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(54) **Laundry detergent particles**

(57) The present invention provides lenticular or disc detergent particle.

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Description

Field of Invention

5 **[0001]** The present invention relates to large laundry detergent particles.

Background of Invention

10 **[0002]** WO9932599 describes a method of manufacturing laundry detergent particles, being an extrusion method in which a builder and surfactant, the latter comprising as a major component a sulphated or sulphonated anionic surfactant, are fed into an extruder, mechanically worked at a temperature of at least 40°C, preferably at least 60°C, and extruded through an extrusion head having a multiplicity of extrusion apertures. In most examples, the surfactant is fed to the extruder along with builder in a weight ratio of more than 1 part builder to 2 parts surfactant. The extrudate apparently required further drying. In Example 6, PAS paste was dried and extruded. Such PAS noodles are well known in the prior art. The noodles are typically cylindrical in shape and their length exceeds their diameter, as described in example 2.

15 **[0003]** US 7,022,660 discloses a process for the preparation of a detergent particle having a coating.

Summary of the Invention

20 **[0004]** We have found that it is possible to have a highly reflective coated detergent particle that is aesthetically pleasing to the eye; the coating contains citric acid. The reflective coated detergent particle also gives the appearance of luxurious moisture. In a further aspect the coating increases the colour intensity when a dye or pigment is incorporated into the coating over that of similar inorganic coatings. A further advantage is that most dyes will be stable in the citric acid coating. Further in the absence of carbonate in a detergent formulation the coated detergent particle provides foaming

25 in hard water environments.

[0005] In a further aspect, the present invention provides a coated detergent particle that is a concentrated formulation with more surfactant than inorganic solid. Only by having the coating encasing the surfactant which is soft can one have such a particulate concentrate where the unit dose required for a wash is reduced. Adding solvent to the core would result by converting the particle into a liquid formulation. On the other hand, having a greater amount of inorganic solid would result in a less concentrated formulation; a high inorganic content would take one back to conventional low surfactant concentration granular powder. The coated detergent particle of the present invention sits in the middle of the two conventional (liquid and granular) formats.

30 **[0006]** In one aspect the present invention provides a coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm (preferably 3 to 8 mm), and z is from 2 to 8 mm (preferably 3 to 8 mm), wherein the particle comprises:

- (i) from 40 to 90 wt %, preferably 50 to 90 wt%, surfactant selected from: anionic surfactant; and, non-ionic surfactant;
- (ii) from 1 to 40 wt % citric acid, preferably 10 to 35 wt%, more preferably 20 to 35 wt %; and,
- (iii) from 0 to 3 wt %, preferably 0.001 to 3 wt % of a perfume,

40 wherein the citric acid is present on the laundry detergent particle as a coating and the surfactant is present as a core.

[0007] Unless otherwise stated all wt % refer to the total percentage in the particle as dry weights.

Detailed Description of the Invention

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SHAPE

[0008] Preferably the coated laundry detergent particle is curved.

50 **[0009]** The coated laundry detergent 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.

[0010] The coated laundry detergent particle may be shaped as a disc.

[0011] Preferably the coated laundry detergent 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.

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CORESURFACTANT

5 **[0012]** The coated laundry detergent particle comprises between 50 to 90 wt% of a surfactant, most preferably 70 to 90 wt %. 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
10 surfactants used are saturated.

Anionic Surfactants

15 **[0013]** Suitable anionic detergent compounds which may be used are usually watersoluble 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 C₈ to C₁₈ alcohols, produced for example from tallow or coconut oil, sodium and potassium alkyl C₉ to C₂₀ benzene sulphonates, particularly sodium linear secondary alkyl C₁₀ to C₁₅ benzene sulphonates; and sodium alkyl glyceryl ether sulphates, especially those
20 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 C₁₀ to C₁₅ alkyl benzene sulphonates and sodium C₁₂ to C₁₈ 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
25 linear.

[0014] 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.

30 **[0015]** Preferably, at least 50 wt % of the anionic surfactant is selected from: sodium C₁₁ to C₁₅ alkyl benzene sulphonates; and, sodium C₁₂ to C₁₈ alkyl sulphates. Even more preferably, the anionic surfactant is sodium C₁₁ to C₁₅ alkyl benzene sulphonates.

[0016] Preferably the anionic surfactant is present in the coated laundry detergent particle at levels between 15 to 85 wt %, more preferably 50 to 80 wt % on total surfactant.

