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(54) Detergent compositions.

(57) For optimum detergency, particulate detergent compositions built with aluminosilicate (25-45 wt%), and containing an anionic surfactant component (5-35 wt%) consisting of primary alcohol sulphate (PAS) (10-100 wt%) optionally with linear alkylbenzene sulphonate (LAS) (0-90 wt%), contain 0-20 wt% sodium carbonate depending on the relative proportions of PAS and LAS present :
0-10 wt% carbonate for 10-60 wt% PAS/40-90 wt% LAS,
0-20 wt% carbonate for 60-80 wt% PAS/20-40 wt% LAS, and
10-20 wt% carbonate for 80-100 wt% PAS/0-20 wt% LAS.

EP 0 460 925 A2

TECHNICAL FIELD

The present invention relates to particulate detergent compositions, particularly those of high bulk density, containing primary alcohol sulphate (PAS), built with alkali metal aluminosilicate, and optionally containing sodium carbonate.

BACKGROUND AND PRIOR ART

Detergent compositions built with zeolite and containing both alkylbenzene sulphonate and PAS are disclosed in JP 62 240 397A (Kao), JP 62 273 300A (Kao), EP 219 314A (Procter & Gamble) and EP 220 024A (Procter & Gamble).

JP 01 142 000A (Nippon Gosen Senzai) discloses a high bulk density detergent composition prepared by neutralising PAS acid with sodium carbonate and zeolite.

EP 342 917A (Unilever) discloses detergent compositions containing PAS having a specific chain length distribution.

Our copending European Patent Application No. 91 300 456.0 filed on 21 January 1991 discloses detergent compositions containing zeolite, sodium carbonate, and branched-chain PAS.

EP 340 013A (Unilever) relates to high-bulk-density detergent powders containing a moderate to high proportion of surfactant (particularly anionic surfactant) and a relatively high proportion of zeolite builder. The preferred and exemplified anionic surfactant is alkylbenzene sulphonate. The powders are preferably prepared by processes involving granulation and densification of a spray-dried powder in a high-speed mixer/granulator having both a stirring action and a cutting action.

The present invention is based on the observation that, in detergent compositions of the general type disclosed in EP 340 013A (Unilever), when PAS is used partially or wholly in place of alkylbenzene sulphonate, surprisingly improved detergency can be achieved by adjustment of the amount of sodium carbonate present.

DEFINITION OF THE INVENTION

The present invention accordingly provides a particulate detergent composition which comprises:

(a) from 17 to 35 wt% of non-soap detergent-active material consisting essentially of:
  (i) from 5 to 35 wt% of an anionic surfactant component consisting of primary alcohol sulphate [10-100 wt% of (i)] optionally together with alkylbenzene sulphonate [0-90 wt% of (i)],
  (ii) optionally from 0 to 10 wt% of nonionic surfactant,
  (iii) optionally from 0 to 10 wt% of anionic surfactant other than primary alcohol sulphate or alkylbenzene sulphonate,
(b) optionally from 0 to 10 wt% of fatty acid soap,
(c) from 25 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate,
(d) from 0 to 10 wt% of sodium carbonate if the anionic surfactant component (a)(i) contains 10-60 wt% of primary alcohol sulphate, from 0 to 20 wt% of sodium carbonate if the anionic surfactant component (a)(i) contains 60-80 wt% primary alcohol sulphate, and from 10 to 20 wt% of sodium carbonate if the anionic surfactant component (a)(i) contains 80-100 wt% primary alcohol sulphate,
(e) optionally other detergent ingredients to 100 wt%.

DETAILED DESCRIPTION OF THE INVENTION

The subject of the invention is a detergent composition in particulate form characterised by a moderate to high content of anionic surfactant, including or consisting of primary alcohol sulphate; a relatively high level of aluminosilicate builder; and a suitably chosen level of sodium carbonate ranging from zero to moderately high.

Preferably the weight ratio of aluminosilicate builder (c) to total non-soap surfactant (a) is within the range of from 0.9:1 to 2.6:1, more preferably from 1.2:1 to 1.8:1.

