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2,653,913

SYNTHETIC DETERGENT CAKES

Willem J. D. van Dijck, The Hague, Netherlands,
and Bradford P. Geyer, Berkeley, Calif., assign-
ors to Shell Development Company, San Fran-
cisco, Calif., a corporation of Delaware

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This invention relates to synthetic detergent compositions capable of being shaped into coherent bars, cakes or other useful forms. It deals particularly with synthetic detergents, especially those of the sulfonate type which includes not only the salts of organic sulfonic acids but also salts of sulfuric acid esters, in the form of bars or cakes having the shape stability, texture and solubility characteristics required for use as a high grade toilet soap.

Synthetic detergents have achieved widespread use as washing powders suitable for laundry, dishwashing and like purposes. Spray or drum dried powders prepared from water soluble salts of alkyl aryl sulfonic acids, alkyl sulfonic acids and alkyl acid sulfates, in particular, are used in this way in very large amounts. However, in spite of numerous attempts, satisfactory bar soaps suitable for toilet and bath use have not hitherto been produced from synthetic detergents. As a result, toilet soaps have had to be produced from fatty acid soaps which are suitable for milling and molding, and even these products have not been entirely satisfactory unless used in soft water.

An important object of the present invention is to provide bar soap detergents which are especially suitable for use in hard water and, while readily soluble and giving rapidly a copious lather, are not so soluble as to become soft and slimy or be uneconomical in use. Another object is to produce from non-soap detergents bars of high active ingredient which do not cause drying but impart a pleasant feel to the skin. Still other objects and advantages of the invention will be apparent from the following more detailed description of the new bar detergents.

According to the invention, bar soap compositions of improved characteristics are produced by blending together in controlled proportions, three essential components, namely:

Percent by weight

A synthetic detergent of the non-soap type	68 to 84
A water dispersible binder coating on the surface of the detergent particles	15 to 30
A protective colloid emollient thickening agent	1 to 2

These percentages are on an anhydrous basis but small amounts of water may be present in the various components.

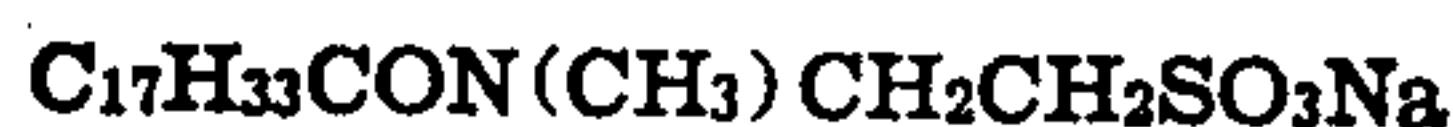
As previously indicated, the preferred synthetic detergents for use in the bar detergents of the invention are the sulfonate type detergents, that is, the water soluble salts of sulfuric

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acid esters and of organic sulfonic acids. Alkali metal salts, particularly the sodium salts of alkyl acid sulfates having about 12 to 20 carbon atoms per molecule, have been found to be particularly suitable. Either salts of primary alkyl acid sulfates, such as are described in United States Patents 1,968,793 and 1,968,794, for instance, or salts of secondary alkyl acid sulfates which may be prepared from the corresponding olefins or secondary alcohols, as described, for example, in United States Patents 2,139,393 and 2,152,163, may be used. These alkyl sulfate salts are an especially advantageous type of synthetic detergent for use in the new detergent bars because they are stable, odorless, highly resistant to acid or alkali, unaffected by hard water, and give a copious lather.

Among the organic sulfonic acid salts which may be used in the new bars of the invention, the preferred members are the alkyl aryl sulfonates having 10 to 18 carbon atoms in the alkyl group. Typical examples of suitable alkyl aryl sulfonates are, for instance, sodium dodecyl benzene sulfonate and sodium dodecyl toluene sulfonate, which may be produced, for example, by alkylating benzene or toluene with an olefin of twelve carbon atoms such as propylene tetramer or a butylene trimer, a less branched dodecene, preferably dodecene-1 or dodecene-2, sulfonating the alkylate and neutralizing the alkyl aryl sulfonic acid obtained with sodium hydroxide. Suitable methods for carrying out this process are described in United States Patents 2,232,117 and 2,456,119 which also show other suitable alkyl aryl sulfonates which may be used in the new bars. Keryl benzene sulfonates and the like, such as are disclosed in United States Patents 2,161,173 and 2,223,364, may also be used in the detergent bars and cakes of the invention. Instead of, or in combination with, the foregoing alkyl aryl sulfonates, one may employ alkyl sulfonic acid salts which may advantageously be prepared as described in United States Patents 2,263,312 and 2,276,090, for example.