Nonionic Surfactants

35 **[0017]** 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
40 detergent compounds are C₆ to C₂₂ 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 C₈ to C₁₈ 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.

45 **[0018]** Preferably the nonionic surfactant is present in the coated laundry detergent particle at levels between 5 to 75 wt % on total surfactant, more preferably 10 to 40 wt% on total surfactant.

[0019] Cationic surfactant may be present as minor ingredients at levels preferably between 0 to 5 wt % on total surfactant.

50 **[0020]** 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

55 **[0021]** In another aspect the surfactant system used is calcium tolerant and this is a preferred aspect because this reduces the need for builder.

[0022] 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.

[0023] 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.

[0024] 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.

[0025] 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

[0026] The coating is preferably applied to the surface of the surfactant core, by deposition from an aqueous solution of citric acid. In the alternative coating can be performed using a slurry. The aqueous solution preferably contains greater than 300g/L, more preferably 500 g/L, of the citric acid. 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.

[0027] A preferred calcium tolerant coated laundry detergent particle comprises 15 to 100 wt% on surfactant of anionic surfactant of which 20 to 30 wt% on surfactant is sodium lauryl ether sulphate.

DYE and PIGMENT

[0028] Dyes are described in Industrial Dyes edited by K.Hunger 2003 Wiley-VCH ISBN 3-527-30426-6.

[0029] Pigments are described in Industrial Inorganic Pigments edited by G. Buxbaum and G. Pfaff (3rd edition Wiley-VCH 2005). Suitable organic pigments are described in Industrial Organic Pigments edited by W. Herbst and K.Hunger (3rd edition Wiley-VCH 2004). Pigments are listed in the colour index international © Society of Dyers and Colourists and American Association of Textile Chemists and Colorists 2002.

[0030] Pigments are practically insoluble coloured particles, preferably they have a primary particle size of 0.02 to 10µm, where the distance represent the longest dimension of the primary particle. The primary particle size is measured by scanning electron microscopy. Most preferably the organic pigments have a primary particle size between 0.02 and 0.2 µm.

[0031] By practically insoluble we mean having a water solubility of less than 500 part per trillion (ppt), preferably 10 ppt at 20°C with a 10 wt% surfactant solution.

[0032] Any class of dye and pigment may be used in the citrate coating.

[0033] Preferred dyes for use in the current invention are selected from anionic and non-ionic dyes Anionic dyes are negatively charged in an aqueous medium at pH 7. Examples of anionic dyes are found in the classes of acid and direct dyes in the Color Index (Society of Dyers and Colourists and American Association of Textile Chemists and Colorists). Anionic dyes preferably contain at least one sulphonate or carboxylate groups. Non-ionic dyes are uncharged in an aqueous medium at pH 7, examples are found in the class of disperse dyes in the Color Index.

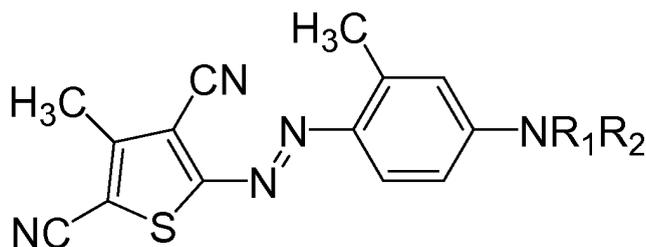
[0034] The dyes may be alkoxyated. Alkoxyated dyes are preferably of the following generic form: Dye-NR₁R₂. The NR₁R₂ group is attached to an aromatic ring of the dye. R₁ and R₂ are independently selected from polyoxyalkylene chains having 2 or more repeating units and preferably having 2 to 20 repeating units. Examples of polyoxyalkylene chains include ethylene oxide, propylene oxide, glycidol oxide, butylene oxide and mixtures thereof.

[0035] A preferred polyoxyalkylene chain is $[(\text{CH}_2\text{CR}_3\text{HO})_x(\text{CH}_2\text{CR}_4\text{HO})_y\text{R}_5]$ in which $x+y \leq 5$ wherein $y \geq 1$ and $z = 0$ to 5, R₃ is selected from: H; CH₃; CH₂O(CH₂CH₂O)_zH and mixtures thereof; R₄ is selected from: H; CH₂O(CH₂CH₂O)_zH and mixtures thereof; and, R₅ is selected from: H; and, CH₃.

