacted material which may be cut into pieces of soap-bar size. These pieces may be subjected to die-press molding to form a molded cake if desired. For large-scale production it is preferable to convert the original diester into pellets by extrusion under low pressure as such pellets are well suited for charging into high pressure extrusion molding equipment. If desired, the original diester may be subjected to mechanical working or crutching to produce a homogenized material, as this improves the appearance of the pressure-hardened mass. A small quantity on the order of 1–5% by weight of lanolin, glycerine, beeswax or other suitable lubricant may be added to improve the performance of the material in the mold, although experience has shown that the mold can be lubricated sufficiently simply by adding a few drops of water. The molding can be carried out at ordinary atmospheric temperatures or elevated temperatures up to the softening point of the hardened diester, which is within the range of 150°–250° C., may be used.

I have found that detergent masses of the above-described diester possess all of the desirable properties enumerated above when they are compacted under pressures of at least 100 lbs. per square inch. When substantially higher pressures up to 300–500 lbs. per square inch are used the cakes are correspondingly harder and have a somewhat longer effective period of use under ordinary household conditions. The novel detergent compositions of my invention therefore consist of the hardened masses obtained by compacting the above-described dialkylsulfosuccinates under pressures of at least 100 lbs. and preferably at least 300 to 500 lbs. per square inch whereby they are transformed from their normally plastic condition into hard, soap-like materials. It is a particularly important advantage of my invention that the products so obtained consist substantially solely of a highly efficient detergent and do not require the presence of an inert

filler or carrier such as was previously considered necessary.

The invention will be further described by the following specific examples to which, however, it is not limited.

Example 1

Di-n-octyl sodium sulfosuccinate was prepared by esterifying maleic anhydride with n-octanol-1, distilling the resulting ester under about 0.5-1 millimeter of mercury pressure and reacting the purified ester with aqueous sodium metabisulfite. The resulting aqueous paste was dried in a tray drier and extruded into pellets about one-half inch in size. The product had the following properties:

Melting point: 150°-180° C. Decomposes above 200° C. Water solubility: 1% at 30° C.

Crystal form: soft wax, easily deformed between the fingers

Acid number: less than 2.5

Hydrolysis: hydrolyzes slowly at pH values above 10.5

pH of 1% solution: 6.5

Solids content: 99.8% minimum

Toxicology: non-poisonous; non-irritating to the skin and to the eye; completely non-dermatitic

Portions of this material were compacted in a molding press as cylinders two inches in diameter and one inch high. The pressure applied to the press was 2500 pounds or slightly more than 400 pounds per square inch of the top surface of the material. The resulting cakes were hard and tough but translucent. A wet cloth rubbed over the surface of one of these cakes readily took up a sufficient quantity for washing a window and yet the cake itself was remarkably resistant to disintegration in water; in fact, it retained its cake form after soaking in water at room temperature for 48 hours. Material dissolved from the surface of the cake was found to possess a detergency and foaming power equal to that of the original product before compacting.

Another portion of the di-n-octyl sodium sulfosuccinate pellets was passed through a soap crutcher and an extrusion press and extruded into an elongated bar from which pieces were cut. These pieces were molded under a pressure of 100 lbs. per square inch pressure into 2¼ by 1¾ inch cakes one-half inch thick using a few drops of water in the mold as a lubricant. The cakes had the same excellent resistance to disintegration in water as those molded under higher pressure but were white in color instead of translucent and the material of the cake was more readily transferred to a moistened cloth.

Example 2

Di-n-nonyl sodium sulfosuccinate is similar in appearance and physical characteristics to di-n-octyl sodium sulfosuccinate but it has a slightly lower solubility in water.

The water solubility of the two compounds at various temperatures is as follows:

Temp., °C...	80.5	75	70	68	58	56.2	40	35	29	25
Di-n-octyl...	9.1	7.4	6.3	4.8	4.0	2.6	2.0	1.5	1.0	0.1
Temp., °C.....					86	83	68	45	40	30
Di-n-nonyl.....					3.9	3.2	1.96	1.0	0.76	0.45

The wetting power of the di-nonyl ester at 30° C. is about one-third of that of di-n-octyl sodium sulfosuccinate but its foaming power is slightly greater and its detergency is about equal to that of the di-n-octyl compound. It is therefore well suited for use as a cake-type detergent in accordance with the present invention.

A sample of pelleted di-n-nonyl sodium sulfosuccinate was compacted in the molding press described in Example 1 under a pressure of 30,000 pounds or about 500 pounds per square inch. The molded cake was hard and had even better resistance to disintegration in water than the corresponding di-n-octyl ester, but the rate at which it was taken up by a moistened cloth was somewhat slower. However the material dissolved from the cake had the same good foaming power and detergency as the original uncompact ester.

What I claim is:

1. A non-dermatitic detergent in cake form consisting of a hardened mass obtained by compacting under a pressure of at least 100 pounds per square inch a normally plastic alkali metal sulfosuccinate selected from the group consisting of di-n-octyl, di-n-nonyl and di-n-decyl sulfosuccinates and mixtures thereof.

2. A method of producing a non-dermatitic detergent in hardened cake form which comprises compacting a normally plastic alkali metal sulfosuccinate selected from the group consisting of di-n-octyl, di-n-nonyl and di-n-decyl sulfosuccinates and mixtures thereof under a pressure of at least 100 pounds per square inch.

References Cited in the file of this patent

UNITED STATES PATENTS

2,028,091	Jaeger	Jan. 14, 1936
2,176,423	Jaeger	Oct. 17, 1939
2,508,578	Marshall	May 23, 1950
2,594,956	Marshall	Apr. 29, 1952

OTHER REFERENCES

McCutcheon: Soaps and Sanitary Chemicals, December 1949, pages 33-35 and 145.

Lesser: Soap and Sanitary Chemicals, June 1950, pages 42-45, 153 and 155.

Keenan: Soap and Sanitary Chemicals, May 1951, pages 27-30 and 135.