Other sulfated and sulfonated detergents which may be used include, for example, the sulfated and sulfonated amides and esters. Typical of the amide derivatives, for instance, are the sodium salt of the condensation product of oleyl chloride with methyltaurine having the formula,



and the corresponding sulfate ester salt,



The sulfated and sulfonated esters, although less preferred because of their lesser stability, are nevertheless excellent detergents for use in the invention and may be prepared by esterifying higher fatty acids with hydroxysulfonic acids and neutralizing the ester to obtain compounds of the type, $C_{17}H_{35}COOCH_2CH_2SO_3Na$, or by sulfonating monoesters of polyhydric alcohols and neutralizing whereby compounds such as $C_{17}H_{35}COOCH_2-CH_2-O-SO_3Na$ and the like are obtained. These, and other commercially available anion active synthetic detergents which are suitable for use in the new compositions of the invention, are described in an article by John W. McCutcheon in "Chemical Industries," November 1947, pages 811 to 824.

The synthetic detergent may be used in a pure or substantially pure form as the individual compound or as a mixture of two or more such compounds of the same or of different types. The commercially available synthetic detergents are usually mixtures of isomers and/or homologous compounds which also contain various amounts of inorganic salts such as sodium sulfate and the like formed in the course of manufacture of the detergent. Small amounts of water soluble, unreactive inorganic salts are not detrimental and, in fact, may be advantageous in the bar detergents of the invention; however, it is preferred that not over 10% such inorganic salts, based upon the weight of synthetic detergent used, be present since larger amounts undesirably reduce the detergency of the composition. Most preferably, the amount of water soluble inorganic salts present should not be more than about 3% to 8% of the weight of the final composition, and where such salts are present, the amount of synthetic detergent therein is correspondingly reduced so as to be in the range of 66% to 81%. It is highly important that salts which are water insoluble or which react with water to produce insoluble compounds be absent from the detergent bars. Salts or other components which are hygroscopic or strongly alkaline should also be avoided.

The dispersible binders which it has been found most advantageous to use are waxes and the like having a melting point of at least 130° F. Most suitable because of their stability, uniformity and the especially desirable characteristics which they impart to the new detergent bars are the higher melting paraffin waxes. Preferred paraffin waxes are those melting from about 140° F. to 180° F. Other types of waxes which have been used are, for example, carnauba wax, montan wax, candelilla wax, Japan wax, Chinese insect wax, spermaceti, and the like. Some of these waxes are more or less colored in their crude form and contain low melting constituents. Any components which lower the melting point of the wax below 130° F. should be removed in all cases before the wax is used in the bar detergents of the invention, and, for the production of the most desirable detergent bars, excessive amounts of colored constituents should also be removed. Instead of natural waxes, artificial waxes such as the higher melting chlorinated naphthalenes and chlorinated paraffins may be used. Partially oxidized waxes, particularly paraffin wax which has been partially oxidized and neutralized, preferably with a basic alkali metal compound, offer special advantages of improved dispersibility and increased detergency of the finished cake or bar.

As protective colloid emollient in the new detergent bars, gel-forming gums are used. Of

these, the water soluble alginates, particularly sodium alginate, have proven outstanding. They impart a very desirable unctuousness and an agreeable feel to the final detergent. However, other slippery gel-forming gums of similar properties may also be used, particularly other water soluble salts of high molecular carbohydrate acids which form insoluble calcium salts. Examples of suitable compounds are, for example, alkali metal or ammonium pectates, tannates, humates, and the like. Although generally more expensive and less desirable, water soluble gums such, for instance, as agar agar, etc. can also be used. Only compounds which form detergent compositions which give clear or substantially clear aqueous solutions should be used, and gums which form sticky solutions are to be avoided.

The components of the new bar detergents must be carefully mixed so that the water dispersible binder forms an outer phase substantially surrounding discrete particles of the synthetic detergent and is not incorporated within the detergent particles themselves. Thus, the water dispersible binder preferably forms a substantially continuous phase holding together particles of synthetic detergent without interfering with the desirable detergent action of the synthetic detergent. The dispersion may be, and preferably is, a double dispersion of particles of detergent and protective colloid emollient in the continuous phase of wax binder. However, in the new detergent bars or cakes the dispersed particles may consist of mixtures of detergent and protective colloid emollient. Mixing may advantageously be carried out in a crutcher, preferably heated to above the melting point of the binder used, for example, to about 150° F. to 200° F. The binder is first added and, as it softens, the detergent, in powder form, is gradually run in with constant agitation until the required amount has been uniformly dispersed in the binder. The protective colloid emollient may be dispersed in the binder before or after the detergent is added, or the two may be separately dispersed in the binder at the same time. Alternatively, the emollient may be mixed with or dispersed in the synthetic detergent before the latter is added to the melted binder wax. During or after incorporation of the detergent and/or the emollient, any builder, perfume or the like which is to be employed may be added. The detergent mixture composition can be dropped out onto frames and stripped, slabbed and cut in the same way as ordinary cake soap. The new detergent bars harden rapidly and may be promptly pressed and wrapped without marring.