[0036] A preferred alkoxyated dye for use in the invention, which also functions as a shading dye, is:

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[0037] Preferably the dye is selected from acid dyes; disperse dyes and alkoxyated dyes.

[0038] Most preferably the colourant is a pigment, even more preferably an organic pigment.

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[0039] Pigments are coloured particles, preferably they have a primary particle size of 0.02 to 10 μ m, where the distance represent the longest dimension of the primary particle. The primary particle size is measured by scanning electron microscopy. Most preferably the organic pigments have a primary particle size between 0.02 and 0.2 μ m.

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[0040] Suitable organic pigments are preferably selected from monoazo pigments, beta-naphthol pigments, naphthol AS pigments, azo pigment lakes, benzimidazolone pigments, metal complex pigments, isoindolinone and isoindoline pigments, phthalocyanine pigments, quinacridone pigments, perylene and perinone pigments, diketopyrrolo-pyrrole pigments, thioindigo pigments, anthraquinone pigments, anthrapyrimidine pigments, flavanthrone pigments, anthanthrone pigments, dioxazine pigments and quinophthalone pigments, most preferably dioxazine and phthalocyanine pigments.

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[0041] Preferred pigments are pigment green 8, pigment yellow 1, pigment yellow 3, pigment orange 1, pigment red 4, pigment red 3, pigment red 22, pigment red 112, pigment red 7, pigment brown 1, pigment red 5, pigment red 68, pigment red 51, pigment 53, pigment red 53:1, pigment red 49, pigment red 49:1, pigment red 49:2, pigment red 49:3, pigment red 64:1, pigment red 57, pigment red 57:1, pigment red 48, pigment red 63:1, pigment yellow 16, pigment yellow 12, pigment yellow 13, pigment yellow 83, pigment orange 13, pigment violet 23, pigment red 83, pigment blue 60, pigment blue 64, pigment orange 43, pigment blue 66, pigment blue 63, pigment violet 36, pigment violet 19, pigment red 122, pigment blue 16, pigment blue 15, pigment blue 15:1, pigment blue 15:2, pigment blue 15:3, pigment blue 15:4, pigment blue 15:6, pigment green 7, pigment green 36, pigment blue 29, pigment green 24, pigment red 101:1, pigment green 17, pigment green 18, pigment green 14, pigment brown 6, pigment blue 27 and pigment violet 16. Most preferably the pigment is selected from pigment blue 15, pigment blue 15:1, pigment blue 15:2, pigment blue 15:3, pigment blue 15:4, pigment blue 15:6.

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[0042] Pigments are preferably present from 0.001 to 0.1wt%.

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[0043] The dye and/or pigment is added to the coating solution or slurry and agitated before applying to the core of the particle. Application may be by any suitable method, preferably spraying on to the core particle as detailed above.

[0044] The dye and/or pigment may be any colour, preferable the dye is blue, violet, green or red. Most preferably the dye and/or pigment is blue or violet.

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[0045] The dye is preferably a shading dye for imparting a perception of whiteness to a laundry textile, preferably acid violet 50, solvent violet 13, disperse violet 27, disperse violet 28, an alkoxyated thiophene, or a cationic phenazine as described in WO 2009/141172 and WO 2009/141173. When a shading dye is present, preferably a further green dye is present to shift the colour of the particle from violet to blue-green.

[0046] The dye may be covalently bound to polymeric species.

[0047] A combination of dyes or pigments may be used.

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[0048] The dye or pigment may also be incorporated into the core.

The coated laundry detergent particle

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[0049] 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.

[0050] 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.

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[0051] 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

[0052] 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

[0053] The adjuncts as described below may be present in the coating or the core. These may be in the core or the coating.

Fluorescent Agent

[0054] 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.

[0055] 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 coumarins. The fluorescer is preferably sulfonated.

[0056] 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-sulfohenyl)-2H-naphthol[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)]aminol}stilbene-2-2' disulfonate, and disodium 4,4'-bis(2-sulfostyryl)biphenyl.

[0057] 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

[0058] 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.

[0059] 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.

[0060] 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.

[0061] The perfume serves to disaggregate the dye to make the dye more visible.

[0062] It is preferred that the coated laundry detergent particle does not contain a peroxygen bleach, e.g., sodium percarbonate, sodium perborate, and peracid.

Polymers

[0063] 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

[0064] One or more enzymes are preferred present in a composition of the invention.

