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(54) Non-evaporative solidification of detergent pastes.

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Description

The present invention relates to the preparation of solid compositions containing non-soap detergents, especially in bar, tablet or powder form, by non-evaporative solidification of aqueous detergent pastes.

The acronym "NSD" will be used herein to refer to the surfactant components of non-soap laundry detergents, which term comprises all those surfactants, other than salts of alkyl and alkenyl carboxylic acids, which are useful as laundry cleaning agents. The term includes the sulphated and sulphonated anionic surfactants, non-ionic, Zwitterionic, amphoteric and semi-polar surfactants and mixtures thereof, including mixtures with minor proportions of soap. The term excludes soap and active mixtures containing major proportions of soap. The term, however, includes those cationic surfactants, which are effective as laundry cleaning agents or fabric softeners generally used in admixture with non-ionic or amphoteric surfactants.

Where the context permits, references herein to "solid" surfactants or detergents are to those which are solid at ambient temperatures.

Until the middle of the present century, soap was the only laundry surfactant of industrial significance. It was used mainly in the form of moulded bars, or sometimes as a powder. It suffers, however, from the major disadvantages that it forms an insoluble curd with alkaline earth metals, which commonly occur in water especially in "hard" water areas.

During the last four decades soap has been largely replaced for laundry use, at least in the industrialised contries, by NSD, which, apart from a small proportion sold as liquid detergents, is invariably supplied as a powder formed by the evaporation of a slurry of NSD, with builders and other auxiliary detergent ingredients, in water. Evaporation is normally effected in a spray drier.

The conventional dried NSD powder, however, has certain disadvantages. The energy cost of evaporating substantial quantities of water is high, and the capital cost and relative sophistication of spray driers is prohibitive for many developing countries seeking to establish an indigenous detergent industry. Moreover, a market persists, even in industrialised countries, for laundry surfactants in the form of bars for repeated use, or of rapidly soluble tablets which can be used as a convenient means of adding measured amounts of surfactant to the wash liquor. Spray drying technology is not well adapted to satisfy this demand.

These difficulties have led to the evolution, especially in some developing countries, of NSD bars for laundry use. Such bars are described in GB—A—1,013,992 and in "Detergent Manufacture" by Marshall Sittig, published by Noyes Data Corp, 1976, pp 325. et seq.

Several techniques have been described for the manufacture of such bars, e.g.: dry mixing of surfactant, builder, and other ingredients, with minimal water added as plasticiser; mixing builder with aqueous NSD solution and evaporating surplus water, and "dry neutralisation", i.e. neutralisation of the substantially anhydrous sulphonic acid precursor of a sulphonated NSD with solid sodium carbonate.

Other references of interest include: CH—A—28 1137, which describes the manufacture of sulphocarboxylates; US—A—3,639,286 which claims and exemplifies the manufacture of solid detergent bars; EP—A—0 014 979 which describes detergent bars containing hydratable salts and AU—B—461648 which claims the neutralisation of a sulphoacid with soda ash in the presence of minimal amounts of water.

In practice the cost of evaporating substantial amounts of water, the physical difficulty of dry mixing the ingredients, and the lack of commercially available or economically viable solidifiable anhydrous surfactants have combined to limit manufacturers to the technique of dry neutralisation. This in turn has effectively limited the choice of NSD for such use to the only commercially available sulphonic acid precursor which is sufficiently stable to be transported from a sulphonation plant to the manufacturing sites in developing countries where NSD bars have hitherto been mainly produced.

The normal technique of manufacture of NSD bars, for nearly 20 years has been to mix the alkyl benzene sulphonic acid precursor with excess sodium carbonate and any other desired ingredients of the finished detergent bar, such as sodium tripolyphosphate or tetrasodium pyrophosphate builder, sodium sulphate filler, non-ionic foam booster, carboxymethyl cellulose soil suspender, sodium silicate, optical brightening agent, perfumes and colourings, at sufficiently elevated temperature to form a plastic mass which may be extruded and cut into solid bars, which in turn may optionally be milled or ground to a powder.

The practical limitation of the dry neutralisation technique to alkyl benzene sulphonate based NSD is a disadvantage, since other surfactant systems are often preferable for reasons of economics and/or effectiveness. In particular, alkyl benzene sulphonates require petrochemical feedstocks on which many developing countries wish to reduce their dependence. Unneutralised sulphonic acids are generally less safe than the corresponding neutralised surfactants to transport and handle.