These amounts and ratios have been found to give excellent detergency and, in the preferred embodiment of the invention according to which the composition is of high bulk density, improved processability and powder properties.

The surfactant system

The detergent composition of the invention comprises from 17 to 35 wt% of non-soap detergent-active material, of which a specified anionic surfactant component is an essential ingredient. The specified anionic sur-

factant component, which constitutes from 5 to 35 wt% of the whole composition, consists either of primary alcohol sulphate (PAS) alone, or of PAS in admixture with linear alkylbenzene sulphonate (LAS). If both LAS and PAS are present, PAS must constitute at least 10 wt%, and preferably at least 20 wt%, of the two together, while LAS can constitute up to 90 wt%, and preferably up to 80 wt%, of the two together.

As explained in more detail below, the amount of LAS present and the relative proportions of LAS and PAS determine the amount of sodium carbonate, if any, present.

The primary alcohol sulphate

An essential ingredient of the surfactant system is a primary alcohol sulphate (PAS), also known as alkyl sulphate. PAS constitutes from 10 to 100 wt% of the essential anionic surfactant component in the compositions of the invention, which itself constitutes from 5 to 35 wt% of the whole composition.

These anionic surfactants have received much attention recently as potential replacements for alkylbenzene sulphonates on environmental grounds, but have shown some deficiencies in all-round detergency performance under a wide range of wash conditions.

Alcohol sulphates may be derived from both synthetic and natural alcohols containing from about 8 to 22 carbon atoms. Examples of suitable alcohols that can be used for PAS manufacture include decyl, lauryl, myristyl, palmityl and stearyl alcohols, and the mixture of fatty alcohols derived by reducing the glycerides of tallow and coconut oils. Natural alcohols, for example, tallow or coconut alcohol, give rise to straight-chain (linear) PAS, while synthetic alcohols, for example those produced by the Oxo process, can give rise to either linear or branched-chain PAS. Both linear and branched PAS are suitable for use in the present invention.

Most commercially available PAS is a mixture containing a spread of chain lengths. The PAS used in the compositions of the present invention is preferably of relatively short chain length, that is to say, it consists wholly or predominantly of material having an alkyl chain length of C_{16} or below, and more preferably consists wholly or predominantly of material having an alkyl chain length of C_{14} or below. Short-chain PAS is especially suitable for products intended solely or predominantly for low-temperature (≦25°C) washing conditions.

Preferred PAS of natural origin is derived from coconut oil, palm kernel oil, babassu oil, or macauba oil.

Especially preferred is coconut alcohol PAS (cocoPAS), either as the natural material consisting predominantly of linear C_{12} and C_{14} alcohols with smaller amounts of shorter- and longer-chain material, or as a "narrow-cut" material enriched in C_{12} and C_{14} alcohols by fractionation. CocoPAS exhibits good low-temperature detergency, is of renewable natural origin, and is biodegradable.

In products intended solely or predominantly for use in low-temperature wash conditions, longer-chain PAS such as tallow PAS is preferably absent, or present in a relatively low proportion, because its low-temperature performance is poor.

The linear alkylbenzene sulphonate

As previously indicated, the main anionic surfactant component, which constitutes 5 to 35 wt% of the composition, may comprise linear alkylbenzene sulphonate (LAS) in addition to PAS, provided that PAS constitutes at least 10 wt%, and preferably at least 20 wt%, of the two together.

Linear alkylbenzene sulphonates are exceedingly well-known ingredients of fabric washing detergent compositions. The alkyl chain length is generally in the C_8-C_{15} range. These materials are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

Optional nonionic surfactant

The compositions of the invention may also contain up to 10 wt% of nonionic surfactant. Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the aliphatic C_{12}-C_{15} primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol; and alkylpolyglycosides.

Optional anionic surfactant

The composition may alternatively or additionally contain up to 10 wt% of one or more anionic surfactants other than LAS and PAS. Examples of suitable anionic surfactants are alkyl ether sulphates, alkyl xylene sulphonates, olefin sulphonates, dialkyl sulphosuccinates and fatty acid ester sulphonates.

