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(54) Title: A PARTICULATE LAUNDRY DETERGENT COMPOSITION COMPRISING A DETERGENT SURFACTANT, CARBONATE AND A FLOURESCENT WHITENING COMPONENT

(57) Abstract: The present invention relates to a solid free-flowing particulate laundry detergent composition comprising a detergent surfactant, carbonate and a fluorescent whitening agent.



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**A PARTICULATE LAUNDRY DETERGENT COMPOSITION COMPRISING A
DETERSIVE SURFACTANT, CARBONATE AND A FLOURESCENT
WHITENING COMPONENT**

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FIELD OF THE INVENTION

The present invention relates to a highly water-soluble solid laundry detergent composition. More specifically, the present invention relates to a solid laundry detergent composition comprising a deterative surfactant, carbonate, a fluorescent whitening agent and low or no levels of zeolite builder and phosphate builder.

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BACKGROUND OF THE INVENTION

Laundry detergent compositions need to have a very good fabric-cleaning performance against a wide variety of soil types. Solid laundry detergents also need to have very good dispensing and dissolution profiles. However, a dichotomy may exist in that some reformulations of the solid laundry detergent composition to improve its fabric-cleaning performance may negatively impact its dispensing and dissolution profiles, and vice versa. It is very difficult to improve the cleaning performance, dispensing profile and dissolution profile of a solid laundry detergent composition at the same time. Furthermore, it is also desirable for highly water-soluble solid laundry detergent compositions to form a clear wash liquor upon dissolution in water. This is because having a clear wash liquor is a desired consumer signal that the solid laundry detergent composition has dissolved.

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Anionic deterative surfactants are incorporated into granular laundry detergent compositions in order to provide a good fabric-cleaning benefit. For example, GB1408969, GB1408970, US4487710, US5663136 and WO2004/041982 all relate to compositions comprising anionic deterative surfactants. However, the anionic deterative surfactant is capable of complexing with free cations, such as calcium and magnesium cations, that are present in the wash liquor in such a manner as to cause the anionic deterative surfactant to precipitate out of solution, which leads to a reduction in the anionic

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deterative surfactant activity. In extreme cases, these water-insoluble complexes may deposit onto the fabric resulting in poor whiteness maintenance and poor fabric integrity benefits. This is especially problematic when the laundry detergent composition is used in hard-water washing conditions when there is a high concentration of calcium cations.

5 The anionic deterative surfactant's tendency to complex with free cations in the wash liquor in such a manner as to precipitate out of solution is mitigated by the presence of builders, such as zeolite builders and phosphate builders, which have a high binding constant with cations such as calcium and magnesium cations. These builders sequester free calcium and magnesium cations and reduce the formation of these undesirable
10 complexes. However, zeolite builders are water-insoluble and their incorporation in laundry detergent compositions leads to poor dissolution of the laundry detergent composition and can also lead to undesirable residues being deposited on the fabric. In addition, detergent compositions that comprise high levels of zeolite builder form undesirable cloudy wash liquors upon contact with water. Whilst phosphate builders
15 allegedly do not have favourable environmental profiles and their use in laundry detergent compositions is becoming less common; for example, due to phosphate legislation in many countries.

Detergent compositions comprising alkyl benzene sulphonate and alkyl ethoxylated sulphate deterative surfactants are described in GB1408969, GB1408970, US4487710 and
20 US5663136. A detergent composition comprising an anionic deterative surfactant and a non-ionic deterative surfactant that allegedly gives enhanced stain removal at a wide range of water-hardness is described in WO2004/041982.

There remains a need for a solid free flowing particulate laundry detergent composition comprising a deterative surfactant having a good fabric-cleaning performance,
25 especially a good greasy stain cleaning performance, good whiteness maintenance, and very good dispensing and dissolution profiles, and which upon dissolution in water gives a clear wash liquor.

SUMMARY OF THE INVENTION

The Inventors have found that there is a tendency for low zeolite builder and low phosphate builder containing laundry detergent particles that comprise carbonate and a fluorescent whitening agent to have poor particle appearance characteristics; in particular there is a tendency for these laundry detergent particles to have an undesirable yellowish hue. Without wishing to be bound by theory, the Inventors believe that these poor particle characteristics, including the undesirable yellowish hue, are a result of the degradation of fluorescent whitening agent (e.g. by alkaline hydrolysis), which is caused by the presence of relatively high levels of carbonate in the particles. The Inventors have found that this problem can be overcome by controlling the levels and location of the carbonate and fluorescent whitening agent in the free flowing particulate laundry detergent composition.