We have now discovered a method for the manufacture of solid NSD laundry formulations which does not require evaporation of substantial quantities of water, or investment in expensive drying plant, but which permits the use of alkyl sulphates, fatty acid sulphonates and fatty ester sulphonates which can be readily obtained from vegetable oil feestocks commonly available in developing countries. Our invention does not require the transport or handling of unneutralised sulphonic acids. Our invention provides a method of manufacture which is cheap, simple and particularly adapted to the technological requirements of developing countries, but which provides products of superior performance capable of satisfying consumer demands in both developing and industrialised countries.

According to our invention, there is provided a method for the manufacture of solid detergent compositions which comprises mixing an aqueous surfactant, which surfactant comprises at least a major proportion of a water-soluble salt of an acid selected from alkyl sulphuric acids, sulphonated fatty acids, sulphonated fatty esters and mixtures thereof, at a concentration and temperature such that said surfactant is present, at least predominantly, in the "G" phase with at least one builder and, optionally, other ingredients of said detergent compositions, and forming the mixture into a solid composition at least primarily by (A) cooling said "G" phase below its solidification temperature and/or (B) adding sufficient electrolyte to elevate said solidification temperature above ambient and/or (C) removing sufficient water, as water of hydration of at least one hydratable compound, to convert said "G" phase into a solid.

Our invention relies essentially on "non-evaporative solidification" which term embraces those processes which rely primarily upon one or more of the following three phenomena to effect solidification, that is to say:

(A) Cooling of a "G" phase composition which exists as a "G" phase only at elevated temperatures and which is solid at ambient temperatures;

(B) Addition to a fluid "G" phase of sufficient electrolyte to elevate the "G" phase/hydrated solid transition temperature to such an extent that the composition is solid at ambient temperatures; and

(C) Removal of sufficient water from a "G" phase by addition thereto of at least one hydratable compound, to convert the NSD into a solid.

The first of the foregoing phenomena (A) requires a "G" phase which exists only at a substantially elevated temperature and which is converted to the hydrated solid phase on cooling to ambient temperature. Suitable higher temperature "G" phase surfactants include sodium, potassium and tris (hydroxyethyl) ammonium alkyl sulphates, olefin sulphonates and alkyl benzene sulphonates.

The second phenomenon (B) depends on the effects of electrolyte in raising the temperature of the "G" phase/solid phase transition boundary, and hence, effectively increasing the temperature at which the "G" phase solidifies, enabling a normally liquid crystalline surfactant composition to be obtained as the hydrated solid at ambient temperature. Suitable electrolytes to be used in raising the transition temperature include, for example, sodium silicate, sodium sulphate, sodium chloride, or sodium carbonate. Typically, where the surfactant is anionic a common cation is employed to maximise the effect, thus where, as will normally be the case, the surfactant is a sodium salt, the preferred electrolyte will be a water soluble, strongly dissociable sodium salt. The amount of electrolyte required will depend upon the particular NSD and the extent to which the other non-evaporative solidication effects such as cooling or hydration of hydratable salts is relied upon.

The third of the aforesaid phenomena (C) involves the abstraction of water from the "G" phase by a hydratable compound, in an amount sufficient to convert the "G" phase into a hydrated solid. Although any anhydrous hydratable salts capable of exerting a dessicant action on a "G" phase surfactant may be used, the most convenient is anhydrous tetrasodium pyrophosphate or sodium tripolyphosphate, which are commonly included in NSD formulations as builders. The hydratable compound may be a partially hydrated compound.

The method of non-evaporative solidification will often rely upon two or all three of the above phenomena. E.g. addition of sodium silicate and anhydrous sodium tripolyphosphate to a "G" phase surfactant will simultaneously increase the electrolyte concentration, increase the active concentration as the anhydrous tripolyphosphate is converted to hexahydrate, and raise the temperature of the mixture. The latter may, for example, be extruded and cooled to form a solid extrudate.

The term "non-evaporative solidification" does not exclude the possibiltiy that some evaporation of moisture occurs during the process. Incidental evaporation of moisture is inevitable when aqueous materials are handled, especially at elevated temperatures. The term merely indicates that techniques and employed which do not rely on evaporation to effect solidification. It is possible within the scope of our invention to use evaporation in conjunction with the non-evporative techniqes. However, if the evaporation requires expensive drying equipment or removes a sufficiently large proportion of the moisture to increase, substantially, the energy costs, then a part of the economic benefit of the invention will have been forfeited.

The NSD feed in the process of our invention is essentially present as a "G" phase, although it may be possible in certain circumstances to add minor proportions of the NSD in other forms, e.g. as a dilute aqueous solution, or as an anhydrous or partially hydrated solid product. In the latter case the addition of the anhydrous surfactant may contribute to the non-evaporative solidification by raising the active concentration, and may in fact constitue some or all of the hydratable compound.

