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(54) BITTER PILL

(57) The present invention provides a high active laundry bitter pill treatment composition. Coated detergent particles having perpendicular dimensions x, y and z, the core comprises from 65 to 95 wt% of surfactant and adjuncts and is coated with from 5 to 30 wt % of sodium carbonate and from 20 and 1000 ppm of denatonium benzoate.

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Description

Field of Invention

[0001] The present invention relates to a concentrated solid laundry detergent composition.

Background of Invention

[0002] WO2014/026856 discloses a water-soluble packaging containing an agent and a water-soluble wrapping. Said water-soluble wrapping contains a bittering agent in a dilution of at least 1:250.

[0003] Bitrex® (Denatonium Benzoate) (CAS 3734-33-6) is the one of the bitterest substances known and is the leading branded safety ingredient used in a variety of domestic and industrial applications.

[0004] Denatonium Benzoate is supplied as a solid or in respective solutions of MEG, MPG ethanol and water.

[0005] We have found that the inclusion of Bitrex® in fragrance oils to be sprayed on a large detergent particle results in spray nozzles becoming blocked.

Summary of the Invention

[0006] We have found that it is possible to include Bitrex® in a near saturated sodium carbonate solution, within 10 wt% of sodium carbonate saturation, and spray the resulting solution on a large particle. The nature of the large particle is such that the spray will adhere to the surface of the particle and the aqueous element substantially evaporated in a fluidized bed to provide a coated large particle.

[0007] In one aspect the present invention provides a coated detergent particle with a core, the coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 0.5 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, the core comprising from 65 to 95 wt% of surfactant and adjuncts and coated with from 5 to 30 wt % of sodium carbonate and from 20 and 1000 ppm of denatonium benzoate, the coating applied as an aqueous solution spray and dried in a fluidized bed. The amounts wt % and ppm amounts described apply to the coated detergent particle as a whole.

[0008] The coating applied as an aqueous solution spray and dried in the fluidized bed. The coating is provided by spraying a sodium carbonate solution that is preferably from 80 to 100 percent, preferably 90 percent, of a saturated aqueous solution of sodium carbonate (as measured at 50 °C) and containing between 0.01 to 5 wt% of Bitrex solution. In the sodium carbonate coating per se the level of denatonium benzoate is from 200 to 10000 ppm on a dry basis. All amounts as described herein are on an anhydrous basis.

[0009] The dry basis is the coating formulation after discounting any moisture that may or may not have been removed in process in particular the fluidised bed.

[0010] Preferably the particulate detergent composition is and sodium tripoly phosphate (STP) free.

[0011] Unless otherwise stated all wt % refer to the total percentage in the particle as dry weights.

Detailed Description of the Invention

Large Particle (uncoated)

[0012] The large particle is preferably curved.

[0013] The large particle may be lenticular (shaped like a whole dried lentil), an oblate ellipsoid, where *z* and *y* are the equatorial diameters and *x* is the polar diameter; preferably y = z.

[0014] The large particle may be shaped as a disc.

[0015] Preferably the large particle does not have hole; that is to say, the coated laundry detergent particle does not have a conduit passing there though that passes through the core, i.e., the coated detergent particle has a topologic genus of zero.

[0016] The large particle has perpendicular dimensions x, y and z, wherein x is from 0.5 to 2 mm, y is from 2 to 8 mm, and z is from 2 to 8 mm. After coating with sodium carbonate and drying in a fluidised bed the coated large particle has approximately the same dimensions as the uncoated large particle. It is more preferred that the large particle contains detergent and other actives.

[0017] The inorganic salt is present on the detergent particle as a coating and the surfactant is present as a core. The inorganic coating reduces agglomeration and deterioration on storage.

[0018] The large particle before coating is preferable as described in any one of WO2013/139702; WO/2012/048950; WO/2012/048947; WO/2012/048949 WO/2012/048951; WO/2012/048948; WO/2012/049178; WO/2012/048926; WO/2012/048945; WO2013/149754; WO2013/149755; WO2013/149753; WO2013/149752; and, WO2014/048857.

[0019] Below are examples of preferred large particles that are coated:

The large particle core is preferably a particulate detergent composition having perpendicular dimensions x, y and z, wherein x is from 0.5 to 2 mm, y is from 2 to 8 mm, and z is from 2 to 8 mm, wherein the particle comprises:

(i) from 20 to 49 wt %, preferably 23 to 45 wt %, more preferably 36 to 42 wt %, of a surfactant selected from: anionic and non-ionic surfactants;

(ii) from 1 to 40 wt %, preferably 2 to 35 wt%, more preferably 3 to 20 wt%, of an active selected from: citric acid and sodium salts thereof;

[0020] The large particle is then coated with (v) from 5 to 40 wt %, preferably 5 to 30 wt %, of sodium carbonate and from 20 to 1000 ppm of denatonium benzoate on a dry basis.

