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(54) **DETERGENT COMPOSITION COMPRISING ZEOLITE AND AMYLASE ENZYME**

ZEOLITH UND AMYLASE ENTHALTENDES WASCHMITTEL

COMPOSITION DETERGENTE COMPRENANT DE LA ZEOLITE ET UNE ENZYME AMYLASE

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US-A- 5 030 377

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Description

[0001] The present invention relates to a particulate laundry detergent composition comprising an amylase enzyme and a small particle size zeolite component as a sequestering agent for water hardness.

[0002] Conventionally, water soluble inorganic phosphates, such as sodium tripolyphosphate, have been used as builders for laundry detergents.

[0003] More recently, alkali metal aluminosilicate ion-exchanges, particularly crystalline water insoluble sodium aluminosilicate zeolites, have been proposed as replacements for the inorganic phosphates.

[0004] For example, EP 21491A (Procter & Gamble) discloses detergent compositions containing a builder system which includes zeolite A, X or P (B) or a mixture thereof. EP 384070A (Unilever) discloses specific zeolite P materials having an especially low silicon to aluminium ratio not greater than 1.33 (hereinafter referred to as zeolite MAP) and describes their use as detergency builders.

[0005] It has been proposed in the literature to use zeolites having a wide range of particle sizes. For instance EP-A-580245 proposes a range of 0.005 μm to 20 μm , W094/00545 proposes sizes of below 6 μm , DE-A-2656009 proposes a range of 0.5 to 12 μm and EP-A-0384070 proposes a zeolite MAP having a d_{50} size of 0.1 to 5 μm , with 0.1 to 1 μm being preferred when the composition is to be a stable liquid.

[0006] It is conventional to propose the use of enzymes in detergent compositions and disclosures which propose the use of enzymes in combination with small particle size MAP include EP 384070 A, EP 448297 A, EP 522726 A, EP 533392 A, EP 544492 A, EP 552053 A and EP 552054A.

[0007] The invention is based in part on the discovery that a problem may occur when a water insoluble zeolite, having a small particle size, is used as a detergency builder in a composition formulated for use in the laundering of fabrics. The problem has been found to be particularly pronounced when the zeolite is zeolite MAP.

[0008] The problem relates to the formation of white residues, which adhere to the fabrics and remain thereon at the end of a laundry washing process. The degree of residue formation may vary. On coloured fabrics the appearance of the white residues tends to be visually more apparent than on white fabrics. White residues frequently form on areas of fabric where there is a stain present, interfering with and preventing the complete removal of the stain. As a result of the visible contrast between the white residues and the coloured fabric, the stained area on which white deposits have formed may be more noticeable than the original stain.

[0009] The invention is based also on the surprising discovery that this problem of white residue formation when the zeolite has a defined small particle size can be reduced by including amylase enzyme in the laundry detergent.

[0010] A particulate laundry detergent composition according to the invention comprises

- (a) surfactant comprising anionic surfactant,
- (b) zeolite builder,
- (c) organic or inorganic cobuilder,

characterised in that

the zeolite builder has a particle size, expressed as a d_{50} value, of less than 1 micrometre and the composition comprises an amylase enzyme.

[0011] The invention also includes use of an amylase enzyme in a particulate laundry composition for reducing white residues on fabrics subjected to laundry washing using the composition, wherein the composition contains surfactant comprising anionic surfactant, zeolite builder having a particle size, expressed as a d_{50} value, of less than 1.0 micrometres, and organic or inorganic cobuilder.

[0012] Preferably the zeolite builder comprises zeolite P having a silicon to aluminium ratio of not greater than 1.33 (zeolite MAP).

Detailed description of the inventionZeolite builder

[0013] The first essential component of the present invention is an aluminosilicate zeolite builder, optionally in conjunction with one or more supplementary builders.

[0014] The zeolite builder is typically present at a level of from 1% to 80%, more preferably from 15% to 40% by weight of the compositions.

[0015] In an essential aspect the zeolite detergent builder has a particle size, expressed as a d_{50} value of less than 1.0 micrometres, more preferably from 0.05 to 0.9 micrometres, most preferably from 0.2 to 0.7 micrometres.

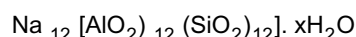
[0016] The d_{50} value indicates that 50% by weight of the particles have a diameter smaller than that figure. The particle size may, in particular be determined by conventional analytical techniques such as microscopic determination

using a scanning electron microscope or by means of a laser granulometer.