These lists are not intended to be exhaustive and for further examples the reader is referred to the standard

literature, for example, "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

Optional soap

As well as non-soap detergents, the composition of the invention may contain up to 10 wt% of fatty acid soap, to provide foam control and additional detergency and builder power. Soap, if present, is not included within the total of 17 to 35 wt% which refers only to non-soap surfactant.

The aluminosilicate detergency builder

The alkali metal (preferably sodium) aluminosilicate builder present in the composition of the invention may be crystalline or amorphous or a mixture thereof. The sodium salts have the general formula

$$0.8\text{-}1.5\ Na_2O.Al_2O_3.0.8\text{-}6\ SiO_2.$$

These materials contain some bound water and are required to have a calcium ion exchange capacity of at least about 50 mg CaO/g. The preferred aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above) and have a particle size of not more than about 100 microns, preferably not more than about 20 microns. Both amorphous and crystalline aluminosilicates can be made readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

Crystalline aluminosilicates (zeolites) are preferred for use in the present invention. Suitable materials are described, for example, in GB 1 473 201 (Henkel) and GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof. Especially preferred for use in the present invention is Type 4A zeolite.

Also of interest is the novel zeolite - maximum aluminium zeolite P - described and claimed in EP 384 070A (Unilever).

The aluminosilicate detergency builder is present in an amount of from 25 to 45 wt% (anhydrous basis), and preferably at the relatively high level of 28 to 45 wt%. This has been found to give processing advantages for high-bulk-density compositions (see below) as well as good cleaning performance.

Optional supplementary detergency builder

If desired, a supplementary builder may also be present. Preferred supplementary builders are organic sequestrant builders. Examples include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates. Alternatively, organic precipitant builders such as alkyl- and alkenylmalonates and succinates, and sulphonated fatty acid salts, may be used.

Especially preferred supplementary builders are polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, especially from to 10 wt%; and monomeric polycarboxylates, more especially citric acid and its salts, suitably used in amounts of from 3 to 20 wt%, more preferably from 5 to 15 wt%.

The composition of the invention preferably does not contain more than 5 wt% of inorganic phosphate builders, and is desirably substantially free of phosphate builders.

Sodium carbonate

The amount of sodium carbonate present is a key feature of the invention.

If the main anionic surfactant component (a)(i) consists of 80-100 wt% PAS and 0-20 wt% LAS, the composition of the invention contains, as an essential ingredient, sodium carbonate in an amount of from 10 to 20 wt%. It has surprisingly been found that the presence of sodium carbonate at this level gives a detergency advantage over similar compositions containing lower amounts of, or no, carbonate; while the amount of carbonate has no significant effect on a similar LAS-only system.

If, on the other hand, the main anionic surfactant component (a)(i) consists of 10-60 wt% PAS and 40-90 wt% LAS, the amount of carbonate should be low or zero -from 0 to 10 wt% - for optimum detergency.

Surprisingly, in this area the detergency performance obtained from combinations of PAS and LAS is better than that of either surfactant alone.

A preferred anionic surfactant component within this area, where low carbonate is preferred, comprises 20-50 wt% PAS and 50-80 wt% LAS, more preferably 30-40 wt% PAS and 60-70 wt% LAS.

At the crossover region of 60-80 wt% PAS and 20-40 wt% LAS, the amount of carbonate appears to have a less marked effect on the detergency and can therefore range from nil to 20 wt%. However, a low carbonate level of nil to 10 wt% is preferred, especially at 60-75 wt% PAS and 25-40 wt% LAS.

High bulk density

According to a preferred embodiment of the invention, the composition has a bulk density of least 650 g/litre, more preferably at least 700 g/litre. Particle porosity preferably does not exceed 0.25, and more preferably does not exceed 0.20.

High-bulk-density compositions in accordance with the invention may be prepared by a variety of processes, batch or continuous, some involving post-tower densification of a spray-dried powder, and others involving wholly non-tower processing.

