(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 774 505 B1

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
21.11.2001 Bulletin 2001/47

(51) Int Cl.[7]: **C11D 3/08**, C11D 3/12, C11D 17/06

(21) Application number: **96306111.4**

(22) Date of filing: **21.08.1996**

(54) **Detergent compositions**

Waschmittelzusammensetzungen

Compositions détergentes

(84) Designated Contracting States:
DE ES FR GB IT

(30) Priority: **17.11.1995 GB 9523571**

(43) Date of publication of application:
21.05.1997 Bulletin 1997/21

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Description

TECHNICAL AREA

[0001] The present invention relates to granular laundry detergent compositions of high bulk density containing zeolite builders.

BACKGROUND AND PRIOR ART

[0002] Laundry detergent compositions containing zeolite builders have been widely disclosed in the art.

[0003] The use of soluble silicates in granular laundry detergent compositions to reduce corrosion of metal washing machine parts is well known in the art. However, in granular compositions containing zeolite, especially dense granular compositions prepared by non-spray-drying processes, the presence of sodium silicate can cause dissolution and solubility problems and lead to the deposit of insoluble residues on washed fabrics.

[0004] The present invention is based on the discovery that sodium silicate may be incorporated in high bulk density zeolite-built granular detergent compositions at an optimum level which minimises corrosion of aluminium and enamel while having no adverse effect on dissolution or solubility.

[0005] EP 552 054A (Unilever) discloses a high bulk density zeolite-built bleaching detergent powder containing 4 wt% of sodium disilicate.

DEFINITION OF THE INVENTION

[0006] The invention accordingly provides the use of water-soluble sodium silicate in an amount of from 2.5 to 6.5 wt% in a granular laundry detergent composition having a bulk density of at least 650 g/litre and comprising one or more detergent surfactants and a builder system comprising from 10% to 70% (anhydrous basis), preferably from 25% to 50% by weight of the total composition of zeolite, to minimise aluminium and enamel corrosion without an adverse effect on solubility.

DETAILED DESCRIPTION OF THE INVENTION

[0007] Preferred granular detergent compositions to which the present invention applies comprise:

(a) a non-spray dried base powder having a bulk density of at least 650 g/litre comprising one or more detergent surfactants and a detergency builder system comprising zeolite, and

(b) from 2.5 to 6.5 wt% of soluble sodium silicate in the form of separate granules.

The soluble sodium silicate

[0008] According to a first preferred embodiment of the invention the separate granules of sodium silicate are in the form of granular sodium disilicate.

[0009] According to a second preferred embodiment of the invention the sodium silicate is in the form of sodium carbonate/sodium silicate cogranules.

[0010] The amount of soluble sodium silicate in the detergent compositions with which the invention is concerned is from 2.5 to 6.5 wt%, preferably from 3 to 6 wt% and most preferably from 3 to 4 wt%. In this range corrosion of both enamel and aluminium is minimised without detriment to solubility properties.

[0011] It has been observed that low levels of sodium silicate, for example, 2 wt%, can actually increase both enamel and aluminium corrosion, as compared with a formulation containing no sodium silicate. At 4 wt% sodium silicate enamel corrosion was reduced, and the optimum level of sodium silicate for preventing enamel corrosion was found to be about 6 wt%. Aluminium corrosion was minimised at levels of 4 wt% sodium silicate and above. Levels of sodium silicate above 6.5 wt%, while continuing to provide good protection against both enamel and aluminium corrosion, were found to be detrimental to both dispensing into the washing machine and solubility, and led to higher residues being left on the washload.

Other components

[0012] The detergent compositions with which the invention is concerned contain as essential ingredients detergent surfactants and detergency builders, the latter comprising zeolite (sodium aluminosilicate). Surfactants and builders

are preferably wholly or predominantly within a non-spray-dried base powder which may suitably constitute from 35 to 97.5 wt%, preferably from 40 to 80 wt% and most preferably from 50 to 75 wt%, of the total product.

[0013] Detergent surfactants are suitably present in an amount of from 5 to 60 wt% based on the whole composition. The surfactants may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

[0014] The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds.

[0015] Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; primary and secondary alkylsulphates, particularly C_8-C_{15} primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

[0016] Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C_8-C_{20} aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C_{10}-C_{15} primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

[0017] The detergent compositions also contain one or more detergency builders. The total amount of detergency builder in the compositions will suitably range from 5 to 80 wt%, preferably from 10 to 60 wt%.

[0018] The builder system includes a crystalline sodium aluminosilicate (zeolite). Sodium aluminosilicates are incorporated in amounts of from 10 to 70% by weight (anhydrous basis), preferably from 25 to 50 wt%.