[0017] Suitable aluminosilicate zeolites have the unit cell formula $\text{Na}_x[(\text{AlO}_2)_z(\text{SiO}_2)_y] \cdot x\text{H}_2\text{O}$ wherein z and y are at least 6; the molar ratio of z to y is from 1.0 to 0.5 and x is at least 5, preferably from 7.5 to 276, more preferably from 10 to 264. The aluminosilicate material are in hydrated form and are preferably crystalline, containing from 10% to 28%, more preferably from 18% to 22% water in bound form.

[0018] The aluminosilicate zeolites can be naturally occurring materials, but are preferably synthetically derived. Synthetic crystalline aluminosilicate ion exchange materials are available under the designations Zeolite A, Zeolite B, Zeolite P, Zeolite X, Zeolite MAP, Zeolite HS and mixtures thereof.

[0019] Zeolite A has the formula



wherein x is from 20 to 30, especially 27. Zeolite X has the formula $\text{Na}_{86} [(\text{AlO}_2)_{86}(\text{SiO}_2)_{106}] \cdot 276 \text{H}_2\text{O}$.

[0020] Zeolite MAP is described in EP 384070A (Unilever). It is defined as an alkali metal aluminosilicate of the zeolite P type having a silicon to aluminium ratio not greater than 1.33, preferably within the range from 0.9 to 1.33 and more preferably within the range of from 0.9 to 1.2.

[0021] Of particular interest is zeolite MAP having a silicon to aluminium ratio not greater than 1.15 and, more particularly, not greater than 1.07.

[0022] Zeolite P having a Si:Al ratio of 1.33 or less may be prepared by the following steps:

(i) mixing together a sodium aluminate having a mole ratio $\text{Na}_2\text{O}:\text{Al}_2\text{O}_3$ within the range of from 1.4 to 2.0 and a sodium silicate having a mole ratio $\text{SiO}_2:\text{Na}_2\text{O}$ within the range of from 0.8 to 3.4 with vigorous stirring at a temperature within the range of from 25°C to boiling point usually 95°C, to give a gel having the following composition; $\text{Al}_2\text{O}_3:(1.75-3.5) \text{SiO}_2:(2.3-7.5)\text{Na}_2\text{O}:\text{P} (80-450)\text{H}_2\text{O}$;

(ii) ageing the gel composition for 0.5 to 10 hours, preferably 2 to 5 hours, at a temperature within the range of from 70°C to boiling point, usually to 95°C, with sufficient stirring to maintain any solids present in suspension;

(iii) separating the crystalline sodium aluminosilicate thus formed, washing to a pH within the range of from 10 to 12.5, and drying, preferably at a temperature not exceeding 150°C, to a moisture content of not less than 5 wt. %.

[0023] Preferred drying methods are spray-drying and flash drying. It appears that oven drying at too high a temperature may adversely affect the calcium binding capacity of the product under certain circumstances.

[0024] Commercial sodium metasilicate pentahydrate dissolved in water and commercial sodium silicate solution (waterglass) are both suitable silica sources for the production of zeolite P in accordance with the invention. The reactants may be added together in any order either rapidly or slowly. Rapid addition at ambient temperature, and slow addition at elevated temperature (90-95°C) both give the desired product.

[0025] Vigorous stirring of the gel during the addition of the reactants, and at least moderate stirring during the subsequent ageing step, however, appear to be essential for the formation of pure zeolite P. In the absence of stirring, various mixtures of crystalline and amorphous materials may be obtained.

[0026] Zeolite MAP generally has a calcium binding capacity of at least 150 mg CaO per g of anhydrous aluminosilicate, as measured by the standard method described in GB 1473201 (Henkel). The calcium binding capacity is normally 160 mg CaO/g and may be as high 170 mg CaO/g.

[0027] Although zeolite MAP like other zeolites contains water of hydration, for the purposes of the present invention amounts and percentages of zeolite are expressed in terms of the notional anhydrous material.

[0028] The amount of water present in hydrated zeolite MAP at ambient temperature and humidity is generally about 20 wt. %.

Amylase

[0029] The second essential component of the compositions is an amylase enzyme, that is to say an enzyme having amylolytic activity.

