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(54) PARTICULATE DETERGENT COMPOSITION COMPRISING PERFUME

PARTIKELFÖRMIGE WASCHMITTELZUSAMMENSETZUNGEN MIT PARFÜM

COMPOSITIONS DE DÉTERGENTS PARTICULAIRES COMPRENANT UN PARFUM

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Description**Field of the invention**

5 [0001] The present invention relates to detergent particles having perfume, particularly to detergent particles having a coating applied around a surfactant core.

Background of the invention

10 [0002] Perfumes are generally incorporated into the detergent compositions either to deliver fragrance in the headspace of the package or to deliver fragrance to the clothes being washed, in order to give a fresh clean smell. The free oil and perfume in particulate or powder forms are added onto the detergent powder by spraying or other means for incorporating the free oils. The perfume added at this stage is held in the small pores present in the powder structure by capillary pressure.

15 [0003] Detergent composition may also be produced by an extrusion process, in such a process the perfume addition is along with the extrudable mass. The perfume is added into the extrudable mass prior to the process of extrusion for multiple reasons. Addition of the perfume prior to the extrusion process makes the product sticky and forming an easily extrudable mass which is also more consumer acceptable upon extrusion. The process of extrusion eliminates the small capillary channels that would normally hold the perfume in the powder detergent composition, thus prior addition of perfume into the extrudable mass ensures that perfume is better held in the extruded particle.

20 [0004] WO2005/059083 A1 (Unilever) discloses a granulated detergent composition having encapsulated perfume. The granulated detergent product includes coated granules which has a functional core and a coating having encapsulated perfume for improving the dispersion of the dry encapsulated perfume throughout the detergent composition without the use of significant shear forces, which may result in noticeable perfume losses.

25 [0005] EP2627748 B1 (2014, Unilever) discloses a particulate detergent composition having an extruded surfactant core having an inorganic coating. The detergent composition has perfume added into the core either as a liquid or as encapsulated perfume particles.

30 [0006] However, addition of the perfume prior to the extrusion has some undesirable effects. Addition of perfume along with the extrudable mass prior to the extrusion process exposes the perfume oil to temperature and process conditions that may cause significant perfume losses. In addition, during the extrusion process breakages are seen in perfume added into extruder in the encapsulated form.

35 [0007] The present inventors have observed perfume losses in the range of 10 to 50% by way of evaporation, when exposed to temperature conditions generally employed during extrusion. Furthermore, the exhaust air carrying such perfume impacts environment and surroundings and thereby the exhaust air requires further treatment before releasing into the environment.

[0008] Thus, it is an object of the present invention to provide a detergent particle that has a core coated with a coating material and perfume.

[0009] It is another object of the present invention to provide free flowing detergent particle having improved perfume delivery.

40 [0010] It is yet another object of the present invention to minimize perfume losses while producing detergent particle.

Summary of the invention

45 [0011] We have now found that it is possible to have a perfume added with the coating of a detergent particle, which lowers the losses that are encountered when the perfume is added into the extrudable mass prior to extrusion while maintaining the perfume delivery. It is also found that the perfume added in the coating of the detergent particle of the present invention does not impact its free-flowing properties. Furthermore, incorporating the perfume at a later stage in the manufacturing process simplifies the changeover process from one product to another by late differentiation.

50 [0012] According to the present invention there is provided a detergent particle having a core and a coating enveloping the core, said detergent particle having perpendicular dimensions x, y and z, wherein x is from 0.2 to 2 mm, y is from 2 to 8 mm, and z is from 2 to 8 mm and the particle comprises:

- i. from 20 to 39wt% of a surfactant;
- ii. from 5 to 40wt% of a coating material; and,
- 55 iii. perfume;

wherein the core includes 95 parts to 100 parts of the total surfactant content and the coating includes 95 parts to 100 parts of the coating material and the perfume and wherein the detergent particle has an intra-particle porosity in the

range from 0 to 0.2 volume fraction.

Detailed description of the invention

SHAPE OF THE DETERGENT PARTICLE

[0013] Disclosed detergent particle has perpendicular dimensions x, y and z, where x is from 0.2 to 2 mm, y is from 2 to 8 mm, and z is from 2 to 8 mm.

[0014] Preferably in a detergent formulation having the detergent particle according to the present invention at least 90 to 100 % of the detergent particle in the x, y and z dimensions are within a 20 %, preferably 10%, variable from the largest to the smallest detergent particle.

[0015] The detergent particles are larger and less spherical than conventional detergent powders. Preferably the detergent particle is curved.

[0016] The 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. The size is such that y and z are at least 2 mm, preferably at least 2.5 mm, still preferably at least 3mm, more preferably at least 4 mm, and x lies in the range 0.2 to 2 mm, preferably 0.5 to 2 mm, more preferably 1 to 2 mm, still preferably 0.6 to 1.6mm.

[0017] Preferred ranges of y lies in the range from 3 to 8 mm, more preferably from 4 to 6 mm, still preferably from 4.5 mm to 5.5 mm. Preferred ranges of z lies in the range from 3 to 8 mm, more preferably from 4 to 6 mm, still preferably from 4.5 mm to 5.5 mm. Preferably the detergent particle may be shaped as a disc. In a preferred embodiment the detergent particle are oblate spheroids. Preferably the coated detergent particle does not have hole; that is to say, the detergent particle does not have a conduit passing there through that passes through the core, i.e., the coated detergent particle has a topologic genus of zero.