[0019] The alkali metal aluminosilicate has the general formula:

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

[0020] These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above). These materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

[0021] Suitable crystalline sodium aluminosilicate ionexchange detergency builders are described, for example, in 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.

[0022] The zeolite may be the commercially available zeolite 4A now widely used in laundry detergent powders. However, according to a preferred embodiment of the invention, the zeolite builder is maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070A (Unilever). Zeolite MAP is defined as an alkali metal aluminosilicate of the zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, and more preferably within the range of from 0.90 to 1.20.

[0023] Especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00. The calcium binding capacity of zeolite MAP is generally at least 150 mg CaO per g of anhydrous material.

[0024] Advantageously a supplementary builder may be present. Especially preferred organic builders are citrates, suitably used in amounts of from 5 to 50 wt%, preferably from 10 to 35 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%.

[0025] Preferred compositions contain sodium citrate. Citrate may be present in the base powder, or as separate granules, or both.

[0026] The detergent compositions according to the invention may also suitably contain a bleach system, preferably comprising a peroxy bleach compound, for example, an inorganic persalt or organic peroxyacid, capable of yielding hydrogen peroxide in aqueous solution. Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate is especially preferred.

[0027] The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

[0028] The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%.

[0029] Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid precursors. An especially preferred bleach precursor suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED).

[0030] A bleach stabiliser (heavy metal sequestrant) may also be present. Suitable bleach stabilisers include ethylenediamine tetraacetate (EDTA) and the polyphosphonates such as Dequest (Trade Mark), EDTMP.

[0031] The detergent compositions may also suitably contain detergency enzymes, for example, proteases, lipases, cellulases and amylases. Enzymes are commonly employed in granular form in amounts of from about 0.1 to about 3.0 wt%.

[0032] The detergent compositions may contain alkali metal, preferably sodium, carbonate, in order to increase detergency and ease processing. Sodium carbonate may suitably be present in amounts ranging from 1 to 60 wt%, preferably from 2 to 40 wt%. However, compositions containing little or no sodium carbonate are also within the scope of the invention.

[0033] Powder flow may be improved by the incorporation in the base powder of a small amount of a powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, or an acrylate or acrylate/maleate polymer. One preferred powder structurant is fatty acid soap, suitably present in an amount of from 1 to 5 wt%.

[0034] Other materials that may be present in the detergent compositions according to the invention include soil release polymers; antiredeposition agents such as cellulosic polymers; fluorescers; inorganic salts such as sodium sulphate; lather control agents or lather boosters as appropriate; dyes; coloured speckles; perfumes; foam controllers; and fabric softening compounds.

[0035] Detergent compositions according to the invention may be prepared by any method suitable for the preparation of granular high bulk density compositions. Mixing and grnaulation processes using a high-speed mixer/granulator are preferably used. Suitable processes are disclosed, for example, in EP 340 013A, EP 367 339A, EP 390 251A and EP 420 317A (Unilever).

[0036] The invention is further illustrated by the following Examples, in which parts and percentages are by weight unless otherwise stated.

EXAMPLES 1 to 3, COMPARATIVE EXAMPLES A to D

[0037] High bulk density granular detergent compositions were prepared to the following general formulation. The base powder was prepared by mixing and granulation in a Lödige high-speed mixer/granulator, and other ingredients were postdosed as shown.

Base powder	
Coconut primary alcohol sulphate	12.00
Nonionic 7EO	6.00
Sodium soap	1.79
Zeolite MAP	23.94
Sodium citrate	3.52
Sodium carbonate	2.38
Sodium carboxymethylcellulose (as received)	0.72
Moisture and salts	5.10
Total base	55.45

Postdosed ingredients		
Sodium carbonate	)	
Sodium bicarbonate	)	
Granular sodium disilicate	)	total 26.38
Sodium carbonate/silicate cogranule*	)	
Soil release polymer granule		7.50
Antifoam/polyvinylpyrrolidone granule		4.00
EDTMP**		1.40
Acrylic/maleic copolymer granule		3.00
Enzyme granules		4.82
Perfume		0.45

*Nabion (Trade Mark) 15 ex Rhône-Poulenc.