The "G" phase is a pumpable fluid which is formed over a narrow range of concentrations which range usually lies somewhere between 45% and 80% by weight of active ingredient and is characterised by a lamellar structure in which the surfactant molecules are associated to form plates of indefinite size separated by planes of water molecules.

Typically when alkyl sulphates and ester sulphonates are prepared in aqueous solutions of increasing concentration, the molecules are first found to associate in spherical clusters (micelles), which micelles with increasing concentration become rod-like. At higher concentrations the micelles become more crowded causing a rise in the viscosity of the solution and, in the great majority of cases, eventually rearrange to form a regular hexagonal array of rod-like surfactant micelles in an aqueous medium (the rigid "M_1" liquid crystal phase). If the concentration of a surfactant in the "M_1" phase is progressively increased a phase change occurs to give either a hydrated solid phase, or, if the temperature is sufficiently elevated, to convert the M_1 phase progressively to a fluid "G" phase until a viscosity minimum is reached. Further increase in the concentration of the "G" phase causes the viscosity to rise until a further phase change occurs, leading to the formation of a hydrated solid phase.

The foregoing description is somewhat simiplified. The term "hydrated solid phase" has been used broadly to include those systems which comprise suspensions of solid or immobile gel phases in one or more viscous or gel phase to provide a more or less rigid material.

Mixture, of alkyl sulphates with other NSD have also been found to provide "G" phases. In general, we have found, to a good approximation, that the proportion of active mixture of NSD required to form a "G" phase can be determined from the formula:

$$\frac{C_1}{g_1} + \frac{C_2}{g_2} + \cdots \frac{C_n}{g_n} = 1,$$

where $C_1 \ldots C_n$ are the concentrations of the individual active components and $g_1 \ldots g_n$ are the concentrations at which each component forms a "G" phase of minimum viscosity. This formula enables the concentration of the mixture corresponding to the minimum viscosity "G" phase to be estimated in a majority of cases. Where g is not known, or a component does not form a "G" phase, or the above formula is not applicable, then any "G" phase can be located very rapidly and easily, using standard laboratory equipment by making a test composition having an active concentration of say 75% (or, where appropriate, whatever concentration has been estimated on the basis of the foregoing formula) and placing a sample on a slide on the block of a heated stage microscope. Examination between crossed polarisers will reveal in which phase the sample is present. The various phases each have a characteristic appearance which is easily identified by comparison for example with the photographs of typical liquid crystal phases in the classic paper by Rosevear, JAOCS Vol. 31 P. 628 (1954) or in J. Colloid and Interfacial Science, Vol. 30 No. 4. P. 500. They may also be identified by X-ray or neutron diffraction.

If the mixture is in an M_1 phase, water may be allowed to evaporate from the edges of the sample under the cover disc and any phase changes observed. If any M_2 phase or hydrated solid is present, water may be added around the edge of the cover disc and allowed to diffuse into the composition. If no "G" phase is located in this way samples may be heated progressively on the block and the operations repeated.

Usually, the composition is pumpable at concentrations within a range of ±10%, preferably ±5%, e.g. ±2.5% of the minimum viscosity concentration. This range tends to be broader at more elevated temperatures. Compositions may be obtained, at the limits of the range in which one or more solid or gel phase is suspended in a continuous "G" phase. Such compositions may be useful according to the invention.

The "G" phase may generally be prepared by converting a suitable precursor to the NSD in the presence of the appropriate quantity of water (e.g. an alkyl sulphuric or fatty ester sulphonic acid may be neutralised with aqueous alkali of the appropriate strength). Mixed NSD may be prepared either by mixing the separately prepared components, each preferably in the "G" phase, or where this is impractical, by preparing one component from its precursor in the presence of the other component as described in our British Patent 2,022,125.

Typically alkyl sulphates avaialble for NSD solid formulations have been either substantially anhydrous solids and therefore difficult to mix with other ingredients, or more usually dilute solutions containing more than 60% water, which cannot be solidified at ambient temperatures except by evaporation of a major proportion of the water content. Non-evaporative solidification has not therefore been contemplated. Our invention takes advantage of a property of the more concentrated fluid "G" phase alkyl sulphates, namely their sensitivity to small changes in active or electrolyte concentration or of temperature, which enables them to be much more easily converted into hydrated solids than the more conventional detergent solutions.