One type of process involves subjecting a particulate starting material (in effect, a particulate detergent of conventional bulk density) to a granulation/densification treatment. The starting material may itself be prepared by spray-drying or by a non-tower process such as dry mixing or granulation.

This treatment may be carried out in a high-speed batch mixer/granulator having both a stirring action and a cutting action, as described and claimed in EP 340 013A (Unilever). Preferably the stirrer and the cutter may be operated independently of one another, and at separately variable speeds. Such a mixer is capable of combining a high energy stirring input with a cutting action, but can also be used to provide other, gentler stirring regimes with or without the cutter in operation. It is thus a highly versatile and flexible piece of apparatus.

A preferred type of batch high-speed mixer/granulator is bowl-shaped and preferably has a substantially vertical stirrer axis. Especially preferred are mixers of the Fukae (Trade Mark) FS-G series manufactured by Fukae Powtech Kogyo Co., Japan; this apparatus is essentially in the form of a bowl-shaped vessel accessible via a top port, provided near its base with a stirrer having a substantially vertical axis, and a cutter positioned on a side wall. The stirrer and cutter may be operated independently of one another, and at separately variable speeds.

The Fukae mixer may also be used to produce compositions of the invention directly from raw materials by high-speed mixing and granulation. PAS and LAS may be introduced into the mixer in, for example, powder, flake, noodle or paste form. It is also possible to use processes in which PAS or LAS or both are prepared by neutralisation in situ, for example, as described and claimed in EP 352 135A (Unilever) and in our copending European Patent Application No. 91 300 422.2.

As indicated previously, the Fukae mixer requires batch operation. Alternatively, continuous processes may be employed, for example, using a continuous high-speed mixer/granulator such as the Lödige (Trade Mark) Recycler, optionally followed by a moderate-speed continuous mixer/granulator such as the Lödige Ploughshare. As with the Fukae mixer, this apparatus can be used for both post-tower and non-tower processes, including in-situ neutralisation. Suitable processes are disclosed in EP 367 339A, EP 390 251A and EP 420 317A (Unilever), and in our copending European Patent Application No. 91 200 740.8.

The granulate obtained from the mixer/granulator may be used as a complete detergent composition in its own right. Alternatively, it may be admixed with other components or mixtures prepared separately, and may form a major or minor part of a final product.

Other ingredients

Detergent compositions in accordance with the invention may if desired or appropriate contain other functional ingredients. Some of these, for example, sodium silicate, inorganic salts such as sodium sulphate, and fluorescers, will be capable of withstanding the granulation/densification process and any steps that precede it, while others, for example, bleach ingredients, lather control agents, enzymes and perfume, are more suitably added afterwards. The skilled detergents worker will have no difficulty in judging when and how the various ingredients that go to make up a fully formulated detergent composition should be incorporated.

EXAMPLES

The following Examples illustrate the invention. Examples identified by numbers are within the invention, while those identified by letters are comparative. Parts and percentages are by weight unless otherwise stated.

Examples 1 to 5, Comparative Examples A to E

Detergent powders were prepared to the formulations shown in Tables 1 and 2. The PAS used was Empicol

(Trade Mark) LX, a narrow-cut (C_{12}/C_{14} enriched) cocoPAS (Na salt) supplied by Albright & Wilson.

Examples 1, 2, A, B and C in Table 1 were high-carbonate compositions, and Examples D, 3, 4, 5 and E in Table 2 were zero-carbonate formulations.

The comparative all-LAS powders C and E were prepared by spray-drying an aqueous slurry of all ingredients except sodium carbonate, sodium sulphate and enzyme, to form a common base powder; densifying the base powder using the Fukae FS-100 high-speed mixer/granulator, as described in EP 340 013A (Unilever); then admixing the relevant inorganic salt (sodium carbonate or sodium sulphate) and enzyme.

The all-PAS powders 1 and D were prepared by dry-mixing the PAS (in powder form) with all other ingredients except sodium carbonate, sodium sulphate and enzyme to form a base, in the Fukae mixer; densifying; then admixing the relevant inorganic salt (sodium carbonate or sodium sulphate) and enzyme.