**Ethylenediaminetetramethylenephosphonate, Dequest (Trdae Mark) 2047 ex Monsanto.

[0038] As indicated above, sodium carbonate, sodium bicarbonate, granular sodium disilicate and the carbonate/silicate cogranule were postdosed in various amounts, to a total of 26.38 wt% to give Examples 1, 2, and 3 within the invention and Comparative Examples A, B, C and D, as follows:

	A	B	1	2
Carbonate	18.38	16.38	14.38	12.38
Bicarbonate	8.00	8.00	8.00	8.00
Disilicate	-	2.00	4.00	6.00

	A	3	C	D
Carbonate	18.38	-	-	-
Bicarbonate	8.00	8.00	4.00	-
Cogranule	-	18.38	22.38	26.38
Silicate content	-	6.0	7.3	8.6

[0039] Enamel corrosion, aluminium corrosion and insoluble residues in the wash were determined as follows.

Measurement of enamel corrosion

[0040] Enamel corrosion was measured using the Bayer Lantern test in accordance with DIN 51154. The Bayer Lantern is a closed thermostated vessel with a number of "windows" to which enamel test pieces are clamped so as to remain in contact with detergent solution with which the vessel is filled. Solutions (10 g/l) of each formulation in demineralised water were stirred in the vessel at 95°C for 24 hours. The enamel test plates were weighed before and after testing and the weight losses recorded.

Measurement of aluminium corrosion

[0041] Aluminium corrosion was measured as follows. Standard aluminium discs (composition Al >99%, Si 0.25%,

Fe 0.4%, Cu 0.05%, Mn 0.05%, Mg 0.05%, Zn 0.07%) of radiums 2.5 cm were suspended in a 1-litre beaker containing a 10 g/l solution in demineralised water of each formulation for three intervals each of 7 hours. The solutions were stirred using a 6 cm magnetic stirrer bar without forming a vortex. The aluminium test plates (discs) were weighted after three 7-hour test intervals and the total weight loss recorded.

Measurement of insoluble residues in the wash

[0042] A washing machine test was used to determine the extent that insoluble residues were deposited on washed articles. The machine used was a Siemens Siwamat (Trade Mark) 3803 front-loading automatic washer, the load was 1 kg of clean cotton, and the test methodology was as follows.

[0043] 10 g doses of the powders under test were placed inside black cotton sachets having dimensions of 10 cm by 10 cm which were then closed. The sachets were then attached with product to the washload (a maximum of ten sachets per load).

[0044] The machine was operated on the wool wash cycle at a wash temperature of 40°C, using water of 15° French hardness and a water inlet temperature of 20°C. At the end of the wash cycle the sachets were removed, line dried flat, opened and turned inside out, and the level of powder residue on the inside surface of each sachet determined by visual assessment using a scoring system of 1 to 3:
a score of 3 corresponds to a residue of approximately 75 wt% of the powder, while 1 indicates no residue. A panel of five assessors was used to judge each sachet and allot a score. With each test product the wash process was carried out six times and the scores were averaged over the six runs.

Results of Examples 1 to 3 and Comparative Examples A to D

[0045]

Enamel corrosion		
Example		Weight loss mg/cm^2
A	(no silicate)	0.85
B	(2% disilicate)	0.99
1	(4% disilicate)	0.45
2	(6% disilicate)	0.06
3	(6.0% silicate as cogranule)	0.06
C	(7.3% silicate as cogranule)	0.27
D	(8.6% silicate as cogranule)	0.82

Aluminium corrosion		
Example		Weight loss mg/cm^2
A	(no silicate)	16.58
B	(2% disilicate)	21.77
1	(4% disilicate)	0.50
2	(6% disilicate)	0.01
3	(6.0% silicate as cogranule)	0.00
C	(7.3% silicate as cogranule)	0.01
D	(8.6% silicate as cogranule)	0.01

Insoluble residues - black sachet test		
Example		Residues score
A	(no silicate)	1.8
B	(2% disilicate)	1.8
1	(4% disilicate)	1.7

(continued)

Insoluble residues - black sachet test		
Example		Residues score
2	(6% disilicate)	1.9
3	(6.0% silicate as cogranule)	1.9
C	(7.3% silicate as cogranule)	2.4
D	(8.6% silicate as cogranule)	2.3

[0046] It will be seen that the powders of Examples 1 to 3 gave good results in all three tests.

Claims

1. Use of water-soluble sodium silicate in an amount of from 2.5 to 6.5 wt% in a granular laundry detergent composition having a bulk density of at least 650 g/litre and comprising one or more detergent surfactants and a builder system comprising from 10% to 70% (anhydrous basis), preferably from 25% to 50% by weight of the total composition of zeolite, to minimise aluminium and enamel corrosion without an adverse effect on solubility.

2. Use as claimed in claim 1, wherein the granular detergent composition comprises:

   (a) a non-spray-dried base powder having a bulk density of at least 650 g/litre comprising one or more detergent surfactants and a detergency builder system comprising zeolite, and

   (b) from 2.5 to 6.5 wt% of soluble sodium silicate in the form of separate granules.