[0021] The large particle core is preferably a particulate detergent composition having perpendicular dimensions x, y and z, wherein x is from 0.5 to 2 mm, y is from 2 to 8 mm, and z is from 2 to 8 mm, wherein the particle comprises:

(i) from 30 to 85 wt % of a surfactant selected from: anionic and non-ionic surfactants.

[0022] The large particle is then coated with (v) as described above

[0023] To exemplify the process the following is provided for manufacturing detergent particles which comprises the steps of:

a) forming a surfactant blend comprising integer (i);

b) drying the liquid surfactant blend of step (a) in an evaporator or drier to a moisture content of less than 5 wt% and cooling the output from the evaporator or dryer;

c) feeding the cooled material together with and integers (ii), (iii), and (iv); and extruding the cooled material to form an extrudate while periodically cutting the extrudate to form hard detergent particles with a diameter, of greater than 2 mm and a thickness of greater than 0.2 mm, provided that the diameter is greater than the thickness; and,

d) coating the extruded hard detergent particles with up to 40 wt% coating material.

CORE

SURFACTANT

[0024] In general, the nonionic and anionic surfactants of the surfactant system may be chosen from the surfactants described "Surface Active Agents" Vol. 1, by Schwartz & Perry, Interscience 1949, Vol. 2 by Schwartz, Perry & Berch, Interscience 1958, in the current edition of "McCutcheon's Emulsifiers and Detergents" published by Manufacturing Confectioners Company or in "Tenside-Taschenbuch", H. Stache, 2nd Edn., Carl Hauser Verlag, 1981. Preferably the surfactants used are saturated.

Anionic Surfactants

[0025] Suitable anionic detergent compounds which may be used are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher C_8 to C_{18} alcohols, produced for example from tallow or coconut oil, sodium and potassium alkyl C_9 to C_{20} benzene sulphonates, particularly sodium linear secondary alkyl C_{10} to C_{15} benzene sulphonates; and sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum. Most preferred anionic surfactants are sodium lauryl ether sulfate (SLES), particularly preferred with 1 to 3 ethoxy groups, sodium C_{10} to C_{15} alkyl benzene sulphonates and sodium C_{12} to C_{18} alkyl sulphates. Also applicable are surfactants such as those described in EP-A-328 177 (Unilever), which show resistance to salting-out, the alkyl polyglycoside surfactants described in EP-A-070 074, and alkyl monoglycosides. The chains of the surfactants may be branched or linear.

[0026] Soaps may also be present. The fatty acid soap used preferably contains from about 16 to about 22 carbon atoms, preferably in a straight chain configuration. The anionic contribution from soap is preferably from 0 to 30 wt % of the total anionic.

[0027] Preferably, at least 50 wt % of the anionic surfactant is selected from: sodium C_{11} to C_{15} alkyl benzene sulphonates; and, sodium C_{12} to C_{18} alkyl sulphates. Even more preferably, the anionic surfactant is sodium C_{11} to C_{15} alkyl benzene sulphonates.

Nonionic Surfactants

[0028] Suitable nonionic detergent compounds which may be used include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Preferred nonionic detergent compounds are C_6 to C_{22} alkyl phenol-ethylene oxide condensates, generally 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic C_8 to C_{18} primary or secondary linear or branched alcohols with ethylene oxide, generally 5 to 50 EO. Preferably, the non-ionic is 10 to 50 EO, more preferably 20 to 35 EO. Alkyl ethoxylates are particularly preferred.

[0029] Cationic surfactant may be present as minor ingredients at levels preferably between 0 to 5 wt % on total surfactant.

[0030] Preferably all the surfactants are mixed together before being dried. Conventional mixing equipment may be used. The surfactant core of the laundry detergent particle may be formed by extrusion or roller compaction and subsequently coated with an inorganic salt.

Calcium Tolerant Surfactant System

[0031] In another aspect the surfactant system used is calcium tolerant and this is a preferred aspect because this reduces the need for builder.

[0032] Surfactant blends that do not require builders to be present for effective detergency in hard water are preferred. Such blends are called calcium tolerant surfactant blends if they pass the test set out hereinafter. However, the invention may also be of use for washing with soft water, either naturally occurring or made using a water softener. In this case, calcium tolerance is no longer important and blends other than calcium tolerant ones may be used.