The LAS/PAS powders (Examples 2, A, B, 3, 4 and 5) were prepared by admixing the appropriate quantities of the LAS base powder of Examples C and E and the PAS base powder of Examples 1 and D, then proceeding as in the other Examples.

The final powders had bulk densities above 720 g/litre. They were free-flowing and showed no tendency to cake.

Detergency measurements were carried out using two different test cloths; Test Cloth 1 carried a mixture of fatty and particulate soil, and Test Cloth 2 a mixture of fatty soil, particulate soil and casein. The powders were dosed at 0.96 g/litre into 35 litres of water (5° French hard in Ca^{2+}, 2° French hard in Mg^{2+}) in a Japanese (National (Trade Mark) Electronic W100) washing machine; the test cloths were washed together with a 2.0 kg soiled cotton load, the wash time being 8 minutes and the rinse time also 8 minutes (running rinse).

Reflectance data at 460 nm of the washed cloths were measured using a Micromatch (Trade Mark) reflectometer. The results are also shown in Tables 1 and 2.

Comparison of Examples 1 and D shows that when cocoPAS was present and LAS absent, detergency was significantly better in the presence of 15.51 wt% sodium carbonate than in the absence of sodium carbonate.

At 75 wt% PAS/25 wt% LAS (Examples 2 and 3), good detergency was obtained at both high and zero carbonate levels.

At 50 wt% PAS/50 wt% LAS (Examples A and 4) and at 25 wt% PAS/75 wt% LAS (Examples B and 5), detergency was significantly better in the absence of carbonate.

When LAS was present and PAS absent (Examples C and E), the level of carbonate had no effect on detergency.

Comparison of Examples 3, 4 and 5 with Examples D and E also shows a synergistic benefit: in the absence of sodium carbonate, the detergency obtained from the PAS/LAS mixture was better than that obtained from either surfactant used alone.

TABLE 1: EXAMPLES 1 & 2, COMPARATIVE EXAMPLES A - C

	1	2	A	B	C
CocoPAS	25.0	18.75	12.50	6.25	-
Linear alkylbenzene sulphonate	-	6.25	12.50	18.75	25.00
Nonionic surfactant	1.5	1.5	1.5	1.5	1.5
Soap	1.0	1.0	1.0	1.0	1.0
Zeolite (anhydr.) (	35.0	35.0	35.0	35.0	35.0
Water with zeolite (	9.99	9.99	9.99	9.99	9.99
Na silicate	4.0	4.0	4.0	4.0	4.0
Acrylic/maleic copolymer	1.5	1.5	1.5	1.5	1.5
Fluorescer	0.18	0.18	0.18	0.18	0.18
SCMC	0.9	0.9	0.9	0.9	0.9
Na carbonate	15.51	15.51	15.51	15.51	15.51
Na sulphate	-	-	-	-	-
Enzyme (alcalase)	0.6	0.6	0.6	0.6	0.6
Salts, water to 100 wt%					
Reflectance increases (δR_{460}*)					
Test cloth 1	20.5	20.3	16.7	16.4	15.2
Test cloth 2	20.0	19.8	17.0	16.7	15.0

EP 0 460 925 A2

TABLE 2: EXAMPLES 3 TO 5, COMPARATIVE EXAMPLES D AND E

	D	3	4	5	E
CocoPAS	25.0	18.75	12.50	6.25	-
Linear alkylbenzene sulphonate	-	6.25	12.50	18.75	25.00
Nonionic surfactant	1.5	1.5	1.5	1.5	1.5
Soap	1.0	1.0	1.0	1.0	1.0
Zeolite (anhydr.) (	35.0	35.0	35.0	35.0	35.0
Water with zeolite (	9.99	9.99	9.99	9.99	9.99
Na silicate	4.0	4.0	4.0	4.0	4.0
Acrylic/maleic copolymer	1.5	1.5	1.5	1.5	1.5
Fluorescer	0.18	0.18	0.18	0.18	0.18
SCMC	0.9	0.9	0.9	0.9	0.9
Na carbonate	-	-	-	-	-
Na sulphate	15.51	15.51	15.51	15.51	15.51
Enzyme (alcalase)	0.6	0.6	0.6	0.6	0.6
Salts, water to 100 wt%					
Reflectance increases (δR_{460}*)					
Test cloth 1	17.8	20.3	18.8	19.1	15.1
Test cloth 2	17.4	21.5	19.0	19.5	15.4