The present invention provides a solid laundry detergent composition comprising: (a) a deterative surfactant; (b) from 0wt% to 10wt% zeolite builder; (c) from 0wt% to 10wt% phosphate builder; (d) carbonate; and (e) a fluorescent whitening agent; wherein the composition is in free-flowing particulate form and comprises at least two separate particulate components, wherein the first particulate component comprises: (i) a deterative surfactant; (ii) at least 10wt%, by weight of the first particulate component, of carbonate; (iii) from 0wt% to 10wt%, by weight of the first particulate component, of a zeolite builder; (iv) from 0wt% to 10wt%, by weight of the first particulate component, of a phosphate builder; and (v) from 0wt% to less than 5wt%, by weight of the first particulate component, of a fluorescent whitening agent; and wherein the second particulate component comprises: (i) at least 0.20wt%, by weight of the second particulate component, of a fluorescent whitening agent; and (ii) from 0wt% to less than 20wt%, by weight of the second particulate component, of carbonate.

DETAILED DESCRIPTION OF THE INVENTION

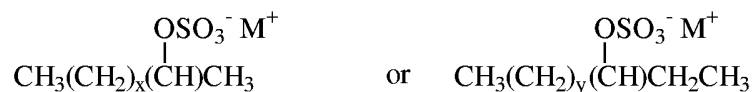
Deterative surfactant

The composition comprises a deterative surfactant. Suitable deterative surfactants include anionic deterative surfactants, nonionic deterative surfactants, cationic deterative surfactants, zwitterionic deterative surfactants, amphoteric deterative surfactants and mixtures thereof.

Suitable anionic deterative surfactants include: alkyl sulphates; alkyl sulphonates; alkyl phosphates; alkyl phosphonates; alkyl carboxylates; and mixtures thereof. The anionic surfactant can be selected from the group consisting of: C₁₀-C₁₈ alkyl benzene sulphonates (LAS) preferably C₁₀-C₁₃ alkyl benzene sulphonates; C₁₀-C₂₀ primary, 5 branched-chain, linear-chain and random-chain alkyl sulphates (AS), typically having the following formula:



10 wherein, M is hydrogen or a cation which provides charge neutrality, preferred cations are sodium and ammonium cations, wherein x is an integer of at least 7, preferably at least 9; C₁₀-C₁₈ secondary (2,3) alkyl sulphates, typically having the following formulae:



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wherein, M is hydrogen or a cation which provides charge neutrality, preferred cations include sodium and ammonium cations, wherein x is an integer of at least 7, preferably at least 9, y is an integer of at least 8, preferably at least 9; C₁₀-C₁₈ alkyl alkoxy carboxylates; mid-chain branched alkyl sulphates as described in more detail in US 20 6,020,303 and US 6,060,443; modified alkylbenzene sulphonate (MLAS) as described in more detail in WO 99/05243, WO 99/05242, WO 99/05244, WO 99/05082, WO 99/05084, WO 99/05241, WO 99/07656, WO 00/23549, and WO 00/23548; methyl ester sulphonate (MES); alpha-olefin sulphonate (AOS) and mixtures thereof.

Preferred anionic deterative surfactants include: linear or branched, substituted or 25 unsubstituted alkyl benzene sulphonate deterative surfactants, preferably linear C₈-C₁₈ alkyl benzene sulphonate deterative surfactants; linear or branched, substituted or unsubstituted alkyl benzene sulphate deterative surfactants; linear or branched, substituted or unsubstituted alkyl sulphate deterative surfactants, including linear C₈-C₁₈ alkyl sulphate deterative surfactants, C₁-C₃ alkyl branched C₈-C₁₈ alkyl sulphate deterative

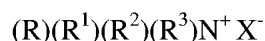
surfactants, linear or branched alkoxyated C₈-C₁₈ alkyl sulphate deterative surfactants and mixtures thereof; linear or branched, substituted or unsubstituted alkyl sulphonate deterative surfactants; and mixtures thereof.

Preferred alkoxyated alkyl sulphate deterative surfactants are linear or branched, substituted or unsubstituted C₈₋₁₈ alkyl alkoxyated sulphate deterative surfactants having an average degree of alkoxylation of from 1 to 30, preferably from 1 to 10. Preferably, the alkoxyated alkyl sulphate deterative surfactant is a linear or branched, substituted or unsubstituted C₈₋₁₈ alkyl ethoxyated sulphate having an average degree of ethoxylation of from 1 to 10. Most preferably, the alkoxyated alkyl sulphate deterative surfactant is a linear unsubstituted C₈₋₁₈ alkyl ethoxyated sulphate having an average degree of ethoxylation of from 3 to 7.