[0033] Calcium-tolerance of the surfactant blend is tested as follows:

> The surfactant blend in question is prepared at a concentration of 0.7 g surfactant solids per litre of water containing sufficient calcium ions to give a French hardness of 40 (4×10^{-3} Molar Ca^{2+}). Other hardness ion free electrolytes such as sodium chloride, sodium sulphate, and sodium hydroxide are added to the solution to adjust the ionic strength to 0.05M and the pH to 10. The adsorption of light of wavelength 540 nm through 4 mm of sample is measured 15 minutes after sample preparation. Ten measurements are made and an average value is calculated. Samples that give an absorption value of less than 0.08 are deemed to be calcium tolerant.

[0034] Examples of surfactant blends that satisfy the above test for calcium tolerance include those having a major part of LAS surfactant (which is not of itself calcium tolerant) blended with one or more other surfactants (co-surfactants) that are calcium tolerant to give a blend that is sufficiently calcium tolerant to be usable with little or no builder and to pass the given test. Suitable calcium tolerant co-surfactants include SLES 1-7EO, and alkyl-ethoxylate nonionic surfactants, particularly those with melting points less than 40°C.

[0035] A LAS/SLES surfactant blend has a superior foam profile to a LAS nonionic surfactant blend and is therefore preferred for hand washing formulations requiring high levels of foam. SLES may be used at levels of up to 30 wt % of the surfactant blend.

Citric Acid and Sodium Salts Thereof

[0036] The particulate detergent composition when dissolved in an aqueous medium is alkaline. In this alkaline solution there are sodium ions present and whilst there may not be any sodium citrate in the particulate detergent composition upon dissolution sodium citrate is generated in solution. It is preferred that sodium citrate is present in the particulate detergent composition.

Phosphonate Sequestrant

[0037] Phosphonate sequestrants are commercially available. Commercially available Dequest™ phosphonate sequestrants are particularly preferred.

[0038] The following are examples of Phosphonate sequestrants: (Dequest® 2010) 1-Hydroxyethylidene -1,1 ,-diphosphonic acid, (Dequest® 2066) Diethylenetriamine penta(methylene phosphonic acid), (Dequest® 7000) 2-

Phosphonobutane - 1,2,4 - tricarboxylic acid, (Dequest® 2066C2) Diethylenetriamine penta(methylene phosphonic acid), Dequest® 2047 ([ethylenebis[nitrilobis(methylene)]] tetrakisphosphonic acid, calcium sodium salt), Dequest® 2016 (1-Hydroxyethylene-1,1,-diphsophonic acid sodium salt, Na4HEDP). Dequest® 2016 and Dequest® 2047 are particularly preferred.

Soil Removal Polymer

[0039] Soil release polymers improve the main wash performance of the compositions when used in the low in wash surfactant process of the present invention.

[0040] One preferred class of polymer is the fabric-substantive polymers comprising at least one of (i) saccharide or (ii) dicarboxylic acid and polyol monomer units. Typically these have soil release properties and while they can have a primary detergency effect they generally assist in subsequent cleaning.

[0041] Generally the soil release polymers for polyester will comprise polymers of aromatic dicarboxylic acids and alkylene glycols (including polymers containing polyalkylene glycols).

[0042] The polymeric soil release agents useful herein especially include those soil release agents having:

(a) one or more nonionic hydrophilic components consisting essentially of:

(i) polyoxyethylene segments with a degree of polymerization of at least 2, or

(ii) oxypropylene or polyoxypropylene segments with a degree of polymerization of from 2 to 10, wherein said hydrophilic segment does not encompass any oxypropylene unit unless it is bonded to adjacent moieties at each end by ether linkages, or

(iii) a mixture of oxyalkylene units comprising oxyethylene and from 1 to about 30 oxypropylene units wherein said mixture contains a sufficient amount of oxyethylene units such that the hydrophilic component has hydrophilicity great enough to increase the hydrophilicity of conventional polyester synthetic fibre surfaces upon deposit of the soil release agent on such surface, said hydrophilic segments preferably comprising at least about 25% oxyethylene units and more preferably, especially for such components having about 20 to 30 oxypropylene units, at least about 50% oxyethylene units; or

(b) one or more hydrophobic components comprising:

(i) C_3 oxyalkylene terephthalate segments, wherein, if said hydrophobic components also comprise oxyethylene terephthalate, the ratio of oxyethylene terephthalate: C_3 oxyalkylene terephthalate units is about 2:1 or lower,