The alkyl sulphate for use according to our invention may be potassium, ammonium or lower alkyl ammonium or alkylolammonium alkyl sulphate. Particularly preferred are sodium alkyl sulphates. Sodium alkyl sulphates do not form "G" phases at ambient temperatures and are normally only available as dilute solutions less than 30% active concentration. Solid anhydrous or hydrated sodium alkyl sulphate cannot normally be melted, but tends to decompose on heating. The instability of alkyl sulphuric acids has prevented the adoption of dry neutralisation for making sodium alkyl sulphate based solid NSD formulations. Yet, alkyl sulphates would be attractive alternatives to alkyl benzene sulphonate for many developing countries who could prepare the former by sulphation of locally produced vegetable based feedstocks rather than imported petrochemical feedstocks required for the latter.

However, if sodium alkyl sulphates are prepared at temperatures of, e.g. 70°C, or even lower where mixed alkyl feedstocks were used, and the water used in the neutralisation is controlled within narrow limits to form the "G" phase, a pumpable and stable hot fluid can be obtained which is mixable with other detergent ingredients and which can be solidified by cooling to form a solid composition.

The solid composition will normally be formed by extrusion. Alternatively, where the hot alkyl sulphate mixture is sufficiently fluid it may be injected into a mould and cooled to form cast bars. Also, depending upon the consistency and physical form of the mixture, it may be milled to a powder and sold as such, or compacted into rapidly soluble tablets.

The alkyl sulphate for use according to preferred embodiments of our invention is typically sodium C_{8-20} linear alkyl sulphates, preferably a mixed C_{10-18} alkyl sulphate. Particularly preferred is lauryl alkyl sulphate. Other alkyl sulphates which may be used include potassium, lithium, ammonium, monoethanolamine, diethanolamine, triethanolamine and mono-, di- tri- and tetra-alkylammonium salts having up to 6 carbon atoms, preferably 1 to 4 carbon atoms per cation. Salts of mixtures of the aforesaid cations and branched chain alkyl sulphates are also useful.

The alkyl sulphate may be used in admixture with any other "G" phase forming anionic NSD, or nonionic, Zwitterionic, amphoteric or semi-polar NSD or mixtures thereof. Cationic surfactants, preferably in conjunction with non-ionic or amphoteric surfactants, may also be used.

Preferably the alkyl sulphate constitutes at least 30% more preferably at least 50%, e.g. at least 75% of the total surfactant. In particular, we prefer the the alkyl sulphate constitute at least 80% and preferably substantially all of the anionic surfactant. Mixtures of alkyl sulphates with minor proportions of non-ionic surfactants, especially alkylolamides, are particularly preferred. The non-ionic surfactant is preferably present in a proportion of from 1 to 49%, especially 2 to 20%, e.g. 3 to 10% of the total weight of surfactant.

The alkyl sulphate may be used in conjunction with, or be replaced by, water soluble salts of a sulphonated fatty acid or ester such as the methyl ester of coconut fatty acid.

The surfactant may contain an at least sparingly water-soluble, alkali metal, ammonium, alkylolamine or amine, alkylbenzene sulphonate, alkyl ether sulphate, olefin sulphonate, alkane sulphonate, alkyl phenol sulphate, alkyl phenol ether sulphate, alkylethanolamide sulphate, or alkylethanolamide ether sulphate, each having at least one alkyl or alkenyl group with from 10 to 20 aliphatic carbon atoms. Said alkyl or alkenyl groups are preferably straight chain primary groups but may optionally be secondary, or branched chain groups. The expression "ether" hereinbefore refers to polyoxyethylene, polyoxypropylene, glyceryl and mixed polyoxyethylene-oxy propylene or mixed glyceryl-oxyethylene or glyceryl-oxy propylene groups, typically containing from 1 to 20 oxyalkylene groups. For example, the sulphonated or sulphated surfactant may be sodium dodecyl benzene sulphonate, potassium hexadecyl benzene sulphonate, sodium dodecyl dimethyl benzene sulphonate, ammonium lauryl monethoxy sulphate, or monoethanolamine cetyl 10 mole ethoxylate sulphate.

Other anionic surfactants useful according to the present invention include fatty alkyl sulphosuccinates, fatty alkyl ether sulphosuccinates, fatty alkyl sulphosuccinamates, fatty alkyl ether sulphosuccinamates, acyl sarcosinates, acyl taurides, isethionates, and alkyl ether carboxylates. Anionic phosphate fatty esters may also be used.

Preferred anionic surfactants are sodium salts. Other salts of commercial interest include those of potassium, lithium, ammonium, monoethanolamine, diethanolamine, triethanolamine, and alkyl amines containing up to six aliphatic carbon atoms.

