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(54) **Detergent compositions**

Reinigungsmittelzusammensetzungen

Compositions détergentes

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## Description

### TECHNICAL FIELD

5 [0001] 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 free from sodium carbonate.

### BACKGROUND AND PRIOR ART

10 [0002] 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).

[0003] JP 01 142 000A (Nippon Gosen Senzai) discloses a high bulk density detergent composition prepared by neutralising PAS acid with sodium carbonate and zeolite.

15 [0004] EP 342 917A (Unilever) discloses detergent compositions containing PAS having a specific chain length distribution.

[0005] EP 439 316A (Unilever) discloses detergent compositions containing zeolite, sodium carbonate, and branched-chain PAS.

20 [0006] 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.

25 [0007] JP 62 240 397A (Lion) discloses detergent compositions containing alkylbenzene sulphonate (3-30 wt%) and specified primary alkyl sulphate (1-30 wt%) in a ratio of 10:1 -1:2, and zeolite builder (5-30 wt%). Sodium carbonate may optionally be present.

[0008] 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

### 30 DEFINITION OF THE INVENTION

[0009] The present invention accordingly provides a particulate detergent composition defined by claim 1.

### DETAILED DESCRIPTION OF THE INVENTION

35 [0010] The subject of the invention is a detergent composition in particulate form characterised by a moderate to high content of anionic surfactant, including primary alcohol sulphate of the kind defined hereinbelow; a relatively high level of aluminosilicate builder and zero sodium carbonate.

40 [0011] 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.

[0012] 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

45 [0013] 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 surfactant component, constitutes from 5 to 35 wt% of the whole composition.

#### 50 The primary alcohol sulphate

55 [0014] An essential ingredient of the surfactant system is a coconut primary alcohol sulphate. PAS constitutes from 25 to 75 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. This essential coconut alcohol PAS (cocoPAS) is a "narrow-cut" material enriched in C<sub>12</sub> and C<sub>14</sub> alcohols by fractionation. CocoPAS exhibits good low-temperature detergency, is of renewable natural origin, and is biodegradable.

The linear alkylbenzene sulphonate

**[0015]** As previously indicated, the main anionic surfactant component, which constitutes 5 to 35 wt% of the composition, consists of the linear alkylbenzene sulphonate (LAS) and the PAS.

**[0016]** Linear alkylbenzene sulphonates are exceedingly well-known ingredients of fabric washing detergent compositions. The alkyl chain length is generally in the C<sub>8</sub>-C<sub>16</sub> 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

**[0017]** 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<sub>12</sub>-C<sub>15</sub> 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

**[0018]** The composition may alternatively or additionally contain up to 10 wt% of one or more anionic surfactants other than the LAS and PAS. Examples of suitable anionic surfactants are alkyl ether sulphates, alkyl xylene sulphonates, olefin sulphonates, dialkyl sulphosuccinates and fatty acid ester sulphonates.

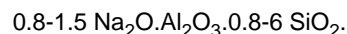
**[0019]** These lists are not intended to be exhaustive and for further examples the reader is referred to the standard literature, for example, "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

Optional soap

**[0020]** 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

**[0021]** 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



**[0022]** 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<sub>2</sub> units (in the formula above) and have a particle size of not more than 100 microns, preferably not more than 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.

**[0023]** Crystalline aluminosilicates (zeolites) are preferred for use in the present invention. Suitable materials are described, for example, In GB-A-1 473 201 (Henkel) and GB-A-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.

**[0024]** Also of interest is the novel zeolite - maximum aluminium zeolite P, described and claimed in EP 384 070A (Unilever).

**[0025]** 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

**[0026]** 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 tri- succinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates. Alternatively, organic precipitant builders such as alkyl- and alkanylmalonates and succinates, and sulphonated

fatty acid salts, may be used.

**[0027]** 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 1 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%.

**[0028]** 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.

#### High bulk density

**[0029]** 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.

**[0030]** 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.

**[0031]** 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.

**[0032]** 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.

**[0033]** A preferred type of batch high-speed mixer/granulator is bowl-shaped and preferably has a substantial 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.

**[0034]** The Fukae mixer may also be used to produce compositions of the invention directly from raw materials by high-speed mixing and granulation. The 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 the PAS or LAS or both are prepared by neutralisation in situ, for example, as described and claimed in EP 352 135A (Unilever) and EP 438 320A (Unilever).

**[0035]** 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-lower and non-lower processes, including in-situ neutralisation. Suitable processes are disclosed in EP 367 339A, EP 390 251A and EP 420 317A (Unilever).

**[0036]** 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

**[0037]** 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 fluorescenters, 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 detergent 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

**[0038]** 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 3, Comparative Examples A to G

**[0039]** 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.

**[0040]** Examples A to E in Table 1 were high-carbonate compositions, and Examples F, 1, 2, 3 and G in Table 2 were zero-carbonate formulations.

**[0041]** The comparative all-LAS powders E and G 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.

**[0042]** The all-PAS powders A and F 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.

**[0043]** The LAS/PAS powders B, C, D, 1, 2 and 3 were prepared by admixing the appropriate quantities of the LAS base powder of Examples E and G and the PAS base powder of Examples A and F, then proceeding as in the other Examples.

**[0044]** The final powders had bulk densities above 720 g/litre. They were free-flowing and showed no tendency to cake.

**[0045]** 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 ( $5^\circ$  French hard in  $Ca^{2+}$ ,  $2^\circ$  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).

**[0046]** 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.

**[0047]** Comparison of Examples A and F 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.

**[0048]** At 75 wt% PAS/25 wt% LAS (Examples B and 1), good detergency was obtained at both high and zero carbonate levels.