(ii) C_4-C_6 alkylene or oxy C_4-C_6 alkylene segments, or mixtures therein,

(iii) poly (vinyl ester) segments, preferably polyvinyl acetate), having a degree of polymerization of at least 2, or (iv) C_1-C_4 alkyl ether or C_4 hydroxyalkyl ether substituents, or mixtures therein, wherein said substituents are present in the form of C_1-C_4 alkyl ether or C_4 hydroxyalkyl ether cellulose derivatives, or mixtures therein, and such cellulose derivatives are amphiphilic, whereby they have a sufficient level of C_1-C_4 alkyl ether and/or C_4 hydroxyalkyl ether units to deposit upon conventional polyester synthetic fibre surfaces and retain a sufficient level of hydroxyls, once adhered to such conventional synthetic fibre surface, to increase fibre surface hydrophilicity, or a combination of (a) and (b).

[0043] Typically, the polyoxyethylene segments of (a)(i) will have a degree of polymerization of from about 200, although higher levels can be used, preferably from 3 to about 150, more preferably from 6 to about 100. Suitable oxy C_4-C_6 alkylene hydrophobic segments include, but are not limited to, end-caps of polymeric soil release agents such as $MO_3S(CH_2)_nOCH_2CH_2O$--, where M is sodium and n is an integer from 4-6, as disclosed in U.S. Pat. No. 4,721,580, issued Jan. 26, 1988 to Gosselink.

[0044] Soil release agents characterized by poly(vinyl ester) hydrophobic segments include graft copolymers of poly(vinyl ester), e.g., C_1-C_6 vinyl esters, preferably poly(vinyl acetate) grafted onto polyalkylene oxide backbones, such as polyethylene oxide backbones. See European Patent Application 0219 048, published Apr. 22, 1987 by Kud, et al. Commercially available soil release agents of this kind include the SOKALAN type of material, e.g., SOKALAN HP-22, available from BASF (West Germany).

[0045] One type of preferred soil release agent is a copolymer having random blocks of ethylene terephthalate and polyethylene oxide (PEO) terephthalate. The molecular weight of this polymeric soil release agent is in the range of from about 25,000 to about 55,000. See U.S. Pat. No. 3,959,230 to Hays, issued May 25, 1976 and U.S. Pat. No. 3,893,929 to Basadur issued Jul. 8, 1975.

[0046] Another preferred polymeric soil release agent is a polyester with repeat units of ethylene terephthalate units contains 10 to 15 wt% of ethylene terephthalate units together with 90 to 80 wt% weight of polyoxyethylene terephthalate units, derived from a polyoxyethylene glycol of average molecular weight 300-5,000. Examples of this polymer include the commercially available material ZELCON 5126 (from DuPont) and MILEASE T (from ICI). See also U.S. Pat. No. 4,702,857, issued Oct. 27, 1987 to Gosselink.

[0047] Another preferred polymeric soil release agent is a sulphonated product of a substantially linear ester oligomer comprised of an oligomeric ester backbone of terephthaloyl and oxyalkyleneoxy repeat units and terminal moieties covalently attached to the backbone. These soil release agents are described fully in U.S. Pat. No. 4,968,451, issued Nov. 6, 1990 to J.J. Scheibel and E. P. Gosselink. Other suitable polymeric soil release agents include the terephthalate polyesters of U.S. Pat. No. 4,711,730, issued Dec. 8, 1987 to Gosselink et al, the anionic end-capped oligomeric esters of U.S. Pat. No. 4,721,580, issued Jan. 26, 1988 to Gosselink, and the block polyester oligomeric compounds of U.S. Pat. No. 4,702,857, issued Oct. 27, 1987 to Gosselink.

[0048] Preferred polymeric soil release agents also include the soil release agents of U.S. Pat. No. 4,877,896, issued Oct. 31, 1989 to Maldonado et al, which discloses anionic, especially sulfoarolyl, end-capped terephthalate esters.

[0049] Still another preferred soil release agent is an oligomer with repeat units of terephthaloyl units, sulfoisoterephthaloyl units, oxyethyleneoxy and oxy-1,2-propylene units. The repeat units form the backbone of the oligomer and are preferably terminated with modified isethionate end-caps. A particularly preferred soil release agent of this type comprises about one sulfoisophthaloyl unit, 5 terephthaloyl units, oxyethyleneoxy and oxy-1,2-propyleneoxy units in a ratio of from about 1.7 to about 1.8, and two end-cap units of sodium 2-(2-hydroxyethoxy)-ethanesulphonate. Said soil release agent also comprises from about 0.5% to about 20%, by weight of the oligomer, of a crystalline-reducing stabilizer, preferably selected from the group consisting of xylene sulfonate, cumene sulfonate, toluene sulfonate, and mixtures thereof.