The surfactants may optionally but preferably contain nonionic surfactants. The nonionic surfactant may preferably be, e.g. a C_{10-22} ethanolamide such as coconut monoethanolamide. Other nonionic surfactants which may optionally be present, include ethoxylated alcohols, ethoxylated carboxylic acids, ethoxylated amines, ethoxylated alkylolamides, ethoxylated alkylphenols, ethoxylated glyceryl esters, ethoxylated sorbitan esters, ethoxylated phosphate esters, and the propoxylated, or ethoxylated and propoxylated, analogues of all the aforesaid ethoxylated nonionics, all having a C_{8-22} alkyl or alkenyl group, or any other nonionic surfactant which has hitherto been incorporated in powder compositions, e.g., amine oxides.

The preferred nonionics for our invention are the less water soluble compounds, for example those ethoxylates having an HLB range of 7—18, e.g 12—15. However, more water soluble ones may sometimes be present, usually in minor amounts. In particular, fatty alkanolamides may be useful, in minor proportions, as internal lubricants to assist in the mixing and plodding of the alkyl sulphates.

Compositions of our invention may also contain amphoteric surfactant, usually as a minor component of the active material.

Amphoteric surfactants include betaines and sulphobetaines formed by substituting a suitable tertiary nitrogen compound having a long chain alkyl or alkenyl group with a β-acid-forming substituting agent such as chloroacetic acid. Examples of suitable tertiary nitrogen containing compounds include: tertiary amines having one or two long chain alkyl or alkenyl groups, optionally a benzyl group and any other substituent a short chain alkyl group; imidazoline having one or two long chain alkyl or alkenyl groups and amidoamines having one or two long chain alkyl or alkenyl groups.

The surfactant is mixed with builders and other auxiliary detergent ingredients.

The term "builder" is sometimes used loosely in the detergent art to include any non-surfactant whose presence in a detergent formulation enhances the cleaning effect of the surfactant. More usually, however, the term is restricted to those typical "builders", which are primarily useful as means of preventing or ameliorating the adverse effects on washing of calcium ions, e.g. by chelation, sequestering, precipitation or absorption of the ions, and secondarily as a source of alkalinity and buffering. The term "builder" as used herein refers to additives which produce the foregoing effects to a substantial extent. It includes sodium tripolyphosphate and other inorganic phosphate and condensed phosphate salts such as sodium or potassium orthophophates, pyrophosphates, metaphosphates or tetraphosphate, as well as phosphonates such as acetodiphosphonates and amino tris methylene phosphonates. It also includes alkali metal carbonates, zeolites and other "molecular sieves" and such organic sequestrants as salts nitrilotriacetic acid, citric acid and ethylene diamine tetracetic acid.

Compositions of our invention may contain fillers which help to provide a suitable physical form of bar.

Other auxiliary detergent ingredients may comprise anti redeposition agents such as sodium carboxy methyl cellulose, perfumes, dyes, optical brightening agents, hydrotropes, buffers, bleach activators, alkalis and the like. Typically such auxiliairies are present in proportions of less than 10% and more usually less than 5% wt. each and less than 25%, e.g., less than 15% total.

Typically, an NSD bar according to our invention will have the following composition:

Surfactant	10—40%
Phosphate or phosphonate builder	5—45%
Carbonate or bicarbonate	0—40%
Silicate	0—20%
Filler	0—50%
Other auxiliaries	0—20%
Water	5—20%

All percentages are by weight and all ingredients (except water) are quoted as the anhydrous compounds.

The filler may comprise any inert solid having suitable physical properties, e.g. sodium sulphate or talc. The other auxiliaries may include any or all of:— sodium carboxymethyl cellulose or other soil suspending agent; enzymes; dyestuffs; perfumes; optical brighteners; oxidising bleaches such as peroxides, perborates, percarbonates or hypochlorites; gums; starches; water soluble synthetic polymers; organic polyelectrolytes such as polyacrylic acid; and hydrotropes. Tablets for addition to washing machines may contain minor proportions of soap as foam depressants, and/or silicone anti foams.

The properties of the "G" phase surfactant may further be modified by the inclusion of mono- di- or tri-glycerides, fatty alcohols, glycerol, glycols, polyethylene glycol or polypropylene glycol, which may help to lubricate the mixture in cases where difficulties are encountered with mixing or extrusion.