[0030] The amylase enzyme is typically incorporated into the compositions in accordance with the invention at a level of from 0.01% to 5%, preferably from 0.1% to 3%, more preferably from 0.2% to 2%, most preferably from 0.3% to 1.5% active enzyme by weight of the composition, on a 60KNU/g (Kilo Novo Units/gram) activity basis.

[0031] The units of 'Kilo Novo Units/gram (KNU/g)' are a well known means of defining amylolytic enzyme activity and are described in GB-1,269,839 A (Novo). In more detail, 1 KNU is the amount of enzyme which breaks down 5.25

grams of starch (Merck, Amylum Solubile Erg. B.6, Batch 9947275) per hour in the method described in GB-1,269,839 A, which has the following standard conditions.

Substrate	Soluble starch
Calcium content in solvent	0.0043 M
Reaction time	7-20 minutes
Temperature	37°C
pH	5.6

[0032] The amylase enzyme may be fungal or bacterial in origin. Amylases obtained by chemical or genetic manipulation of fungal or bacterial derived strains are also useful herein. The amylase enzyme is preferably an α -amylase.

[0033] Preferred amylases include, for example, α -amylases obtained from a special strain of *B. licheniformis*, described in more detail in GB-1,269,839 A. Reported deposit numbers for *B. licheniformis* strains capable of producing α -amylases include NCIB 8061, NCIB 8059, ATCC 6634, ATCC 6598, ATCC 11945, ATCC 8480 and ATCC 9945a.

[0034] Preferred commercially available α -amylases include for example, those sold under the tradename Rapidase and Maxamyl by Gist-Brocades; those sold under the tradename Taka-Therm L-340 by Miles Laboratories, Elkhart, Indiana; those sold under the tradename Rohalase AT by Rohm and Haas, West Philadelphia, PA; and those sold under the tradenames Termamyl 60T and 120T, Fungamyl and BAN by Novo Industries A/S.

[0035] In a preferred aspect, the amylases have been designed to have improved stability, particularly having improved stability to oxidation, for example in a bleaching environment, and improved thermal stability. Stability can be measured using any of the technical tests known in the art including those referred to in WO 94/02597 A. Stability-enhanced amylases are commercially available from Novo Industries A/S or from Genencor International.

[0036] Highly preferred amylases with enhanced oxidative stability are derived using site-directed mutagenesis from one or more of the *Bacillus* amylases, especially the *Bacillus* α -amylases, regardless of whether one, two or multiple amylase strains are the immediate precursors. Preferred amylases of this type are described in WO 94/02597 A, and comprise a mutant in which substitution is made, using alanine or threonine, preferably threonine, of the methionine residue located in position 197 of the *B. licheniformis* α -amylase, sold under the tradename Termamyl, or the homologous position variation of a similar parent amylase, such as *B. amyloliquefaciens*, *B. subtilis*, or *B. stearothermophilus*.

[0037] Other preferred amylases having enhanced oxidative stability, derived from *B. licheniformis* NCIB806, are described by Genencor International in a paper entitled "Oxidatively Resistant α -Amylases" which was presented at the 207th American Chemical Society National Meeting, March 13-17 1994, by C. Mitchinson. Methionine (Met) was identified as the most likely residue to be modified. Met was substituted, one at a time, in positions 8, 15, 197, 256, 304, 366 and 438 leading to specific mutants, particularly important being M197L and M197T with the M197T variant being the most stable expressed variant.

[0038] Other preferred amylases having enhanced oxidative stability include those described in WO 94/18314 A (Genencor International) and WO 94/02597 A (Novo). Any other oxidative stability-enhanced amylase can be used, for example as derived by site-directed mutagenesis from known chimeric, hybrid or simple mutant parent forms of available amylases. Other enzyme modifications are acceptable including those described in WO 95/09909 A (Novo).

[0039] It will be appreciated that enzymes for incorporation into solid detergent compositions are generally sold commercially as enzyme prills containing active enzyme supported on a variety of inert host materials, which for example, can include alkali metal sulfates, carbonates and silicates. Optionally, organic binder materials are also incorporated. In a preferred aspect, the calcium content of these enzyme prills is minimized to ensure good in-product storage stability of the enzyme.

Additional detergent components

[0040] The detergent composition according to the invention contains surfactant and cobuilder and may contain other detergent components such as fluorescers, antiredeposition agents, inorganic salts such as sodium sulphate, other enzymes, lather control agents, fabric softening agents, pigments, coloured speckles and perfumes.