[0065] Preferably the level of each enzyme is from 0.0001 wt% to 0.5 wt% protein on product.

[0066] Especially contemplated enzymes include proteases, alpha-amylases, cellulases, lipases, peroxidases/oxidases, pectate lyases, and mannanases, or mixtures thereof.

[0067] 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).

[0068] 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 W009/111258.

[0069] Preferred commercially available lipase enzymes include Lipolase™ and Lipolase Ultra™, Lipex™ (Novozymes A/S) and Lipoclean™.

[0070] 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.

[0071] 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 A₁ and A₂ 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.

[0072] 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™, Dyrzym™, Esperase™, Everlase™, Polarzyme™, and Kan-nase™, (Novozymes A/S), Maxatase™, Maxacal™, Maxapem™, Properase™, Purafect™, Purafect OxP™, FN2™, and FN3™ (Genencor International Inc.).

[0073] 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. Preferably cutinases are of microbial origin, in particular of bacterial, of fungal or of yeast origin.

[0074] 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.).

[0075] 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).

[0076] 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).

[0077] Further enzymes suitable for use are disclosed in W02009/087524, W02009/090576, W02009/148983 and W02008/007318.

Enzyme Stabilizers

[0078] 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.

[0079] Where alkyl groups are sufficiently long to form branched or cyclic chains, the alkyl groups encompass branched,

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cyclic and linear alkyl chains. The alkyl groups are preferably linear or branched, most preferably linear.

[0080] 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.

[0081] Sequesterants may be present in the coated laundry detergent particles.

[0082] It is preferred that the coated detergent particle has a core to shell ratio of from 3 to 1:1, most preferably 2.5 to 1.5:1; the optimal ratio of core to shell is 2:1.

EXPERIMENTAL

Example 1: (particle manufacture)

Preparation of core of particle

[0083] 1962.5g of dried, milled surfactant blend (LAS/PAS/NI 68/17/15 by weight) was thoroughly mixed with 37.38g of perfume oil. The mixture was then extruded using a ThermoFisher 24HC twin screw extruder, operated at a rate of 8kg/hr. Inlet temperature of the extruder was set at 20°C, rising to 40°C just prior to the die-plate. The die-plate used was drilled with 6 circular orifices of 5mm diameter.

[0084] The extruded product was cut after the die-plate using a high speed cutter set up to produce particle with a thickness of ~1.1 mm.

Coating of Particle

[0085] 1200g of the extrudate above were charged to the fluidising chamber of a Strea 1 laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated using 1285g of a solution containing 640g of citric acid in 960 g of water, using a top-spray configuration.

[0086] The coating solution was fed to the spray nozzle of the Strea 1 via a peristaltic pump (Watson-Marlow model 101 U/R) at an initial rate of 3.3g/min, rising to 9.1g/min during the course of the coating trial.

[0087] The Fluid bed coater was operated with an initial air inlet air temperature of 55°C increasing to 90°C during the course of the coating trial whilst maintaining the outlet temperature in the range 45-50°C throughout the coating process.

[0088] Similar particles were prepared with a sodium sulphate and sodium carbonate coating.

Panel Test

[0089] Ten out of ten panellists found that large coated detergent particles coated with citric acid were shinier than those coated with sodium sulphate or sodium sulphate and more aesthetically pleasing to the eye. In similar test the panel also found that those with dye incorporated into the coating in citric acid were of greater colour intensity than similar particles with a sodium sulphate coating.

Example 2 Coloured particles

[0090] 2 batches of coloured particles were made with the core particle described in example 1. A reference particle was coated with sodium carbonate, and an example of the current invention made with citric acid. 1200g of the core particles were coated with the following solutions:

Reference particle

[0091] Solution for coating made up as follows:

1400g demin water
600g sodium carbonate
2g dye solution
2.1g Tinopal CBSX

[0092] Of this solution 1714g was sprayed onto the core particles so that the coated particle contains 30 wt% (dry weight) of sodium carbonate as a coating.

Citric acid particle

[0093] Solution for coating made up as follows:

- 5 960g of demin water
- 640g citric acid
- 1.6g dye solution

[0094] Of this solution 1285g was sprayed onto the crystals so that the coated particle contains 30wt% (dry weight) of citric acid as a coating.

[0095] The dye solution for both granules contained 50g of Liquitint™ Pink AL a liquid preparation of a red dye ex Milliken and 5g of Pigmosol Blue 6900 (Pigment Blue 15:1 ex BASF).