[0050] The most preferred soil release polymers are the water soluble/miscible or dispersible polyesters such as: linear polyesters sold under the Repel-O-Tex brand by Rhodia (Gerol), or the Texcare brand by Clariant, especially Texcare SRN170, and heavily branched polyesters such as those available from Sasol and described in US 7119056. The polyesters are preferably nonionic and comprise a mid block of spaced apart terephthalate repeat units and at least one end block based on polyethylene glycol with a lower alkyl or hydrogen termination.

Inorganic salts:

[0051] It will be appreciated by those skilled in the art that while multiple layered coatings, of the same or different coating materials, could be applied, a single coating layer is preferred, for simplicity of operation, and to maximise the thickness of the coating. The amount of coating should lay in the range 1 to 40 wt% of the particle, preferably 4 to 30 wt%, more preferably 5 to 25 wt% for the best results in terms of anti-caking properties of the detergent particles.

[0052] The coating is preferably applied to the surface of the surfactant core, by deposition from an aqueous solution of the water soluble inorganic salt. In the alternative coating can be performed using a slurry. The aqueous solution preferably contains greater than 50g/L, more preferably 200 g/L of the salt. An aqueous spray-on of the coating solution in a fluidised bed has been found to give good results and may also generate a slight rounding of the detergent particles during the fluidisation process. Drying and/or cooling may be needed to finish the process.

The coated laundry detergent particle

[0053] Preferably, the coated laundry detergent particle comprises from 10 to 100 wt %, more preferably 50 to 100 wt %, even more preferably 80 to 100 wt %, most preferably 90 to 100 wt % of a laundry detergent formulation in a package.

[0054] The package is that of a commercial formulation for sale to the general public and is preferably in the range of 0.01 kg to 5 kg, preferably 0.02 kg to 2 kg, most preferably 0.5 kg to 2 kg.

[0055] Preferably, the coated laundry detergent particle is such that at least 90 to 100 % of the coated laundry detergent particles in the in the x, y and z dimensions are within a 20 %, preferably 10%, variable from the largest to the smallest coated laundry detergent particle.

Water content

[0056] The particle preferably comprises from 0 to 15 wt % water, more preferably 0 to 10 wt %, most preferably from 1 to 5 wt % water, at 293K and 50% relative humidity. This facilitates the storage stability of the particle and its mechanical properties.

Other Adjuncts

[0057] The adjuncts as described herein may be present in the coating.

Fluorescent Agent

[0058] The coated laundry detergent particle preferably comprises a fluorescent agent (optical brightener). Fluorescent agents are well known and many such fluorescent agents are available commercially. Usually, these fluorescent agents are supplied and used in the form of their alkali metal salts, for example, the sodium salts. The total amount of the fluorescent agent or agents used in the composition is generally from 0.005 to 2 wt %, more preferably 0.01 to 0.1 wt %. Suitable Fluorescer for use in the invention are described in chapter 7 of Industrial Dyes edited by K.Hunger 2003 Wiley-VCH ISBN 3-527-30426-6.

[0059] Preferred fluorescers are selected from the classes distyrylbiphenyls, triazinylaminostilbenes, bis(1,2,3-triazol-2-yl)stilbenes, bis(benzo[b]furan-2-yl)biphenyls, 1,3-diphenyl-2-pyrazolines and courmarins. The fluorescer is preferably sulfonated.

[0060] Preferred classes of fluorescer are: Di-styryl biphenyl compounds, e.g. Tinopal (Trade Mark) CBS-X, Di-amine stilbene di-sulphonic acid compounds, e.g. Tinopal DMS pure Xtra and Blankophor (Trade Mark) HRH, and Pyrazoline compounds, e.g. Blankophor SN. Preferred fluorescers are: sodium 2 (4-styryl-3-sulfophenyl)-2H-napthol[1,2-d]triazole, disodium 4,4'-bis{[(4-anilino-6-(N methyl-N-2 hydroxyethyl) amino 1,3,5-triazin-2-yl)]amino}stilbene-2-2' disulfonate, disodium 4,4'-bis{[(4-anilino-6-morpholino-1 ,3,5-triazin-2-yl)]amino} stilbene-2-2' disulfonate, and disodium 4,4'-bis(2-sulfostyryl)biphenyl. Tinopal® DMS is the disodium salt of disodium 4,4'-bis{[(4-anilino-6-morpholino-1,3,5-triazin-2-yl)]amino} stilbene-2-2' disulfonate. Tinopal® CBS is the disodium salt of disodium 4,4'-bis(2-sulfostyryl)biphenyl.