Surfactant

[0041] The detergent composition according to the invention includes a surfactant selected from anionics, nonionics, zwitterionics, ampholytics and cationics.

[0042] The surfactant is preferably present in the detergent compositions at a level of from 1% to 50%, preferably

from 3% to 30%, most preferably from 5% to 20% by weight of the compositions.

[0043] Many suitable detergent-active compounds are available and fully described in the literature (for example "Surface Active Agents and Detergents" Volumes I and II by Schwartz, Perry and Berch).

[0044] Examples of suitable additional anionic surfactants include anionic sulfates, olefin sulphonates, alkyl xylene sulphonates, dialkylsulphosuccinates, and fatty acid ester sulphonates. Sodium salts are generally preferred.

Anionic sulfate surfactant

[0045] Anionic sulfate surfactants suitable for use herein include the linear and branched primary alkyl sulfates, alkyl ethoxysulfates, fatty oleoyl glycerol sulfates, alkyl phenol ethylene oxide ether sulfates, the C₅-C₁₇ acyl-N-(C₁-C₄ alkyl) and -N-(C₁-C₂ hydroxyalkyl) glucamine sulfates, and sulfates of alkylpolysaccharides such as the sulfates of alkylpolyglucoside (the nonionic nonsulfated compounds being described herein).

[0046] Alkyl ethoxysulfate surfactants are preferably selected from the group consisting of the C₆-C₁₈ alkyl sulfates which have been ethoxylated with from 0.5 to 20 moles of ethylene oxide per molecule. More preferably, the alkyl ethoxysulfate surfactant is a C₆-C₁₈ alkyl sulfate which has been ethoxylated with from 0.5 to 20, preferably from 0.5 to 5, moles of ethylene oxide per molecule.

Anionic sulfonate surfactant

[0047] Anionic sulfonate surfactants suitable for use herein include the salts of C₅-C₂₀ linear alkylbenzene sulfonates, alkyl ester sulfonates, C₆-C₂₂ primary or secondary alkane sulfonates, C₆-C₂₄ olefin sulfonates, sulfonated polycarboxylic acids, alkyl glycerol sulfonates, fatty acyl glycerol sulfonates, fatty oleyl glycerol sulfonates, and any mixtures thereof.

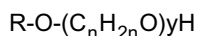
Nonionic surfactant

[0048] The nonionic surfactant is preferably a hydrophobic nonionic surfactant, particularly an alkoxyated nonionic surfactant, having a hydrophilic lipophilic balance (hlb) value of < 9.5, more preferably < 10.5.

[0049] Examples of suitable hydrophobic alkoxyated nonionic surfactants include alkoxyated adducts of fatty alcohols containing an average of less than 5 alkylene oxide groups per molecule.

[0050] The alkylene oxide residues may, for example, be ethylene oxide residues or mixtures thereof with propylene oxide residues.

[0051] Preferred alkylene oxide adducts of fatty alcohols useful in the present invention can suitably be chosen from those of the general formula:



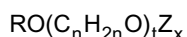
wherein R is an alkyl or alkenyl group having at least 10 carbon atoms, most preferably from 10 to 22 carbon atoms, y is from 0.5 to 3.5 and n is 2 or 3.

[0052] Preferred nonionic surfactants include primary C₁₁-C₁₅ aliphatic alcohols condensed with an average of no more than five ethylene oxide groups per mole of alcohol, having an ethylene oxide content of less than 50% by weight, preferably from 25% to less than 50% by weight.

[0053] A particularly preferred aliphatic alcohol ethoxylated is a primary alcohol having an average of 12 to 15 carbon atoms in the alkyl chain condensed with an average of three ethoxy groups per mole of alcohol.

[0054] Specific examples of suitable alkoxyated adducts of fatty alcohols are Synperonic A3 (ex ICI), which is a C₁₃-C₁₅ alcohol with about three ethylene oxide groups per molecule and Empilan KB3 (ex Marchon), which is lauric alcohol 3EO.

[0055] Another class of nonionic surfactants comprises alkyl polyglucoside compounds of general formula



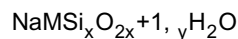
wherein Z is a moiety derived from glucose; R is a saturated hydrophobic alkyl group that contains from 12 to 18 carbon atoms; t is from 0 to 10 and n is 2 or 3; x is from 1.1 to 4, the compounds including less than 10% unreacted fatty alcohol and less than 50% short chain alkyl polyglucosides. Compounds of this type and their use in detergent compositions are disclosed in EP-B 0070074, 0070077, 0075996 and 0094118.