The invention will be illustrated by the following examples in which all percentages are by weight based on the total weight of the composition:—

	Ex. 1	Ex. 2	Ex. 3	Ex. 4
Sodium lauryl sulphate paste*	30%	30%	30%	30%
Palm kernel monoethanolamide powder	—	3%	—	—
Fatty alcohol	—	—	1%	1%
Anhydrous tetra-sodium pyrophosphate	18%	18%	18%	18%
Anhydrous sodium carbonate	12%	11%	12%	12%
Sodium bicarbonate	6%	6%	6%	6%
Calcite (Registered Trade Mark "OMYA" D40 powder)	19%	18%	19%	18%
Talc	1%	1%	1%	1%
Sodium carboxymethyl cellulose	1%	1%	1%	1%
Anhydrous sodium sulphate	6%	6%	6%	7%
Sodium silicate liquor (46% $Na_2O \cdot 2SiO_2$ solids)	—	—	—	16
Water	7%	6%	6%	—

*The sodium lauryl sulphate paste had the following composition:—

Active matter (MMW=303)	65%
Free fatty matter	1.25%
Sodium sulphate	4%
Water	Balance

The composition of the fatty alcohol was:—

$\%C_{10}$	2.7
$\%C_{12}$	59.8
$\%C_{14}$	19.4
$\%C_{16}$	8.0
$\%C_{18}$	10.0
$\%C_{20}$	0.1

The above examples were prepared in the following way.

1) The powdered ingredients were mixed together in a 1 gallon (approximately 4.5 litres) capacity "Hobart" dough mixture machine.

2) Fatty alcohol, where used, was then added in the liquid state and stirred into the powder mix.

3) The sodium lauryl sulphate paste, pre-heated to a temperature of about 60°C, to convert it into the mobile "G" phase, was then added and stirred into the powder mix.

4) Finally, either the sodium silicate liquor or water was incorporated.

The resulting phase was then allowed to age for a minimum period of 1/2 hour to allow it to harden off.

The mixtures were then passed 3—4 times through a 30 rpm. 200 lbs/ft (approximately 300 kg/m) "sunlab" plodding machine to make them plastic enough to be extruded through the same machine in the form of bars.

Claims

1. A method for the manufacture of solid detergent compositions which comprises mixing an aqueous surfactant, which surfactant comprises at least a major proportion of a water-soluble salt of an acid selected from alkyl sulphuric acids, sulphonated fatty acids, sulphonated fatty esters and mixtures thereof, at a concentration and temperature such that said surfactant is present, at least predominantly, in the "G" phase, with at least one builder and, optionally, other ingredients of said detergent compositions, and forming the mixture into a solid composition at least primarily by (A) cooling said "G" phase below its

solidification temperature and/or (B) adding sufficient electrolyte to elevate said solidification temperature above ambient and/or (C) removing sufficient water, as water of hydration of at least one hydratable compound, to convert said "G" phase into a solid.

2. A method according to claim 1 wherein the surfactant comprises at least 50% by weight of alkyl sulphate.

3. A method according to claim 2 wherein the surfactant comprises at least 75% by weight of alkyl sulphate.

4. A method according to any foregoing claim wherein the surfactant comprises from 1 to 44% by wt. non-ionic surfactant.

5. A method according to claim 4 wherein the surfactant comprises from 2 to 20% by weight of non-ionic surfactant.

6. A method according to any foregoing claim wherein the surfactant consists essentially of anionic and non-ionic surfactant.

7. A method according to any foregoing claim wherein at least 80% by wt. of the total anionic surfactant is alkyl sulphate.

8. A method according to claim 7 wherein the anionic surfactant consists essentially of alkyl sulphate.

9. A method according to any foregoing claim wherein the alkyl sulphate consists essentially of sodium alkyl sulphate.

10. A method according to any foregoing claim wherein the alkyl sulphate is a lauryl alkyl sulphate.

11. A method according to any foregoing claim wherein the nonionic surfactant comprises an alkanolamide.

12. A method according to claim 11 wherein the alkanolamide is coconut mono- or di-ethanolamide.

13. A method according to any foregoing claim wherein the final composition contains up to 50% by wt. of a filler.

14. A method according to claim 13 wherein the filler is talc or sodium sulphate.

15. A method according to any foregoing claim wherein the final composition contains from 5 to 30% by weight of builder.

16. A method according to any foregoing claim wherein the builder is a condensed phosphate or phosphonate salt.

17. A method according to claim 16 wherein the builder is sodium tripolyphosphate or tetra sodium pyrophosphate.

18. A method according to any foregoing claim wherein the final composition comprises up to 40% by wt. of carbonate, and/or bicarbonate.

19. A method according to any foregoing claim wherein the composition comprises up to 20% by weight of sodium silicate.

20. A method according to any foregoing claim wherein the "G" phase surfactant is mixed with the builder at elevated temperature and solidification is effected, at least in part, cooling the mixture to ambient temperature.

21. A method according to any foregoing claim wherein solidification is effected, at least in part, by adding an, at least partially, anhydrous, hydratable salt to the mixture, which combines with water in the mixture to form a hydrate.