Alternatively, a conventional soap amalgamator may be used for the initial mixing which may be finished on a heated soap mill and milled detergent converted into continuous bars in a plodder. These bars can be cut and stamped or pressed in the usual way.

Some typical examples of detergent cakes prepared as in the foregoing are the following which give in all cases products of excellent properties with respect to detergency, foaming, shape stability and stickiness:

Example I

	Per cent
Sodium secondary alkyl sulfates from cracked wax olefins of 12 to 18 carbon atoms (containing 9.6% sodium sulfate)	80.5
Paraffin wax, M. P. 140° F.	18
Sodium alginate	1.5

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Example II

	Per cent
Sodium dodecyl benzene sulfonate-----	82
Paraffin wax, M. P. 140° F-----	16
Sodium alginate -----	2

Example III

	Per cent
Sodium primary alkyl sulfates from hydro- genation of coconut oil-----	76.4
Paraffin wax, M. P. 154° F-----	22.0
Sodium alginate -----	1.6

It will be seen that the new compositions offer many advantages and are capable of considerable variation, not only in regard to the proportions in which the components may be blended but also with respect to the starting materials which may be used. Thus, while sulfonate type detergents, particularly the alkyl sulfate salts, have been emphasized, it will be apparent that the invention is not limited thereto since the corresponding phosphoric acid ester salts may be used in the same way. Still other variations in the compositions may be made without departing from the invention which is not limited to the examples given by way of illustration nor by any theory proposed in explanation of the improved bar detergents which are obtained.

We claim as our invention:

1. A detergent in bar or cake form consisting essentially of a member of the group consisting of water-soluble anionic synthetic organic sulfonated and sulfated detergents, a paraffin wax binder therefor which has a melting point of at least 130° F., and as an emollient a member of the group consisting of alkali metal and ammonium pectates, tannates, humates and alginates, in the proportions of 68% to 84% by weight of said detergent, 15% to 30% of said binder, and 1% to 2% of said emollient.

2. A detergent in bar or cake form consisting essentially of a water-soluble salt of an alkyl acid sulfate detergent having 12 to 20 carbon atoms per molecule, a paraffin wax binder therefor which has a melting point of at least 130° F., and as an emollient a member of the group consisting of alkali metal and ammonium pectates, tannates, humates and alginates, in the proportions of 68% to 84% by weight of said detergent, 15% to 30% of said binder, and 1% to 2% of said emollient.

3. A detergent in bar or cake form consisting essentially of 68% to 84% by weight of a water-soluble salt of a sulfonic acid having a hydrocarbon radical of 12 to 24 carbon atoms in the molecule, 15% to 30% of paraffin wax having a melting point of at least 130° F., and 1% to 2% of a member of the group consisting of alkali metal and ammonium pectates, tannates, humates and alginates.

4. A detergent in bar or cake form consisting essentially of a sodium salt of an alkyl acid sul-

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fate having 12 to 20 carbon atoms per molecule, a paraffin wax melting at about 140° F. to 180° F., and sodium alginate in the proportions of 68% to 84% by weight of said sodium alkyl sulfate, 15% to 30% by weight of said paraffin wax, and 1% to 2% of sodium alginate.

5. A detergent in bar or cake form consisting essentially of a water soluble salt of an alkyl aryl sulfonate as a detergent, a paraffin wax melting at about 140° F. to 180° F., and sodium alginate, in the proportions of 68% to 84% by weight of said sulfonate, 15% to 30% by weight of said paraffin wax, and 1% to 2% of sodium alginate, said detergent bar containing not more than 8% by weight of water-soluble inorganic salts.

6. A detergent bar or cake consisting essentially of discrete particles of a sodium salt of an alkyl acid sulfate having 12 to 20 carbon atoms per molecule having the sulfate group attached to a carbon atom not more than twice removed from the end of the chain, the surface of said particles being substantially coated with a paraffin wax melting at about 140° F. to 180° F. and forming a substantially continuous phase binding together particles of said sodium alkyl acid sulfate and sodium alginate, in the proportions of 68% to 84% by weight of said sodium alkyl sulfate, 15% to 30% by weight of said paraffin wax, and 1% to 2% of sodium alginate, said detergent bar containing not more than 8% by weight of water-soluble inorganic salts.

7. A detergent bar or cake consisting essentially of discrete particles of a sodium alkyl benzene monosulfonate having an alkyl group of 10 to 16 carbon atoms, the surface of said particles being substantially coated with a paraffin wax melting at about 140° F. to 180° F. and forming a substantially continuous phase binding together particles of said sodium alkyl benzene sulfonate and sodium alginate, in the proportions of 68% to 84% by weight of said sulfonate, 15% to 30% by weight of said paraffin wax, and 1% to 2% of sodium alginate, said detergent bar containing not more than 8% by weight of water-soluble inorganic salts.

WILLEM J. D. VAN DIJCK.
BRADFORD P. GEYER.

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