Cobuilders

[0056] In addition to zeolite MAP, the builder system includes an organic or inorganic cobuilder.

[0057] Suitable organic cobuilders can be monomeric or polymeric carboxylates such as citrates or polymers of acrylic, methacrylic and/or maleic acids in neutralised form. Suitable inorganic cobuilders include carbonates and amorphous and crystalline layered silicates.

[0058] Suitable crystalline layered silicates have the composition:



where M is sodium or hydrogen, preferably sodium; x is a number from 1.9 to 4; and y is a number from 0 to 20. Such materials are described in US Patents No. 4664839; No. 4728443 and No. 4820439 (Hoechst AG). Especially preferred are compounds in which x = 2 and y = 0. The synthetic material is commercially available from Hoechst AG as 8-Na₂Si₂O₅ (SKS6) and is described in US Patent No. 4664830.

[0059] The total amount of detergency builder in the granular composition typically ranges from 10 to 80 wt.%, more preferably from 15 to 60 wt% and most preferably from 10 to 45 wt%.

Bleach

[0060] Detergent compositions according to the invention may also suitably contain a bleach system. This preferably comprises one or more peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with bleach precursors to improve bleaching action at low temperatures.

[0061] The bleach system preferably comprises a peroxy bleach compound, preferably an inorganic persalt, optionally in conjunction with a peroxyacid bleach precursor. Suitable persalts include sodium perborate monohydrate and tetrahydrate and sodium percarbonate, with sodium percarbonate being most preferred.

[0062] Preferred bleach precursors are peracetic acid precursors, such as tetraacetylene diamine (TAED); peroxybenzoic acid precursors.

Making process

[0063] The detergent compositions of the invention may be prepared by any suitable method. The particulate detergent compositions are suitably prepared by any tower (spray-drying) or non-tower process.

[0064] In processes based around a spray-drying tower, a base powder is first prepared by spray-drying a slurry and then other components unsuitable for processing via the slurry can be sprayed on or admixed (postdosed).

[0065] The zeolite builder is suitable for inclusion in the slurry, although it may be advantageous for processing reasons for part of the zeolite builder to be incorporated post-tower. The crystalline layered silicate, where this is employed, is also incorporated via a non-tower process and is preferably postdosed.

[0066] Alternatively, particulate detergent compositions in accordance with the invention may be prepared by wholly non-tower processes such as granulation.

[0067] The granular detergent compositions of the invention be prepared to any suitable bulk density. The composition preferably have a bulk density of at least 400 g/l preferably at least 550 g/l, most preferably at least 700 g/l and, with particular preference at least 800 g/l.

[0068] The benefits of the present invention are particularly evident in powders of high bulk density, for example, of 700 g/l or above. Such powders may be prepared either by post-tower densification of spraydried powder, or by wholly non-tower methods such as dry mixing and granulation; in both cases a high-speed mixer/granulator may advantageously be used. Processes using high-speed mixer/granulators are disclosed, for example, in EP340 013A, EP 367 339A, EP 390 251A and EP 420 317A (Unilever).

[0069] Illustrative compositions according to the present invention are presented in the following Examples.

[0070] In the detergent compositions, the abbreviated component identifications have the following meanings:

LAS : C₁₁-C₁₃ linear alkyl benzene sulfonate

45AS : Branched sodium alkyl sulfate surfactant containing C₁₄-C₁₅ alkyl chains

246AS : Sodium alkyl sulfate surfactant containing a alkyl chain length weight distribution of 15% C₁₂ alkyl chains, 45% C₁₄ alkyl chains, 35% C₁₆ alkyl chains, 5% C₁₈ alkyl chains

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TAS : Sodium alkyl sulfate surfactant containing predominantly C₁₆ - C₁₈ alkyl chains derived from tallow oil.