Preferred anionic deterative surfactants are selected from the group consisting of: linear or branched, substituted or unsubstituted, C₁₂₋₁₈ alkyl sulphates; linear or branched, substituted or unsubstituted, C₁₀₋₁₃ alkylbenzene sulphonates, preferably linear C₁₀₋₁₃ alkylbenzene sulphonates; and mixtures thereof. Highly preferred are linear C₁₀₋₁₃ alkylbenzene sulphonates. Highly preferred are linear C₁₀₋₁₃ alkylbenzene sulphonates that are obtainable, preferably obtained, by sulphonating commercially available linear alkyl benzenes (LAB); suitable LAB include low 2-phenyl LAB, such as those supplied by Sasol under the tradename Isochem[®] or those supplied by Petresa under the tradename Petrelab[®], other suitable LAB include high 2-phenyl LAB, such as those supplied by Sasol under the tradename Hyblene[®].

Suitable cationic deterative surfactants include: alkyl pyridinium compounds; alkyl quaternary ammonium compounds; alkyl quaternary phosphonium compounds; alkyl ternary sulphonium compounds; and mixtures thereof. The cationic deterative surfactant can be selected from the group consisting of: alkoxyate quaternary ammonium (AQA) surfactants as described in more detail in US 6,136,769; dimethyl hydroxyethyl quaternary ammonium as described in more detail in US 6,004,922; polyamine cationic surfactants as described in more detail in WO 98/35002, WO 98/35003, WO 98/35004, WO 98/35005, and WO 98/35006; cationic ester surfactants as described in more detail in US 4,228,042, US 4,239,660, US 4,260,529 and US 6,022,844; amino surfactants as described in more detail in US 6,221,825 and WO 00/47708, specifically amido propyldimethyl amine; and

mixtures thereof. Preferred cationic deterative surfactants are quaternary ammonium compounds having the general formula:



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wherein, R is a linear or branched, substituted or unsubstituted C₆₋₁₈ alkyl or alkenyl moiety, R¹ and R² are independently selected from methyl or ethyl moieties, R³ is a hydroxyl, hydroxymethyl or a hydroxyethyl moiety, X is an anion which provides charge neutrality, preferred anions include halides (such as chloride), sulphate and sulphonate.

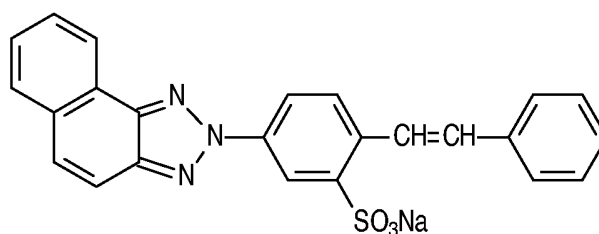
10 Preferred cationic deterative surfactants are mono-C₆₋₁₈ alkyl mono-hydroxyethyl di-methyl quaternary ammonium chlorides. Highly preferred cationic deterative surfactants are mono-C₈₋₁₀ alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride, mono-C₁₀₋₁₂ alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride and mono-C₁₀ alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride.

15 Suitable non-ionic deterative surfactant can be selected from the group consisting of: C₈-C₁₈ alkyl ethoxylates, such as, NEODOL[®] non-ionic surfactants from Shell; C₆-C₁₂ alkyl phenol alkoxylates wherein the alkoxylate units are ethyleneoxy units, propyleneoxy units or a mixture thereof; C₁₂-C₁₈ alcohol and C₆-C₁₂ alkyl phenol condensates with ethylene oxide/propylene oxide block polymers such as Pluronic[®] from BASF; C₁₄-C₂₂ 20 mid-chain branched alcohols, BA, as described in more detail in US 6,150,322; C₁₄-C₂₂ mid-chain branched alkyl alkoxylates, BAE_x, wherein x = from 1 to 30, as described in more detail in US 6,153,577, US 6,020,303 and US 6,093,856; alkylpolysaccharides as described in more detail in US 4,565,647, specifically alkylpolyglycosides as described in more detail in US 4,483,780 and US 4,483,779; polyhydroxy fatty acid amides as 25 described in more detail in US 5,332,528, WO 92/06162, WO 93/19146, WO 93/19038, and WO 94/09099; ether capped poly(oxyalkylated) alcohol surfactants as described in more detail in US 6,482,994 and WO 01/42408; and mixtures thereof.

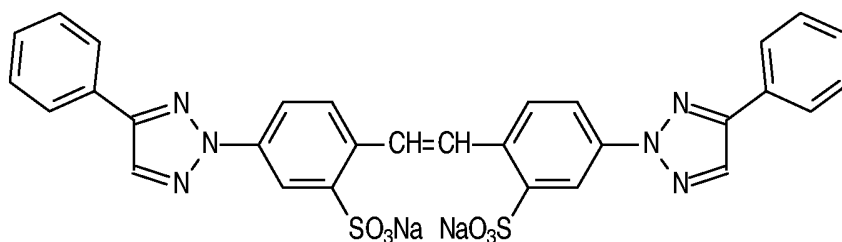
The non-ionic deterative surfactant could be an alkyl polyglucoside and/or an alkyl alkoxylated alcohol. Preferably the non-ionic deterative surfactant is a linear or branched, 30 substituted or unsubstituted C₈₋₁₈ alkyl ethoxylated alcohol having an average degree of ethoxylation of from 1 to 10, more preferably from 3 to 7.