**[0049]** At 50 wt% PAS/50 wt% LAS (Examples C and 2) and at 25 wt% PAS/75 wt% LAS (Examples D and 3), detergency was significantly better in the absence of carbonate.

**[0050]** When LAS was present and PAS absent (Examples E and G), the level of carbonate had no effect on detergency.

**[0051]** Comparison of Examples 1, 2 and 3 with Examples F and G 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: COMPARATIVE EXAMPLES A - E

	A	B	C	D	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	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 ( $\delta R_{460}^*$ )					
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

TABLE 2: EXAMPLES 1 TO 3 COMPARATIVE EXAMPLES F AND G

		F	1	2	3	G
5	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
10	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
15	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%					
20	<u>Reflectance increases (<math>\delta R_{460}^*</math>)</u>					
	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

## Claims

1. A particulate detergent composition having a bulk density of at least 650 g/litre comprising anionic surfactant, alkali metal aluminosilicate builder and optional sodium carbonate, and optional other detergent ingredients, **characterised in that** It comprises:

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

- (i) from 5 to 35 wt% of an anionic surfactant component consisting of from 25 to 75 wt% of coconut primary alcohol sulphate which is a narrow-cut material enriched in C<sub>12</sub> and C<sub>14</sub> alcohols by fractionation and from 25 to 75 wt% of linear alkylbenzene sulphonate,  
(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) the composition being free of sodium carbonate,  
(e) optionally other detergent ingredients to 100 wt%.

2. A detergent composition as claimed in claim 1, **characterised in that** it contains from 28 to 15 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate.

3. A detergent composition as claimed in either 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.

## Patentansprüche

1. Teilchenförmige Waschmittelzusammensetzung mit einer Schüttdichte von zumindest 650 g/l umfassend ein anionisches Tensid, Alkalialuminosilicatgerüststoff und gegebenenfalls Natriumcarbonat und gegebenenfalls andere Waschmittelinhaltsstoffe, **dadurch gekennzeichnet, dass** sie:

(a) 17 bis 35 Gew.-% eines waschaktiven Nichtseifen-Materials bestehend aus:

- (i) 5 bis 35 Gew.-% einer anionischen Tensidkomponente, die aus 25 bis 75 Gew.-% primärem Kokosalkoholsulfat, das ein Material "engen Zuschnitts" ist, das durch Fraktionierung an C<sub>12</sub>- und C<sub>14</sub>-Alkoholen angereichert ist, und 25 bis 75 Gew.-% linearem Alkylbenzolsulfonat besteht,  
 (ii) gegebenenfalls 0 bis 10 Gew.-% nichtionischem Tensid,  
 (iii) gegebenenfalls 0 bis 10 Gew.-% eines anderen anionischen Tensids außer primärem Alkoholsulfat oder Alkylbenzolsulfonat,

- (b) gegebenenfalls 0 bis 10 Gew.-% Fettsäureseife,  
 (c) 25 bis 45 Gew.-% (wasserfreie Basis) kristallines oder amorphes Alkalialuminosilicat,  
 (d) wobei die Zusammensetzung frei von Natriumcarbonat ist,  
 (e) gegebenenfalls andere Waschmittelinhaltsstoffe auf 100 Gew.-% umfasst.

2. Waschmittelzusammensetzung nach Anspruch 1, **dadurch gekennzeichnet, dass** sie 28 bis 45 Gew.-% (wasserfreie Basis) kristallines oder amorphes Alkalialuminosilicat enthält.

3. Waschmittelzusammensetzung nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** das Gewichtsverhältnis von Aluminosilicat (c) zu waschaktivem Nichtseifenmaterial (a) 0,9:1 bis 2,6:1 ist.

## Revendications

1. Composition détergente particulière ayant une densité apparente d'environ 650 g/litre, comprenant un tensioactif anionique, un adjuvant aluminosilicate de métal alcalin et un carbonate de sodium facultatif, et facultativement d'autres ingrédients détergents, **caractérisée en ce qu'elle comprend** :

(a) de 17 à 35% en poids d'une matière active détergente non savonneuse constituée de

- (i) 5 à 35% en poids d'un composant tensioactif anionique constitué de 25 à 75% en poids de sulfate d'alcool primaire de coprah qui est une matière à fraction étroite enrichie en alcools en C<sub>12</sub> à C<sub>14</sub> par fractionnement, et de 25 à 75% en poids d'alkylbenzène-sulfonate linéaire,  
 (ii) facultativement, 0 à 10% en poids d'un tensioactif non ionique,  
 (iii) facultativement, 0 à 10% en poids d'un tensioactif anionique supplémentaire autre que le sulfate d'alcool primaire ou l'alkylbenzène-sulfonate,

- (b) facultativement, de 0 à 10% en poids d'un savon d'acide gras,  
 (c) de 25 à 45% en poids (base anhydre) d'aluminosilicate de métal alcalin cristallin ou amorphe,  
 (d) la composition étant exempte de carbonate de sodium  
 (e) facultativement, d'autres ingrédients détergents jusqu'à 100% en poids.

2. Composition détergente selon la revendication 1, **caractérisée en ce qu'elle** contient de 28 à 45% en poids (base anhydre) d'aluminosilicate de métal alcalin cristallin ou amorphe.

3. Composition détergente selon l'une ou l'autre des revendications précédentes, **caractérisée en ce que** le rapport pondéral de l'aluminosilicate (c) à la matière détergente active non savonneuse (a) est de 0,9 : 1 à 2,6 : 1.