22. A method according to claim 21 wherein the hydratable salt comprises the builder.

23. A method according to any foregoing claim wherein solidification is effected, at least in part, by adding an electrolyte to the mixture sufficient to raise the "G" phase/hydrated solid transition temperature of the surfactant above ambient.

24. A method according to any foregoing claim wherein the composition is plodded and extruded as solid bars.

25. A method according to any foregoing claim wherein the composition is milled to a powder or flake.

26. A method according to any foregoing claim wherein the composition is formed into tablets.

Patentansprüche

1. Verfahren zur Herstellung fester Detergens-Zusammensetzungen, bei welchem ein oberflächenaktives Mittel auf wässeriger Basis, das zumindest einen größeren Anteil eines wasserlöslichen Salzes einer Säure, ausgewählt aus den Alkylschwefelsäuren, sulfonierten Fettsäuren, sulfonierten Fettestern und deren Mischungen, enthält, in einer Konzentration und bei einer Temperatur, bei welcher das oberflächenaktive Mittel zumindest hauptsächlich in der "G"-Phase vorliegt, mit zumindest einem Builder und gegebenenfalls anderen Bestandteilen der Detergens-Zusammensetzungen gemischt und die Mischung in eine feste Zusammensetzung übergeführt wird, was zumindest hauptsächlich erfolgt durch (A) Abkühlen der "G"-Phase auf eine Temperatur unterhalb ihrer Verfestigungstemperatur und/oder (B) Zusatz einer ausreichenden Elektrolytmenge zur Anhebung dieser Verfestigungstemperatur über die Raumtemperatur und/oder (C) Abtrennung ausreichender Wassermengen als Hydrationswasser mindestens einer hydratisierbaren Verbindung, um die "G"-Phase in einem Feststoff umzuwandeln.

2. Verfahren nach Anspruch 1, bei welchem das oberflächenaktive Mittel zumindest 50 Gew.-% Alkylsulfat enthält.

3. Verfahren nach Anspruch 2, bei welchem das oberflächenaktive Mittel zumindest 75 Gew.-% Alkylsulfat enthält.

4. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem das oberflächenaktive Mittel 1 bis 44 Gew.-% nichtionisches oberflächenaktives Mittel enthält.

5. Verfahren nach Anspruch 4, bei welchem das oberflächenaktive Mittel 2 bis 20 Gew.-% nichtionisches oberflächenaktives Mittel enthält.

6. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem das oberflächenaktive Mittel hauptsächlich aus einem anionischen und einen nichtionischen oberflächenaktiven Mittel besteht.

7. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem zumindest 80 Gew.-% des gesamten anionischen oberflächenaktiven Mittels aus einem Alkylsulfat bestehen.

8. Verfahren nach Anspruch 7, bei welchem das anionische oberflächenaktive Mittel im wesentlichen aus Alkylsulfat besteht.

9. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem das Alkylsulfat im wesentlichen aus Natriumalkylsulfat besteht.

10. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem das Alkylsulfat aus einem Laurylalkylsulfat besteht.

11. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem das nichtionische oberflächenaktive Mittel ein Alkanolamid enthält.

12. Verfahren nach Anspruch 11, bei welchem das Alkanolamid aus Kokossäuremono- oder -diethanolamid besteht.

13. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die endgültige Zusammensetzung bis zu 50 Gew.-% eines Füllstoffes enthält.

14. Verfahren nach Anspruch 13, bei welchem der Füllstoff aus Talkum oder Natriumsulfat besteht.

15. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die endgültige Zusammensetzung 5 bis 30 Gew.-% eines Builders enthält.

16. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem der Builder aus einem kondensierten Phosphat- oder Phosphonatsalz besteht.

17. Verfahren nach Anspruch 16, bei welchem der Builder aus Natriumtripolyphosphat oder Tetranatriumpyrophosphat besteht.

18. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die endgültige Zusammensetzung bis zu 40 Gew.-% Carbonat und/oder Bicarbonat enthält.

19. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die Zusammensetzung bis zu 20 Gew.-% Natriumsilikat enthält.

20. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem das sich in der "G"-Phase befindende oberflächenaktive Mittel bei erhöhter Temperatur mit dem Builder gemischt und die Verfestigung zumindest teilweise durch Abkühlen der Mischung auf Raumptemperatur erreicht wird.

21. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die Verfestigung zumindest teilweise erzielt wird durch Zusatz eines zumindest teilweise wasserfreien, hydratisierbaren Salzes, welches sich mit Wasser in der Mischung unter Bildung eines Hydrats vereinigt, zu der Mischung.