Fluorescent whitening agent

The composition comprises a fluorescent whitening agent. Fluorescent whitening agents are typically capable of absorbing light in the ultraviolet wavelength band and re-emitting light in the visible band. Typically, light in the blue region of the visible wavelength band is re-emitted, giving recently treated fabrics a consumer preferred bluish hue. Suitable fluorescent whitening agents include substituted stilbenesulphonic acids or salts thereof that are substituted by at least one triazol-2-yl moiety. A suitable fluorescent whitening agent is a molecule having the general formula:



Another suitable fluorescent whitening agent is a molecule having the general formula:



Another suitable fluorescent whitening agent is sodium dibenzobiphenyldisulfonate. A suitable fluorescent whitening agent is disodium 4,4'-bis-(2-sulphostyryl)biphenyl. Other suitable fluorescent whitening agents are described in more detail by L. Ho Tan Tai, in Formulating Detergents and Personal Care Products: A complete guide to Product Development, AOCS Press, Champaign, 2000, p122-137.

Preferred fluorescent whitening agents comprise at least one, preferably at least two, triazine ring moieties. A preferred fluorescent whitening agent is disodium 4,4'-bis{[4-anilino-6-morpholino-s-triazin-2-yl]-amino}-2,2'-stilbenedisulphonate.

5 Carbonate

The composition comprises carbonate. It may be preferred for the composition to comprise from 1wt% to 50wt%, or from 5wt% to 25wt% or from 10wt% to 20wt%, by weight of the composition, of carbonate. A preferred carbonate is a carbonate salt, typically sodium carbonate and/or sodium bicarbonate. A highly preferred carbonate is
10 sodium carbonate. Preferably, the composition may comprise from 10wt% to 20wt% by weight of the composition, of sodium carbonate. However, it may also be preferred for the composition to comprise from 2wt% to 8wt% by weight of the composition, of sodium bicarbonate.

The carbonate, or at least part thereof, is typically in particulate form, typically
15 having a weight average particle size in the range of from 200 to 500 micrometers. However, it may be preferred for the carbonate, or at least part thereof, to be in micronised particulate form, typically having a weight average particle size in the range of from 4 to 40 micrometers; this is especially preferred when the carbonate, or at least part thereof, is in the form of a co-particulate admixture with a deterative surfactant, such as an
20 anionic deterative surfactant.

First particulate component

The first particulate component comprises: (i) a deterative surfactant; (ii) at least 10wt%, preferably at least 12wt%, or at least 15wt%, or even at least 20wt% by weight of
25 the first particulate component, of carbonate; (iii) from 0wt% to 10wt%, preferably from 0wt% to 8wt%, or from 0wt% to 6wt%, or from 0wt% to 4wt%, or from 0wt% to 2wt%, or from 0wt% to 1wt% by weight of the first particulate component, of a zeolite builder; (iv) from 0wt% to 10wt%, preferably from 0wt% to 8wt%, or from 0wt% to 6wt%, or from 0wt% to 4wt%, or from 0wt% to 2wt%, or from 0wt% to 1wt% by weight of the
30 first particulate component, of a phosphate builder; and (v) from 0wt% to less than 5wt%, preferably from 0wt% to 4wt%, or from 0wt% to 3wt%, or from 0wt% to 2wt%, or from

0wt% to 1wt%, or from 0wt% to 0.1wt% by weight of the first particulate component, of a fluorescent whitening agent. Typically, the first particulate component comprises one or more adjunct components; the remaining portion, if any, of the first particulate component is typically made up of adjunct components.

5 If the first particulate component comprises a fluorescent whitening agent, then preferably the weight ratio of carbonate to fluorescent whitening agent is at least 1:1, preferably at least 10:1, or at least 20:1, or at least 30:1, or at least 40:1, or at least 50:1, or at least 60:1, or at least 100:1, or at least 120:1, or at least 150:1, and preferably to 1,000:1, or to 900:1, or to 800:1, or to 700:1, or to 600:1, or to 500:1. However,
10 preferably the first particulate component is substantially free of fluorescent whitening agent. By substantially free of fluorescent whitening agent it is typically meant that the first particulate component comprises no deliberately added fluorescent whitening agent.

Preferably, the first particulate component is substantially free of zeolite builder. By substantially free of zeolite builder it is typically meant that the first particulate
15 component comprises no deliberately added zeolite builder.

Preferably, the first particulate component is substantially free of phosphate builder. By substantially free of phosphate builder it is typically meant that the first particulate component comprises no deliberately added phosphate builder.