24AE3S : C₁₂-C₁₄ alkyl ethoxysulfate containing an average of three ethoxy groups per mole

35E3 : AC₁₃₋₁₅ primary alcohol condensed with an average of 3 moles of ethylene oxide

25E3 : A C₁₂-C₁₅ primary alcohol condensed with an average of 3 moles of ethylene oxide

24EY : A C₁₂₋₁₄ linear primary alcohol condensed with an average of Y moles of ethylene oxide

Citrate : Sodium citrate

Carbonate : Anhydrous sodium carbonate

Perborate : Sodium perborate tetrahydrate

Percarbonate : Sodium percarbonate

TAED : Tetra acetyl ethylene diamine

Silicate : Amorphous Sodium Silicate (SiO₂:Na₂O ratio normally follows)

CMC : Carboxymethylcellulose

Suds

Suppressor : 25% paraffin wax Mpt 50°C, 17% hydrophobic silica, 58% paraffin oil

Zeolite MAP : Hydrated sodium aluminosilicate zeolite MAP having a silicon to aluminium ratio of 1.07 having a particle size, expressed as a d₅₀ value, of 0.5 micrometres

Zeolite A : Hydrated sodium aluminosilicate zeolite A having a particle size, expressed as a d₅₀ value, of 0.6 micrometres

MA/AA : Copolymer of 1:4 maleic/acrylic acid, average molecular weight about 80,000.

Amylase : Amylolytic enzyme sold under the tradename Termamyl 60T by Novo Industries A/S (60 KNU/ gram enzyme activity)

BSA : Amylolytic enzyme - M197T variant, having enhanced oxidative stability (60 KNU/gram enzyme activity)

Protease : Proteolytic enzyme sold by Novo Industries A/S under the tradename Savinase of activity 4.0 KNPU/ gram.

Lipase : Lipolytic enzyme sold by Novo Industries A/S under the tradename lipolase of activity 100,000 LU/ gram

Example 1

[0071] The following granular laundry detergent compositions were prepared (parts by weight) in accordance with the invention. All amylase enzyme levels relate to levels of active enzyme, expressed on a 60 KNU/g activity basis.

	A	B	C	D	E
246AS	7.6	6.5	4.8	6.8	-

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(continued)

	A	B	C	D	E
TAS	-	-	-	-	8.6
24AE3S	2.4	-	1.2	1.7	-
25E3	3.26	-	-	-	6.3
35E3	-	5.0	5.0	5.0	-
Zeolite MAP	20.0	25.0	20.0	-	16.0
Zeolite A	-	-	-	25.0	15.0
Carbonate	15.0	15.0	20.0	10.0	12.0
MA/AA	4.25	4.25	4.25	4.25	2.0
Perborate	-	16.0	-	16.0	20.0
Percarbonate	20.0	-	20.0	-	-
TAED	5.0	5.0	5.0	5.0	6.7
Amylase	0.2	0.5	-	0.2	0.1
BSA	-	-	0.1	-	-
Protease	0.04	0.08	-	0.05	0.05
Silicate (2.0 ration	4.0	-	-	4.0	3.0

[0072] Water and miscellaneous (Including suds suppressor, sodium sulphate, perfume) to balance

Example 2

[0073] The following granular laundry detergent compositions of density 850 gram/litre are prepared (parts by weight) in accordance with the invention. All amylase levels relate to levels of active enzyme, expressed on a 60 KNU/g activity basis.

	F	G	H	I	J
45AS	9.0	8.5	9.5	9.0	6.0
LAS	-	-	-	-	3.0
24E3	2.8	2.9	3.0	2.8	2.8
24E5	6.5	6.4	6.5	6.2	6.5
Zeolite MAP	32.0	35.0	25.0	-	16.0
Zeolite A	-	-	-	30.0	15.0
Citrate	3.3	3.0	3.5	3.5	3.0
Carbonate	9.0	9.0	9.0	10.0	12.0
MA/AA	-	-	-	-	2.0
CMC	0.8	0.5	0.8	10.0	0.8
Perborate	-	-	-	-	16.0
Percarbonate	20.0	18.0	20.0	22.0	-
TAED	4.7	4.7	4.7	4.7	4.7
Amylase	0.1	0.3	-	0.5	0.2

(continued)

	F	G	H	I	J
BSA	-	-	0.4	-	-
Protease	2.4	2.0	1.5	2.0	1.0
Lipase	0.35	0.35	0.4	0.3	0.2
Silicate (1.6 ratio)	5.1	6.0	4.5	5.0	5.0

[0074] Water and miscellaneous (Including suds suppressor, sodium sulphate, perfume) to balance