3. Use as claimed in claim 2, wherein the soluble silicate is in the form of granular sodium disilicate.

4. Use as claimed in claim 2, wherein the soluble silicate is in the form of a sodium carbonate/sodium silicate cogranule.

5. Use as claimed in any preceding claim, wherein the granular detergent composition comprises from 3 to 6 wt% of soluble sodium silicate.

6. Use as claimed in any preceding claim, wherein the granular detergent composition comprises from 3 to 4 wt% of soluble sodium silicate.

7. Use as claimed in any preceding claim, wherein the granular detergent composition further comprises sodium citrate.

8. Use as claimed in any preceding claim, wherein the granular detergent composition further comprises sodium percarbonate.

Patentansprüche

1. Verwendung eines wasserlöslichen Natriumsilicats in einer Menge von 2.5 bis 6,5 Gew.-% in einer körnigen Waschmittelzusammensetzung, die eine Schüttdichte von mindestens 650 g/Liter hat und eine oder mehrere waschaktive Substanzen und ein Buildersystem umfaßt, das 10 bis 70 Gew.-% (wasserfreie Basis), vorzugsweise 25 bis 50 Gew.-%, der gesamten Zusammensetzung an Zeolith umfaßt, wodurch die Korrosion von Aluminium und Emaille ohne nachteiligen Einfluß auf die Löslichkeit minimiert wird.

2. Verwendung nach Anspruch 1, wobei die körnige Waschmittelzusammensetzung umfaßt:

   (a) ein nicht durch Zerstäubungstrocknung erhaltenes Grundpulver mit einer Schüttdichte von mindestens 650 g/l, das eine oder mehrere waschaktive Substanzen und ein Buildersystem umfaßt, das einen Zeolith umfaßt,

und
(b) von 2,5 bis 6,5 Gew.-% eines löslichen Natriumsilicats in Form einzelner Körner.

3. Verwendung nach Anspruch 2, wobei das lösliche Silicat in Form von körnigem Natriumdisilicat vorliegt.

4. Verwendung nach Anspruch 2, wobei das lösliche Silicat in Form eines gemischten Natriumcarbonat/Natriumsilicat-Korns vorliegt.

5. Verwendung nach einem der vorstehenden Ansprüche, wobei die körnige Waschmittelzusammensetzung 3 bis 6 Gew.-% lösliches Natriumsilicat umfaßt.

6. Verwendung nach einem der vorstehenden Ansprüche, wobei die körnige Waschmittelzusammensetzung 3 bis 4 Gew.-% lösliches Natriumsilicat umfaßt.

7. Verwendung nach einem der vorstehenden Ansprüche, wobei die körnige Waschmittelzusammensetzung außerdem Natriumcitrat umfaßt.

8. Verwendung nach einem der vorstehenden Ansprüche, wobei die körnige Waschmittelzusammensetzung außerdem Natriumpercarbonat umfaßt.

Revendications

1. Utilisation de silicate de sodium soluble dans l'eau dans une quantité allant de 2,5 à 6,5 % en poids dans une composition détergente de lessive granulaire ayant une densité d'au moins 650 g/litre et comprenant un ou plusieurs tensioactifs détergents et un système édificateur constituant de 10 % à 70 % (base anhydre), de préférence de 25 % à 50 % en poids de la composition totale de zéolite afin de minimiser la corrosion de l'aluminium et de l'émail sans que cela ait d'effet nocif sur la solubilité.

2. Utilisation selon la revendication 1, dans laquelle la composition détergente granulaire comprend :

(a) une poudre de base non séchée par vaporisation, ayant une densité en masse d'au moins 650 grammes/litre comprenant un ou plusieurs tensioactifs détergents et un système d'édificateur de détergence comprenant de la zéolite, et

(b) de 2,5 à 6,5 % en poids de silicate de sodium soluble dans l'eau sous la forme de granules séparées.

3. Utilisation selon la revendication 2, dans laquelle le silicate soluble est sous la forme de disilicate de sodium granulaire.

4. Utilisation selon la revendication 2, dans laquelle le silicate de sodium est sous la forme d'une cogranule de carbonate de sodium/silicate de sodium.

5. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle la composition détergente granulaire comprend de 3 à 6 % en poids de silicate de sodium soluble.

6. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle la composition détergente granulaire comprend de 3 à 4 % en poids de silicate de sodium soluble.

7. Utilisation selon l'une quelconque des revendications précédentes dans laquelle la composition détergente granulaire comprend en outre du citrate de sodium.

8. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle la composition détergente granulaire comprend en outre du percarbonate de sodium.