Preferably, the first particulate component comprises a deterative surfactant, more
20 preferably an anionic deterative surfactant. Preferably, the first particulate component comprises from 4wt% to 60wt%, more preferably from 6wt%, or from 8wt%, or from 10wt%, or from 12wt%, and preferably to 55wt%, or to 50wt%, or to 45wt%, or to 40wt% by weight of the first particulate component, of an anionic deterative surfactant.

If the composition comprises sodium carbonate, then preferably the first particulate
25 component comprises at least 2wt%, or at least 5wt%, or at least 10wt%, or at least 15wt%, or even at least 20wt% by weight of the first particulate component, of sodium carbonate.

The first particulate component may be in any suitable particulate form, including spray-dried form and non-spray-dried form. Most preferably, the first particulate
30 component is in spray-dried form. However, the first particulate component may be in the

form of an agglomerate, extrudate, needle, noodle, flake, preferably in the form of an agglomerate.

Typically, the first particulate component has a particle size distribution such that it has a weight average particle size of from 250 micrometers to 850 micrometers, and
5 wherein no more than 10wt%, preferably no more than 5wt%, of the first particulate component has a particle size of less than 210 micrometers, and wherein no more than 10wt%, preferably no more than 5wt%, of the first particulate component has a particle size greater than 1,180 micrometers.

10 Second particulate component

The second particulate component comprises: (i) at least 0.20wt%, preferably at least 0.5wt%, or at least 1wt%, or at least 5wt%, or at least 10wt%, or at least 20wt%, or at least 30wt%, or at least 40wt%, or at least 50wt%, or at least 60wt%, or at least 70wt%, or at least 80wt%, or even at least 90wt% by weight of the second particulate component,
15 of a fluorescent whitening agent; and (ii) from 0wt% to less than 20wt%, preferably from 0wt% to 15wt%, or from 0wt% to 10wt%, or from 0wt% to 8wt%, or from 0wt% to 6wt%, or from 0wt% to 4wt%, or from 0wt% to 2wt%, or from 0wt% to 1wt% by weight of the second particulate component, of carbonate. Preferably, the second particulate component is substantially free of carbonate. By substantially free of carbonate it is
20 typically meant that the second particulate component comprises no deliberately added carbonate. Typically, the second particulate component comprises one or more adjunct components; the remaining portion, if any, of the second particulate component is typically made up of adjunct components.

If the composition comprises sodium carbonate, then preferably the second
25 particulate component comprises from 0wt% to less than 20wt%, preferably from 0wt% to 15wt%, or from 0wt% to 10wt%, or from 0wt% to 8wt%, or from 0wt% to 6wt%, or from 0wt% to 4wt%, or from 0wt% to 2wt%, or even from 0wt% to 1wt% by weight of the second particulate component, of sodium carbonate. Preferably, the second particulate component is substantially free of sodium carbonate. By substantially free of sodium
30 carbonate it is typically meant that the second particulate component comprises no deliberately added sodium carbonate.

Preferably, the second particulate component is substantially free of zeolite builder. By substantially free of zeolite builder it is typically meant that the second particulate component comprises no deliberately added zeolite builder.

Preferably, the second particulate component is substantially free of phosphate
5 builder. By substantially free of phosphate builder it is typically meant that the second particulate component comprises no deliberately added phosphate builder.

The second particulate component may be in any suitable particulate form, including spray-dried form and non-spray-dried form, preferably non-spray-dried form. Preferably, the second particulate component is in the form of an agglomerate, extrudate,
10 needle, noodle, flake, preferably an agglomerate.

Typically, the second particulate component has a particle size distribution such that it has a weight average particle size of from 250 micrometers to 850 micrometers, and wherein no more than 10wt%, preferably no more than 5wt%, of the second particulate component has a particle size of less than 210 micrometers, and wherein no more than
15 10wt%, preferably no more than 5wt%, of the second particulate component has a particle size greater than 1,180 micrometers.

Solid laundry detergent composition

The composition is in free-flowing particulate form; this means that the
20 composition is in the form of separate discrete particles; separate particles typically means that the particles in the composition are individual units of particulate matter that are physically distinct from one another. The composition can be in any free-flowing particulate form, such as in the form of an agglomerate, a spray-dried powder, an extrudate, a flake, a needle, a noodle, a bead, or any combination thereof.

25 The detergent composition typically has a bulk density of from 450g/l to 1,000g/l, preferred low bulk density detergent compositions have a bulk density of from 550g/l to 650g/l and preferred high bulk density detergent compositions have a bulk density of from 750g/l to 900g/l.

During the laundering process, the composition is typically contacted with water to
30 give a wash liquor having a pH of from above 7 to less than 13, preferably from above 7

to less than 10.5. This is the optimal pH to provide good cleaning whilst also ensuring a good fabric care profile.