Perfume

[0061] Preferably the composition comprises a perfume. The perfume is preferably in the range from 0.001 to 3 wt %, most preferably 0.1 to 1 wt %. Many suitable examples of perfumes are provided in the CTFA (Cosmetic, Toiletry and Fragrance Association) 1992 International Buyers Guide, published by CFTA Publications and OPD 1993 Chemicals Buyers Directory 80th Annual Edition, published by Schnell Publishing Co.

[0062] It is commonplace for a plurality of perfume components to be present in a formulation. In the compositions of the present invention it is envisaged that there will be four or more, preferably five or more, more preferably six or more or even seven or more different perfume components.

[0063] In perfume mixtures preferably 15 to 25 wt% are top notes. Top notes are defined by Poucher (Journal of the Society of Cosmetic Chemists 6(2):80 [1955]). Preferred top-notes are selected from citrus oils, linalool, linalyl acetate, lavender, dihydromyrcenol, rose oxide and cis-3-hexanol.

Polymers

[0064] The composition may comprise one or more further polymers. Examples are carboxymethylcellulose, poly (ethylene glycol), poly(vinyl alcohol), polyethylene imines, ethoxylated polyethylene imines, water soluble polyester polymers polycarboxylates such as polyacrylates, maleic/acrylic acid copolymers and lauryl methacrylate/acrylic acid copolymers.

Enzymes

[0065] One or more enzymes are preferred present in a composition of the invention.

[0066] Preferably the level of each enzyme is from 0.0001 wt% to 0.5 wt% protein on product.

[0067] Especially contemplated enzymes include proteases, alpha-amylases, cellulases, lipases, peroxidases/oxidases, pectate lyases, and mannanases, or mixtures thereof.

[0068] Suitable lipases include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Examples of useful lipases include lipases from *Humicola* (synonym *Thermomyces*), e.g. from *H. lanuginosa* (*T. lanuginosus*) as described in EP 258 068 and EP 305 216 or from H. *insolens* as described in WO 96/13580, a *Pseudomonas* lipase, e.g. from *P. alcaligenes* or *P. pseudoalcaligenes* (EP 218 272), *P. cepacia* (EP 331 376), *P. stutzeri* (GB 1,372,034), *P. fluorescens, Pseudomonas* sp. strain SD 705 (WO 95/06720 and WO 96/27002), *P. wisconsinensis* (WO 96/12012), a *Bacillus* lipase, e.g. from *B. subtilis* (Dartois et al. (1993), Biochemica et Biophysica Acta, 1131, 253-360), *B. stearothermophilus* (JP 64/744992) or *B. pumilus* (WO 91/16422).

[0069] Other examples are lipase variants such as those described in WO 92/05249, WO 94/01541, EP 407 225, EP 260 105, WO 95/35381, WO 96/00292, WO 95/30744, WO 94/25578, WO 95/14783, WO 95/22615, WO 97/04079 and

WO 97/07202, WO 00/60063, WO 09/107091 and WO09/111258.

[0070] Preferred commercially available lipase enzymes include Lipolase™ and Lipolase Ultra™ , Lipex™ (Novozymes A/S) and Lipoclean™.

[0071] The method of the invention may be carried out in the presence of phospholipase classified as EC 3.1.1.4 and/or EC 3.1.1.32. As used herein, the term phospholipase is an enzyme which has activity towards phospholipids.

[0072] Phospholipids, such as lecithin or phosphatidylcholine, consist of glycerol esterified with two fatty acids in an outer (sn-1) and the middle (sn-2) positions and esterified with phosphoric acid in the third position; the phosphoric acid, in turn, may be esterified to an amino-alcohol. Phospholipases are enzymes which participate in the hydrolysis of phospholipids. Several types of phospholipase activity can be distinguished, including phospholipases A_1 and A_2 which hydrolyze one fatty acyl group (in the sn-1 and sn-2 position, respectively) to form lysophospholipid; and lysophospholipase (or phospholipase B) which can hydrolyze the remaining fatty acyl group in lysophospholipid. Phospholipase C and phospholipase D (phosphodiesterases) release diacyl glycerol or phosphatidic acid respectively.

[0073] Suitable proteases include those of animal, vegetable or microbial origin. Microbial origin is preferred. Chemically modified or protein engineered mutants are included. The protease may be a serine protease or a metallo protease, preferably an alkaline microbial protease or a trypsin-like protease. Preferred commercially available protease enzymes include Alcalase™, Savinase™, Primase™, Duralase™, Dyrazym™, Esperase™, Everlase™, Polarzyme™, and Kannase™, (Novozymes A/S), Maxatase™, Maxacal™, Maxapem™, Properase™, Purafect™, Purafect OxP™, FN2™, and FN3™ (Genencor International Inc.).