22. Verfahren nach Anspruch 21, bei welchem das hydratisierbare Salz einen Builder enthält.

23. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die Verfestigung zumindest teilweise erzielt wird durch Zusatz eines Elektrolyten zu der Mischung in ausreichender Menge zur Anhebung der Übergangstemperatur des oberflächenaktiven Mittels von "G"-Phase in hydratisierten Feststoff über die Raumtemperatur.

24. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die Zusammensetzung stranggepreßt und in Form fester Stangen extrudiert wird.

25. Verfahren nach irgendeinem der vorhergehenden Ansprüche, bei welchem die Zusammensetzung zu einem Pulver oder in Flockenform vermahlen wird.

26. Verfharen nach irgendeinem der vorhergehenden Ansprüche, bei welchem die Zusammensetzung zu Tabletten verformt wird.

Revendications

1. Procédé de fabrication de compositions détergentes solides qui comprend le mélange d'un tensioactif aqueux, ce tensioactif comprenant au moins une proportion majeure d'un sel hydrosoluble d'un acide choisi parmi les acides alkylsulfuriques, les acides gras sulfonés, les esters gras sulfonés et leurs mélanges, en une concentration et à une température telles que ledit tensioactif soit présent, au moins de façon prédominante, dans la phase "G", avec au moins un adjuvant et, éventuellement, d'autres ingrédients desdites compositions détergentes, et la mise en forme du mélange pour donner une composition solide, au moins principalement (A) en refroidissant ladite phase "G" au-dessous de sa température de solidification et/ou (B) en ajoutant suffisamment d'électrolyte pour élever ladite température de solidification au-dessus de la température ambiante et/ou (C) en éliminant suffisamment d'eau, sous forme d'eau d'hydration d'au moins un composé hydratable, pour transformer ladite phase "G" en un solide.

2. Procédé selon la revendication 1, dans lequel le tensioactif comprend au moins 50% en poids d'alkylsulfate.

3. Procédé selon la revendication 2, dans lequel le tensioactif comprend au moins 75% en poids d'alkylsulfate.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel le tensioactif comprend de 1 à 44% en poids de tensioactif non ionique.

5. Procédé selon la revendication 4, dans lequel le tensioactif comprend de 2 à 20% en poids de tensioactif non ionique.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel le tensioactif est essentiellement constitué de tensioactif anionique et de tensioactif non ionique.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel au moins 80% en poids du tensioactif anionique total est un alkylsulfate.

8. Procédé selon la revendication 7, dans lequel le tensioactif anionique est essentiellement constitué d'alkylsulfate.

9. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'alkylsulfate est essentiellement constitué d'alkylsulfate de sodium.

10. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'alkylsulfate est un laurylalkylsulfate.

11. Procédé selon l'une quelconque des revendication précédentes, dans lequel le tensioactif non ionique comprend un alcanolamide.

12. Procédé selon la revendication 11, dans lequel l'alcanolamide est du mono- ou du di-éthanolamide de noix de coco.

13. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition finale contient jusqu'à 50% en poids d'une charge.

14. Procédé selon la revendication 13, dans lequel la charge est du talc ou du sulfate de sodium.

15. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition finale contient de 5 à 30% en poids d'un adjuvant.

16. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'adjuvant est un sel phosphate du phosphonate condensé.

17. Procédé selon la revendication 16, dans lequel l'adjuvant est du tripolyphosphate de sodium ou du pyrophosphate tétrasodique.

18. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition finale comprend jusqu'à 40% en poids de carbonate et/ou de bicarbonate.

19. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition comprend jusqu'à 20% en poids de silicate de sodium.

20. Procédé selon l'une quelconque des revendications précédentes, dans lequel le tensioactif en phase "G" est mélangé à l'adjuvant à température élevée et la solidification est réalisée, au moins en partie, par refroidissement du mélange à la température ambiante.

21. Procédé selon l'une quelconque des revendications précédentes, dans lequel la solidification est réalisée, au moins en partie, par addition d'un sel hydratable, au moins partiellement anhydre, au mélange, qui se combine à l'eau du mélange pour former un hydrate.

22. Procédé selon la revendication 21, dans lequel le sel hydratable comprend l'adjuvant.

23. Procédé selon l'une quelconque des revendications précédentes, dans lequel la solidification est réalisée, au moins en partie, par addition d'un électrolyte au mélange, en quantité suffisante pour élever la température de transition phase "G"/solide hydraté du tensioactif au-dessus de la température ambiante.

24. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition est boudinée et extrudée en pains solides.

25. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition est moulue pour donner une poudre ou des paillettes.

26. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition est mise sous forme de tablettes.