The composition may be made by any suitable method including agglomeration, spray-drying, extrusion, mixing, dry-mixing, liquid spray-on, roller compaction, spheronisation or any combination thereof.

The weight ratio of the first particulate component to the second particulate component is in the range of from 1:1 to 1,000:1, preferably from 5:1, or from 10:1, or from 15:1, or from 20:1, or from 25:1, or from 30:1, or from 40:1, or from 50:1, or from 60:1, and preferably to 900:1, or to 800:1, or to 700:1, or to 600:1.

The composition comprises from 0wt% to 10wt%, preferably to 8wt%, or to 6wt%, or to 4wt%, or to 2wt%, or even to 1wt% zeolite builder. Preferably, the composition is substantially free of zeolite builder. By substantially free of zeolite builder, it is typically meant that no zeolite builder is deliberately incorporated into the composition. Typical zeolite builders are zeolite A, zeolite P and zeolite MAP.

The composition comprises from 0wt% to 10wt%, preferably to 8wt%, or to 6wt%, or to 4wt%, or to 2wt%, or even to 1wt% phosphate builder. Preferably, the composition is substantially free of phosphate builder. By substantially free of phosphate builder, it is typically meant that no phosphate builder is deliberately incorporated into the composition. A typical phosphate builder is sodium tri-polyphosphate.

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Adjunct components

The composition typically comprises one or more adjunct components. These adjunct components include: bleach such as percarbonate and/or perborate, preferably in combination with a bleach activator such as tetraacetyl ethylene diamine, oxybenzene sulphonate bleach activators such as nonanoyl oxybenzene sulphonate, caprolactam bleach activators, imide bleach activators such as N-nonanoyl-N-methyl acetamide, preformed peracids such as N,N-phthaloylamino peroxycaproic acid, nonylamido peroxyadipic acid or dibenzoyl peroxide; chelants such as diethylene triamine pentaacetate, diethylene triamine penta(methyl phosphonic acid), ethylene diamine-N'N'-disuccinic acid, ethylene diamine tetraacetate, ethylene diamine tetra(methylene phosphonic acid) and hydroxyethane di(methylene phosphonic acid); enzymes such as

amylases, carbohydrases, cellulases, laccases, lipases, oxidases, peroxidases, proteases, pectate lyases and mannanases; suds suppressing systems such as silicone based suds suppressors; photobleach; filler salts; fabric-softening agents such as clay, silicone and/or quaternary ammonium compounds; flocculants such as polyethylene oxide; dye transfer inhibitors such as polyvinylpyrrolidone, poly 4-vinylpyridine N-oxide and/or co-polymer of vinylpyrrolidone and vinylimidazole; fabric integrity components such as hydrophobically modified cellulose and oligomers produced by the condensation of imidazole and epichlorhydrin; soil dispersants and soil anti-redeposition aids such as polymeric carboxylates, alkoxyated polyamines including ethoxylated ethyleneimine polymers; anti-redeposition components such as carboxymethyl cellulose and polyesters; perfumes; and dyes.

EXAMPLES

15 A particulate laundry detergent composition and process of making it.

Aqueous slurry composition.

Component	% w/w Aqueous slurry
A compound having the following general structure: bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n)(CH ₃)-N ⁺ -C _x H _{2x} -N ⁺ -(CH ₃)- bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n), wherein n = from 20 to 30, and x = from 3 to 8, or sulphated or sulphonated variants thereof	1.23
Ethylenediamine disuccinic acid	0.35
Magnesium sulphate	0.72
Acrylate/maleate copolymer	6.41
Linear alkyl benzene sulphonate	12.18
Hydroxyethane di(methylene phosphonic acid)	0.32
Sodium carbonate	12.87
Sodium sulphate	38.60
Soap	0.78

Water	26.13
Miscellaneous	0.41
Total Parts	100.00

Preparation of a spray-dried powder.

An aqueous slurry having the composition as described above is prepared having a moisture content of 26.13%. The aqueous slurry is heated to 72°C and pumped under high pressure (from $5.5 \times 10^6 \text{ Nm}^{-2}$ to $6.0 \times 10^6 \text{ Nm}^{-2}$), into a counter current spray-drying tower with an air inlet temperature of from 270°C to 300°C. The aqueous slurry is atomised and the atomised slurry is dried to produce a solid mixture, which is then cooled and sieved to remove oversize material (>1.8mm) to form a spray-dried powder, which is free-flowing. Fine material (<0.15mm) is elutriated with the exhaust the exhaust air in the spray-drying tower and collected in a post tower containment system. The spray-dried powder has a moisture content of 1.0wt%, a bulk density of 420g/l and a particle size distribution such that 95.2wt% of the spray-dried powder has a particle size of from 150 to 710 micrometers. The composition of the spray-dried powder is given below.