[0074] The method of the invention may be carried out in the presence of cutinase, classified in EC 3.1.1.74. The cutinase used according to the invention may be of any origin.

[0075] Preferably cutinases are of microbial origin, in particular of bacterial, of fungal or of yeast origin.

[0076] Suitable amylases (alpha and/or beta) include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Amylases include, for example, alpha-amylases obtained from *Bacillus,* e.g. a special strain of *B. licheniformis,* described in more detail in GB 1,296,839, or the *Bacillus* sp. strains disclosed in WO 95/026397 or WO 00/060060. Commercially available amylases are Duramyl™, Termamyl™, Termamyl Ultra™, Natalase™, Stainzyme™, Fungamyl™ and BAN™ (Novozymes A/S), Rapidase™ and Purastar™ (from Genencor International Inc.).

[0077] Suitable cellulases include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Suitable cellulases include cellulases from the genera *Bacillus, Pseudomonas, Humicola, Fusarium, Thielavia, Acremonium,* e.g. the fungal cellulases produced from *Humicola insolens, Thielavia terrestris, Myceliophthora thermophila,* and *Fusarium oxysporum* disclosed in US 4,435,307, US 5,648,263, US 5,691,178, US 5,776,757, WO 89/09259, WO 96/029397, and WO 98/012307. Commercially available cellulases include Celluzyme™, Carezyme™, Endolase™, Renozyme™ (Novozymes A/S), Clazinase™ and Puradax HA™ (Genencor International Inc.), and KAC-500(B)™ (Kao Corporation).

[0078] Suitable peroxidases/oxidases include those of plant, bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Examples of useful peroxidases include peroxidases from *Coprinus,* e.g. from *C. cinereus,* and variants thereof as those described in WO 93/24618, WO 95/10602, and WO 98/15257. Commercially available peroxidases include Guardzyme™ and Novozym™ 51004 (Novozymes A/S).

[0079] Further enzymes suitable for use are disclosed in WO2009/087524, WO2009/090576, WO2009/148983 and WO2008/007318.

[0080] We have found that the composition of the invention serves to boost enzyme cleaning. We have noticed that proteases, e.g., Savinase™ , and lipases, e.g., Lipex™, are particularly boosted in efficacy. The boosting of the enzyme efficacy is ascribed to the high citrate, sequestrate levels.

Enzyme Stabilizers

[0081] Any enzyme present in the composition may be stabilized using conventional stabilizing agents, e.g., a polyol such as propylene glycol or glycerol, a sugar or sugar alcohol, lactic acid, boric acid, or a boric acid derivative, e.g., an aromatic borate ester, or a phenyl boronic acid derivative such as 4-formylphenyl boronic acid, and the composition may be formulated as described in e.g. WO 92/19709 and WO 92/19708.

[0082] Where alkyl groups are sufficiently long to form branched or cyclic chains, the alkyl groups encompass branched, cyclic and linear alkyl chains. The alkyl groups are preferably linear or branched, most preferably linear.

[0083] The indefinite article "a" or "an" and its corresponding definite article "the" as used herein means at least one, or one or more, unless specified otherwise. The singular encompasses the plural unless otherwise specified.

[0084] Sequesterant may be present in the coated laundry detergent particles.

Experimental

Trial I

[0085] Taking the coated product of the formulation, in table x below, 100, 500 and 1000 PPM Bitrex, of the total composition, were added. To ensure the maximum impact of the flavour of the Bitrex it was added to the fragrance oil that is sprayed on as the last processing step.

Ingredient	**Trial 1 (g)**	**Trial 2 (g)**	**Trial 3 (g)**
NaLAS	209	20.9	20.9
Non-ionic surfactant-C12-C15-alkyl ethoxylate 30 EO	37	37	37
CP5 (sodium salt of an acrylic acid maleic acid copolymer)	30	30	30
Phosphonate Sequestrant	55	55	55
Citric Acid	246	246	246
Enzymes	30	30	30
Soil release polymer	20	20	20
Silicon Antifoam	5	5	5
Sodium carbonate	358	358	358
Fragrance	11	11	11
Bitrex	0.1	0.5	1
TOTAL	**1000**	**1000**	**1000**

[0086] The Bitrex was added to the fragrance, mixed for 10 minutes and the combination sprayed onto the remaining product. In all cases the spray nozzle blocked before the fragrance addition was complete.

Trial 2

[0087] Taking the uncoated product of the formulation, in the table below, 100, 500 and 1000 PPM of Bitrex, of the total composition were added into the part of the sodium carbonate solution to be added in the fluid bed.