Claims

1. A particulate laundry detergent composition comprising
 - (a) surfactant comprising anionic surfactant,
 - (b) zeolite builder and
 - (c) organic or inorganic cobuilder,

characterised in that

the zeolite builder has a particle size, expressed as a d_{50} value, of less than 1.0 micrometres and the composition comprises an amylase enzyme.
2. A detergent composition according to claim 1 wherein said zeolite builder has a particle size, expressed as a d_{50} value, of from 0.05 to 0.9 micrometres.
3. A detergent composition according to either of claims 1 or 2 wherein said zeolite builder comprises zeolite P having a silicon to aluminium ratio of not greater than 1.33 (zeolite MAP).
4. A detergent composition according to any of claims 1 to 3 wherein said zeolite builder is crystalline and contains 10-28% bound water and is present at a level of from 15% to 40% by weight of the composition.
5. A detergent composition according to any of claims 1 to 4 wherein said amylase enzyme is an α -amylase.
6. A detergent composition according to any of claims 1 to 6 wherein said amylase enzyme is present at a level of from 0.1% to 5%, active enzyme by weight of the composition, on a 60 KNU/g activity basis.
7. A detergent composition according to any of claims 1 to 6 containing surfactant at a level of from 5% to 50% by weight of the composition.
8. A detergent composition according to any preceding claim wherein said cobuilder comprises a crystalline layered silicate or monomeric or polymeric carboxylate.
9. A detergent composition according to any preceding claim containing a bleach system comprising a peroxy bleach compound and a peroxyacid bleach precursor.
10. A detergent composition according to claim 9 wherein said peroxy bleach compound is sodium percarbonate.
11. A particulate detergent composition according to any preceding claim which is a granular composition which has a bulk density of at least 550g/l.
12. A particulate detergent composition according to any preceding claim having bulk density of at least 700g/l and made by post tower densification of spray dried powder or by dry mixing and granulation.
13. Use of an amylase enzyme in a particulate laundry composition for reducing white residues on fabrics subjected to laundry washing using the composition, wherein the composition contains surfactant comprising an anionic

surfactant, zeolite builder having a particle size, expressed as a d_{50} value, of less than 1.0 micrometres, and an organic or inorganic cobuilder.

Patentansprüche

1. Teilchenförmige Wäschewaschmittelzusammensetzung, umfassend

- (a) Tensid, umfassend anionisches Tensid,
- (b) Zeolithbuilder, und
- (c) organischen oder anorganischen Cobuilder,

dadurch gekennzeichnet, dass

der Zeolithbuilder eine Teilchengröße, ausgedrückt als ein d_{50} -Wert, von weniger als 1,0 μm besitzt und die Zusammensetzung ein Amylaseenzym umfasst.

2. Waschmittelzusammensetzung nach Anspruch 1, wobei der Zeolithbuilder eine Teilchengröße, ausgedrückt als ein d_{50} -Wert, von 0,05 bis 0,9 μm aufweist.

3. Waschmittelzusammensetzung nach Anspruch 1 und/oder 2, wobei der Zeolithbuilder Zeolith P mit einem Silicium-zu-Aluminium-Verhältnis von nicht größer als 1,33 (Zeolith MAP) umfasst.

4. Waschmittelzusammensetzung nach mindestens einem der Ansprüche 1 bis 3, wobei der Zeolithbuilder kristallin ist und 10-28% gebundenes Wasser enthält und in einem Anteil von 15 bis 40 Gew.-% der Zusammensetzung vorliegt.

5. Waschmittelzusammensetzung nach mindestens einem der Ansprüche 1 bis 4, wobei das Amylaseenzym eine α -Amylase ist.

6. Waschmittelzusammensetzung nach mindestens einem der Ansprüche 1 bis 6, wobei das Amylaseenzym in einem Anteil von 0,1 bis 5%, aktives Enzym bezogen auf Gewicht der Zusammensetzung, auf einer 60 KNU/g Aktivitätsbasis, vorliegt.

7. Waschmittelzusammensetzung nach mindestens einem der Ansprüche 1 bis 6, enthaltend Tensid in einem Anteil von 5 bis 50 Gew.-% der Zusammensetzung.

8. Waschmittelzusammensetzung nach mindestens einem vorangehenden Anspruch, wobei der Cobuilder ein kristallines Schichtsilicat oder monomeres oder polymeres Carboxylat umfasst.