Spray-dried powder composition.

Component	% w/w Spray-dried powder
A compound having the following general structure: bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n)(CH ₃)-N ⁺ -C _x H _{2x} -N ⁺ -(CH ₃)-bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n), wherein n = from 20 to 30, and x = from 3 to 8, or sulphated or sulphonated variants thereof	1.65
Ethylenediamine disuccinic acid	0.47
Magnesium sulphate	0.96
Acrylate/maleate copolymer	8.59
Linear alkyl benzene sulphonate	16.33
Hydroxyethane di(methylene phosphonic acid)	0.43
Sodium carbonate	17.25

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Sodium sulphate	51.76
Soap	1.04
Water	1.00
Miscellaneous	0.52
Total Parts	100.00

Preparation of an anionic surfactant particle

The anionic deterative surfactant particle is made on a 554g batch basis using a Tilt-A-Pin then Tilt-A-Plow mixer (both made by Processall). 85g sodium sulphate supplied is added to the Tilt-A-Pin mixer along with 273g sodium carbonate. 196g of 70% active C₂₅E₃S paste (sodium ethoxy sulphate based on C_{12/15} alcohol and ethylene oxide) is added to the Tilt-A-Pin mixer. The components are then mixed at 1200rpm for 10 seconds. The resulting powder is then transferred into a Tilt-A-Plow mixer and mixed at 200rpm for 2 minutes to form particles. The particles are then dried in a fluid bed dryer at a rate of 2500 l/min at 120°C until the equilibrium relative humidity of the particles is less than 15%. The dried particles are then sieved and the fraction through 1180µm and on 250µm is retained. The composition of the anionic deterative surfactant particle is as follows:

- 25.0%w/w C₂₅E₃S sodium ethoxy sulphate
- 17.0%w/w sodium sulphate
- 54.57%w/w sodium carbonate
- 3.43%w/w water

Preparation of a cationic deterative surfactant particle

The cationic surfactant particle is made on a 17kg batch basis on a Morton FM-50 Loedige mixer. 5.1kg of sodium sulphate and 5.1kg micronised sodium carbonate are premixed in the Morton FM-50 Loedige mixer. 5.8kg of 50% active mono-C₈-C₁₀ alkyl, mono-hydroxyethyl, di-methyl, ammonium chloride (cationic deterative surfactant) aqueous solution is added to the Morton FM-50 Loedige mixer whilst both the main drive and the chopper are operating. After approximately two minutes of mixing, a 1.0kg 1:1

weight ratio mix of micronised sodium sulphate and micronised sodium carbonate is added to the mixer. The resulting agglomerate is collected and dried using a fluid bed dryer on a basis of 2500l/min air at 100-140°C for 30 minutes. The resulting powder is sieved and the fraction through 1400µm is collected as the cationic deterative surfactant particle. The composition of the cationic deterative surfactant particle is as follows:

- 20 %w/w mono-C₈-C₁₀ alkyl, mono-hydroxyethyl, di-methyl, ammonium chloride
- 38.5 %w/w sodium carbonate
- 38.5 %w/w sodium sulphate
- 3.0 %w/w moisture and miscellaneous

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Preparation of a fluorescent whitening agent particle

A 1kg mixture of disodium 4,4'-bis{[4-anilino-6-morpholino-s-triazin-2-yl]-amino}-2,2'-stilbenedisulphonate powder and disodium 4,4'-bis-(2-sulphostyryl)biphenyl powder, in a powder weight ratio of 6.7 :1 is prepared by dosing 0.87kg of a disodium 4,4'-bis{[4-anilino-6-morpholino-s-triazin-2-yl]-amino}-2,2'-stilbenedisulphonate powder (having an activity of 86wt%) and 0.13kg of disodium 4,4'-bis-(2-sulphostyryl)biphenyl powder (having an activity of 90wt%) into a five litre capacity vertical screw mixer, and mixing the powders together to form a fluorescent whitening agent particle. The fluorescent whitening agent particle has the following composition:

- 74.8 %w/w Dianilino-Dimorpholino amino stilbene derivative
- 11.7 % w/w 4, 4' - distryl biphenyl
- 13.5 % w/w water and miscellaneous

Preparation of a granular laundry detergent composition

10.87kg of the spray-dried powder described above, 4.76kg of the anionic deterative surfactant particle described above, 1.18kg of the cationic deterative surfactant particle described above and 8.19kg (total amount) of other individually dosed dry-added material are dosed into a 1m diameter concrete batch mixer operating at 24rpm. Once all of the materials are dosed into the mixer, the mixture is mixed for 5 minutes to form a granular laundry detergent composition. The formulation of the granular laundry detergent composition is described below:

A granular laundry detergent composition.