[0096] The reflectance spectra of both granules were measured on a reflectometer and the colour expresses as the CIE L*a*b* values (UV excluded). The results are shown below:

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	L*	a*	b*
Reference (Na ₂ CO ₃)	69.3	9.2	-19.1
Citric acid coated	63.9	20.3	-28.7

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[0097] The citric acid coated granules are more colourful with larger magnitude of a* and b* than the carbonate coated granules. The citric acid coated granules contain less dye than the carbonate coated.

[0098] In contrast to powders being coated with citric acid the larger particle also has greater brightness in ambient light than powders.

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Example 3 Spotting

[0099] 25 of the coloured citric acid particles of example 2 were scattered on a 20 x 20 cm of piece of white woven Egyptian Cotton cloth placed in 500ml of demin water and left for 40 minutes. The cloth was then washed. No spots were observed from the citric acid coated particles.

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Claims

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1. A coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein the particle comprises:

- (i) from 40 to 90 wt % surfactant selected from: anionic surfactant; and, non-ionic surfactant;
 - (ii) from 1 to 40 wt % citric acid; and,
 - (iii) from 0 to 3 wt % of a perfume,
- wherein the citric acid is present on the laundry detergent particle as a coating and the surfactant is present as a core.

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2. A coated detergent particle according to claim 1, wherein the coated detergent particle comprises from 10 to 35 wt% of a citric acid as a coating

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3. A coated detergent particle according to claim 1 or 2, wherein the citric acid coating comprises a dye or pigment in the range from 0.0001 to 0.1 wt % dye or pigment.

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4. A coated detergent particle according to any preceding claim, wherein the core comprises a dye or pigment in the range from 0.0001 to 0.1 wt % dye or pigment.

5. A coated detergent particle according to any one of the preceding claims, wherein the coated detergent particle comprises from 15 to 85 wt % anionic surfactant on surfactant and from 5 to 75 wt % non-ionic surfactant on surfactant.

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6. A coated detergent particle according to any one of claims 1 to 5, wherein the coated detergent particle comprises 15 to 100 wt % anionic surfactant on surfactant of which 20 to 30 wt % is sodium lauryl ether sulphate.

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7. A coated detergent particle according to any one of the preceding claims, wherein the anionic surfactant is selected from alkyl benzene sulphonates; alkyl ether sulphates; alkyl sulphates.
- 5 8. A coated detergent particle according to claim 7, wherein the anionic surfactant is selected from sodium lauryl ether sulfate with 1 to 3 ethoxy groups, sodium C₁₀ to C₁₅ alkyl benzene sulphonates and sodium C₁₂ to C₁₈ alkyl sulphates.
9. A coated detergent particle according to any one of the preceding claims, wherein the non-anionic surfactant is 10 to 50 EO.
- 10 10. A coated detergent particle according to claim 9, wherein the non-ionic surfactant is the condensation products of aliphatic C₈ to C₁₈ primary or secondary linear or branched alcohols with 20 to 35 ethylene oxide groups.
11. A coated detergent particle according to claim 3 or 4, wherein the core comprises a dye which is a blue or violet shading dye.
- 15 12. A coated detergent particle according to claim 3 or 4, wherein the coating comprises a dye is a blue or violet shading dye.
13. A coated detergent particle according to any one of the preceding claims, wherein the particle comprises from 0 to 20 15 wt % water.
14. A coated detergent particle according to claim 13, wherein the particle comprises from 1 to 5 wt % water.
15. A coated detergent particle according to any one of the preceding claims, wherein the coated detergent particle comprises from 10 to 100 wt % of a detergent formulation in a package.
- 25 16. A coated detergent particle according to claim 15, wherein the coated detergent particle comprises from 50 to 100 wt % of a detergent formulation in a package.
17. A coated detergent particle according to claim 16, wherein the coated detergent particle comprises from 80 to 100 wt % of a detergent formulation in a package.
- 30 18. A coated detergent particle according to claim 17, wherein the coated detergent particle comprises from 90 to 100 wt % of a detergent formulation in a package.
- 35 19. A coated detergent particle according to any one of the preceding claims, wherein at least 90 to 100 % of the coated detergent particles in the in the x, y and z dimensions are within a 20 % variable from the largest to the smallest coated detergent particle.
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EUROPEAN SEARCH REPORT

Application Number
EP 10 18 7522

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