9. Waschmittelzusammensetzung nach mindestens einem vorangehenden Anspruch, enthaltend ein Bleichsystem, umfassend eine Peroxybleichverbindung und einen Peroxysäure-Bleichvorläufer.

10. Waschmittelzusammensetzung nach Anspruch 9, wobei die Peroxybleichverbindung Natriumpercarbonat ist.

11. Teilchenförmige Waschmittelzusammensetzung nach mindestens einem vorangehenden Anspruch, welche eine granuläre Zusammensetzung mit einer Schüttdichte von mindestens 550 g/l ist.

12. Teilchenförmige Waschmittelzusammensetzung nach mindestens einem vorangehenden Anspruch mit einer Schüttdichte von mindestens 700 g/l und hergestellt durch nachgeschaltete Turmverdichtung von sprühgetrocknetem Pulver oder durch Trockenmischen und Granulation.

13. Verwendung eines Amylaseenzyms in einer teilchenförmigen Wäschewaschmittelzusammensetzung zur Verringerung weißer Rückstände auf Textilien, welche dem Wäschewaschen unter Verwendung der Zusammensetzung unterzogen werden, wobei die Zusammensetzung Tensid, umfassend ein anionisches Tensid, Zeolithbuilder mit einer Teilchengröße, ausgedrückt als ein d_{50} -Wert, von weniger als 1.0 μm , und einen organischen oder anorganischen Cobuilder enthält.

Revendications

1. Composition détergente de blanchissage particulaire comprenant

- (a) un tensioactif comprenant un tensioactif anionique,
- (b) un adjuvant zéolite et
- (c) un co-adjuvant organique ou minéral,

caractérisée en ce que

l'adjuvant zéolite a une taille particulaire, exprimée comme une valeur d_{50} , inférieure à 1,0 micromètre et la composition comprend une enzyme amylase.

2. Composition détergente selon la revendication 1, dans laquelle ledit adjuvant zéolite a une taille particulaire, exprimée comme une valeur d_{50} , de 0,05 à 0,9 micromètre.

3. Composition détergente selon l'une quelconque des revendications 1 ou 2, dans laquelle ledit adjuvant zéolite comprend la zéolite P ayant un rapport du silicium à l'aluminium non supérieur à 1,33 (zéolite MAP).

4. Composition détergente selon l'une quelconque des revendications 1 à 3, dans laquelle ledit adjuvant zéolite est cristallin et comprend 10-28 % d'eau liée et est présent en une quantité de 15 % à 40 % en poids de la composition.

5. Composition détergente selon l'une quelconque des revendications 1 à 4, dans laquelle ladite enzyme amylase est une α -amylase.

6. Composition détergente selon l'une quelconque des revendications 1 à 6, dans laquelle ladite enzyme amylase est présente en une quantité de 0,1 % à 5 %, d'enzyme active en poids de la composition, sur une base d'activité de 60 KNU/g.

7. Composition détergente selon l'une quelconque des revendications 1 à 6, contenant un tensioactif en une quantité de 5 % à 50 % en poids de la composition.

8. Composition détergente selon l'une quelconque des revendications précédentes, dans laquelle ledit co-adjuvant comprend un silicate lamellaire cristallin ou un carboxylate monomère ou polymère.

9. Composition détergente selon l'une quelconque des revendications précédentes, contenant un système de blanchiment comprenant un composé de blanchiment peroxy et un précurseur de blanchiment peroxyacide.

10. Composition détergente selon la revendication 9, dans laquelle ledit composé de blanchiment peroxy est le percarbonate de sodium.

11. Composition détergente particulaire selon l'une quelconque des revendications précédentes, qui est une composition granulaire qui a une masse volumique apparente d'au moins 550 g/l.

12. Composition détergente particulaire selon l'une quelconque des revendications précédentes, ayant une masse volumique apparente d'au moins 700 g/l et préparée par densification postérieure en tour ou par pulvérisation d'une poudre sèche ou par mélange à sec et granulation.

13. Utilisation d'une enzyme amylase dans une composition de blanchissage particulaire pour réduire les résidus blancs sur des tissus soumis au lavage par blanchissage utilisant la composition, dans laquelle la composition contient un tensioactif comprenant un tensioactif anionique, un adjuvant zéolite ayant une taille particulaire, exprimée comme une valeur d_{50} , inférieure à 1,0 micromètre, et un co-adjuvant organique ou minéral.