Component	% w/w granular laundry detergent composition
Spray-dried powder	43.47
Citric acid	5.00
Sodium percarbonate (having from 12% to 15% active AvOx)	13.26
Photobleach particle	0.01
Lipase (11.00mg active/g)	0.70
Amylase (21.55mg active/g)	0.33
Protease (56.00mg active/g)	0.43
Tetraacetyl ethylene diamine agglomerate (92wt% active)	3.95
Suds suppressor agglomerate (11.5wt% active)	0.87
Green/blue carbonate speckle	0.50
Anionic deterative surfactant particle	19.04
Cationic deterative surfactant particle	4.70
Carboxy methyl cellulose	1.43
Sodium sulphate	5.51
Fluorescent whitening agent particle	0.17
Solid perfume particle	0.63
Total Parts	100.00

CLAIMS

1. A solid laundry detergent composition comprising:

- (a) a deterative surfactant;
- (b) from 0wt% to 10wt% zeolite builder;
- (c) from 0wt% to 10wt% phosphate builder;
- (d) carbonate; and
- (e) a fluorescent whitening agent;

wherein the composition is in free-flowing particulate form and comprises at least two separate particulate components,

wherein the first particulate component comprises:

- (i) a deterative surfactant;
- (ii) at least 10wt%, by weight of the first particulate component, of carbonate;
- (iii) from 0wt% to 10wt%, by weight of the first particulate component, of a zeolite builder;
- (iv) from 0wt% to 10wt%, by weight of the first particulate component, of a phosphate builder; and
- (v) from 0wt% to less than 5wt%, by weight of the first particulate component, of a fluorescent whitening agent; and

wherein the second particulate component comprises:

- (i) at least 0.20wt%, by weight of the second particulate component, of a fluorescent whitening agent; and
- (ii) from 0wt% to less than 20wt%, by weight of the second particulate component, of carbonate.

2. A composition according to any preceding claim, wherein if the first particulate component comprises a fluorescent whitening agent, then the weight ratio of carbonate to fluorescent whitening agent present in the first particulate component is at least 30:1.

3. A composition according to claim 1, wherein the first particulate component is substantially free of fluorescent whitening agent.

4. A composition according to any preceding claim, wherein the first particulate component is substantially free of zeolite builder and phosphate builder.
5. A composition according to any preceding claim, wherein the first particulate component comprises an anionic deterative surfactant.
6. A composition according to any preceding claim, wherein the second particulate component is substantially free from carbonate.
7. A composition according to any preceding claim, wherein the second particulate component comprises at least 50wt%, by weight of the second particulate component, of a fluorescent whitening agent.
8. A composition according to any preceding claim, wherein the composition comprises sodium carbonate, wherein the first particulate component comprises at least 10wt%, by weight of the first particulate component, of sodium carbonate, and wherein the second particulate component comprises from 0wt% to 10wt%, by weight of the second particulate component, of sodium carbonate.
9. A composition according to any preceding claim, wherein the weight ratio of the first particulate component to the second particulate component is in the range of from 60:1 to 600:1.
10. A composition according to any preceding claim, wherein the first particulate component is in spray-dried form, and wherein the second particulate component is in non-spray-dried form.
11. A composition according to any preceding claim, wherein the first particulate component has a particle size distribution such that it has a weight average particle size of from 250 micrometers to 850 micrometers, and wherein no more than 10wt% of the first

particulate component has a particle size of less than 210 micrometers, and wherein no more than 10wt% of the first particulate component has a particle size greater than 1,180 micrometers.

12. A composition according to any preceding claim, wherein the second particulate component has a particle size distribution such that it has a weight average particle size of from 250 micrometers to 850 micrometers, and wherein no more than 10wt% of the second particulate component has a particle size of less than 210 micrometers, and wherein no more than 10wt% of the second particulate component has a particle size greater than 1,180 micrometers.

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2006/050438

A. CLASSIFICATION OF SUBJECT MATTER
INV. C11D3/42 C11D3/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 080 711 A (BROUWER ET AL) 27 June 2000 (2000-06-27) column 20, line 55 - column 26, line 61 examples 1,5-13,34 claims 1-13	1-12
A	WO 2004/041982 A (UNILEVER PLC; UNILEVER NV; HINDUSTAN LEVER LIMITED) 21 May 2004 (2004-05-21) cited in the application claims 1-31; examples 4-6	1-12

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
"&" document member of the same patent family

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 6080711	A	27-06-2000	NONE	
WO 2004041982	A	21-05-2004	AU 2003277486 A1	07-06-2004
			BR 0315968 A	20-09-2005
			CA 2503144 A1	21-05-2004
			EP 1558717 A1	03-08-2005
			US 2004097394 A1	20-05-2004