EP 0 460 925 A2

Claims

1 A particulate detergent composition comprising anionic surfactant, alkali metal aluminosilicate builder and optional sodium carbonate, and optionally other detergent ingredients, characterised in that it comprises:

(a) from 17 to 35 wt% of non-soap detergent-active material consisting essentially of:

(i) from 5 to 35 wt% of an anionic surfactant component consisting of primary alcohol sulphate [10-100 wt% of (i)] optionally together with alkylbenzene sulphonate [0-90 wt% of (i)],

(ii) optionally from 0 to 10 wt% of nonionic surfactant,

(iii) optionally from 0 to 10 wt% of further anionic surfactant other than primary alcohol sulphate or alkylbenzene sulphonate,

(b) optionally from 0 to 10 wt% of fatty acid soap,

(c) from 25 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate,

(d) from 0 to 10 wt% of sodium carbonate if the anionic surfactant component (a)(i) contains 10-60 wt% of primary alcohol sulphate, from 0 to 20 wt% of sodium carbonate if the anionic surfactant component (a)(i) contains 60-80 wt% primary alcohol sulphate, and from 10 to 20 wt% of sodium carbonate if the anionic surfactant component (a)(i) contains 80-100 wt% primary alcohol sulphate,

(e) optionally other detergent ingredients to 100 wt%.

2 A detergent composition as claimed in claim 1, characterised in that the anionic surfactant component (a)(i) comprises from 20 to 100 wt% of primary alcohol sulphate and from 0 to 80 wt% of linear alkylbenzene sulphonate.

3 A detergent composition as claimed in claim 2, characterised in that the anionic surfactant component (a)(i) comprises from 60 to 80 wt% of primary alcohol sulphate and from 20 to 40 wt% of linear alkylbenzene sulphonate, and the composition contains from 0 to 10 wt% of sodium carbonate.

4 A detergent composition as claimed in claim 3, characterised in that the anionic surfactant component (a)(i) comprises from 60 to 75 wt% of primary alcohol sulphate and from 25 to 40 wt% of linear alkylbenzene sulphonate, and the composition contains from 0 to 10 wt% of sodium carbonate.

5 A detergent composition as claimed in claim 2, characterised in that the anionic surfactant component (a)(i) comprises from 20 to 50 wt% of primary alcohol sulphate and from 50 to 80 wt% of linear alkylbenzene sulphonate, and the composition contains from 0 to 10 wt% of sodium carbonate.

6 A detergent composition as claimed in claim 5, characterised in that the anionic surfactant component (a)(i) comprises from 30 to 40 wt% of primary alcohol sulphate and from 60 to 70 wt% of linear alkylbenzene sulphonate, and the composition contains from 0 to 10 wt% of sodium carbonate.

7 A detergent composition as claimed in any preceding claim, characterised in that the primary alcohol sulphate consists wholly or predominantly of material having an alkyl chain length of C_{16} or below.

8 A detergent composition as claimed in claim 7, characterised in that the primary alcohol sulphate consists wholly or predominantly of material having an alkyl chain length of C_{14} or below.

9 A detergent composition as claimed in claim 7 or claim 8, characterised in that the primary alcohol sulphate comprises coconut alcohol sulphate.

10 A detergent composition as claimed in any preceding claim, characterised in that it contains from 28 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate.

11 A detergent composition as claimed in any preceding claim, characterised in that the weight ratio of aluminosilicate (c) to non-soap detergent-active material (a) is from 0.9:1 to 2.6:1.

12 A detergent composition as claimed in any preceding claim, having a bulk density of at least 650 g/litre.