Ingredient	**Trial 1 (g)**	**Trial 2 (g)**	**Trial 3 (g)**
NaLAS	209	209	209
Non-ionic surfactant-C12-C15-alkyl ethoxylate 30 EO	37	37	37
CP5 (sodium salt of an acrylic acid maleic acid copolymer)	30	30	30
Phosphonate Sequesterant	55	55	55
Citric Acid	246	246	246
Enzymes	30	30	30
Soil release polymer	20	20	20
Silicon Antifoam	5	5	5
Sodium carbonate	300	300	300
Sodium carbonate (coating)	58	58	58
Bitrex	0.1	0.5	1
Fragrance (spray on)	11	11	11

(continued)

Ingredient	Trial 1 (g)	Trial 2 (g)	Trial 3 (g)
TOTAL	**1000**	**1000**	**1000**

[0088] The base particle was added into the Glatt Fluid Bed coater. The Bitrex was added to the concentrated sodium carbonate solution. The following conditions were used for the coating

Air Inlet temperature	80°C
Bed Temperature	45°C
Sodium Carbonate spray temperature	50°C
Air Flow rate	2.5 m/s

[0089] The spray nozzle was not blocked by the Bitrex concentrated sodium carbonate solution.

[0090] After the coating step the fragrance oil was added to the coated product as a spray.

Example Composition

[0091]

Ingredient	% Inclusion
NaLAS	20.9
Non-ionic surfactant-C12-C15-alkyl ethoxylate 30 EO	3.7
CP5 (sodium salt of an acrylic acid maleic acid copolymer)	3.0
Phosphonate Sequestrant	5.5
Citric Acid	24.6
Enzymes	3.0
Soil release polymer	2.0
Silicon Antifoam	0.5
Sodium carbonate	35.8
Fragrance	1.1
TOTAL	**100.0**

Example Composition

[0092]

Component	[%]
LAS/30 EO Base	40.3%
Dequest 2016	7.7%
Citric acid	10.6%
Na Citrate	32.3%
enzyme	3.5%
Soil Release Polymer	2.8%
Fragrance	1.4%
Moisture	1.4%

(continued)

Component	[%]
TOTAL	100.0%

Claims

1. A coated detergent particle with a core, the coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 0.5 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, the core comprising from 65 to 95 wt% of surfactant and adjuncts and coated with from 5 to 30 wt % of sodium carbonate and from 20 and 1000 ppm of denatonium benzoate, the coating applied as an aqueous solution spray and dried in a fluidized bed.

2. A coated detergent particle according to claim 1, wherein the core is a particulate detergent composition having perpendicular dimensions x, y and z, wherein x is from 0.5 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein the particle comprises:

   (i) from 20 to 39 wt %, preferably 23 to 35 wt %, more preferably 26 to 32 wt %, of a surfactant selected from: anionic and non-ionic surfactants;
   (ii) from 15 to 40 wt %, preferably 20 to 35 wt%, more preferably 25 to 30 wt%, of an active selected from: citric acid and sodium salts thereof;
   (iii) from 2 to 8 wt %, preferably 3 to 6 wt%, of a phosphonate sequestrant; and,
   (iv) from 0 to 3 wt %, preferably 1 to 2.5 wt%, of a soil removal polymer.

3. A coated detergent particle according to claim 1, wherein the core is a particulate detergent composition having perpendicular dimensions x, y and z, wherein x is from 0.5 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein the particle comprises from 20 to 39 wt % of a surfactant selected from: anionic and non-ionic surfactants.

4. A coated detergent particle according to claim 1 or 2, wherein the total level of denatonium benzoate in the coated detergent particle is in the range from 20 to 1000 ppm of which from 80 to 100 wt% is in the sodium carbonate coating.

5. A coated detergent particle according to claim 1 or 2, wherein the inorganic salt comprises sodium carbonate in the range from 10 to 100% sodium carbonate.

6. A coated detergent particle according to any one of the preceding claims, wherein the particle comprises from 0 to 15 wt % water.

7. A coated detergent particle composition according to claim 6, wherein the particle comprises from 1 to 5 wt % water.

8. A coated detergent particle according to any one of the preceding claims, wherein the coating is applied as a sodium carbonate solution that is from 80 to 100 percent of a saturated aqueous solution of sodium carbonate, as measured at 50 °C, and containing between 0.01 to 5 wt% of Bitrex in solution.

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EUROPEAN SEARCH REPORT

Application Number
EP 16 15 0474

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The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	29 June 2016	Pfannenstein, Heide

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 16 15 0474

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