[0018] Porosity of the detergent particle of the present invention having a coating enveloping the core is in the range from 0 to 0.2 volume fraction, more preferably from 0.01 to 0.1, still preferably from 0.04 to 0.1, most preferably from 0.05 to 0.1. It is surprisingly found that the detergent particle having intra-particle porosity in the range from 0 to 0.1 volume fraction is capable of holding the perfume in the coating of the detergent particle of the present invention without altering the flow properties of the detergent particle.

[0019] Preferably the detergent particle of the present invention includes an extruded core and the intra particle porosity of the detergent particle having an extruded core and coated with a coating material is preferably in the range of 0 to 0.1 volume fraction. The extruded particle has a lower intra-particle porosity as compared to a detergent particle prepared by a high shear mixer granulator. The typical values of the intra-particle porosity of a high shear mixer granulated detergent particle is in the range from 0.3 to 0.5 volume fraction. Without being bound by theory it is believed that a high shear mixer granulated detergent particle due to the capillary pressure resulting from the intra particle porosity is capable of effectively entrapping liquid added to the particle surface, such as perfume. On the other hand, a detergent particle preferably having an extruded core and subsequently coated with the coating material has lower intra-particle porosity and has low liquid absorption capability via capillary absorption.

Porosity measurement:

[0020] The porosity of the detergent particle of the present invention was measured determined using a Quantachrom Helium Pycnometer, Model Ultra pycnometer 1000. The intra particle porosity was calculated E_p using the following equation

$$BD = U(1 - E_p)(1 - E_b)$$

wherein;

BD = bulk density of the solid (detergent particle)

U = absolute density of the solid as measured in a Helium pycnometry (measure of the volume excluding the pores and the void spaces between particles of the solid)

E_b = bed porosity

E_p = solid porosity

[0021] The intra particle porosity E_p for the detergent particle was calculated to be 0 to 0.2, more preferably 0 to 0.18, more preferably 0 to 0.1.

COMPOSITION OF THE DETERGENT PARTICLE

[0022] Disclosed detergent particle has a core and a coating applied to the core, which coating envelops the core. The appearance of the coated particle is pleasing if the core of the detergent particle is formed by extrusion. The core of the detergent particle includes the surfactant and the coating includes the coating material and the perfume. Preferably the coating material forms a layer enveloping the core and the perfume forms a layer enveloping at least an outer surface of the coating material.

[0023] It is preferred that the detergent particle has a core to coating ratio of from 3:1 to 20:1, most preferably 5:1 to 15:1; the optimal ratio of core to coating ratio is 9:1.

Core

[0024] The detergent particle according to the present invention has 20 wt% to 39wt% surfactant and the core of the detergent particle includes 95 parts to 100 parts of the total surfactant content.

[0025] Preferably the detergent particle includes 96 parts to 100 parts, still preferably 97.5 parts to 100 parts and still more preferably 98 to 100 parts of the total surfactant content.

Surfactant

[0026] 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 surfactant used are saturated.

[0027] The detergent particle of the present invention includes 20 to 39wt% of a surfactant, preferably 25 to 39wt% and still preferably 30 wt% to 35wt% surfactant. The surfactant is preferably selected from anionic surfactant, non-ionic surfactant or mixtures thereof. Preferably 15 to 85 parts of the total surfactant content in the detergent particle is anionic surfactant and from 5 to 75 parts of the total surfactant content is non-ionic surfactant.

Anionic surfactant:

[0028] 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 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 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 linear.

[0029] 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.

[0030] 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.

Nonionic surfactant:

[0031] 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 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.

[0032] Preferably all the surfactants are mixed together before being dried. Conventional mixing equipment may be used. The surfactant core of the detergent particle may be formed by extrusion or roller compaction and subsequently coated with an inorganic salt. Preferably the core of the detergent particle is an extrudate.

Calcium tolerant surfactant system:

[0033] In another aspect the surfactant system used is calcium tolerant and this is a preferred aspect because this reduces the need for builder.

[0034] 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.

[0035] 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.

[0036] 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 to 7 EO, and alkyl-ethoxylate nonionic surfactants, particularly those with melting points less than 40°C.

[0037] The core is primarily surfactant, it may also include detergency additives, such as shading dye, enzymes and polymers.

Coating

[0038] Disclosed detergent particle has a coating enveloping the core. The coating includes 95 parts to 100 parts of the total coating material content and the total perfume content of the detergent particle. The coating material forms a layer enveloping the core and the perfume forms a layer on a surface of the coating material layer. It is preferred that the coating material forms a layer enveloping at least a portion of the outer surface of the core, more preferably the coating material forms a layer enveloping at least 50%, more preferably at least 60%, still preferably at least 80% and further preferably completely envelops the outer surface of the coating material.

[0039] Preferably the coating includes 96 parts to 100 parts, more preferably 98 parts to 100 parts most preferably 99 parts to 100 parts of the total coating material content and the total perfume content of the detergent particle.

[0040] The coating of the detergent particle comprises less than 5wt%, even preferably less than 2.5wt%, further preferably less than 1wt%, still preferably less than 0.5wt% surfactant, more specifically a non-ionic surfactant.

Coating material

[0041] Although the skilled person might assume that any known coating material may be used, for instance organic coating material, including polymer, it has been found to be particularly advantageous to use an inorganic coating material deposited by crystallisation from an aqueous solution as this appears to give positive dissolution benefits and the coating gives a good colour to the detergent particle, even at lower coating levels.

[0042] The coating material is preferably applied to the surface of the surfactant core, by deposition from an aqueous solution of the coating material. In the alternative, the coating material layer is applied in the form of a slurry. It is highly preferred to coat by spraying an aqueous solution of the coating material in a fluidised bed drier. The bed is typically fluidized with heated air in order to dry or partially dry moisture from the spray coating as it is applied. The spraying is achieved via nozzles capable of forming a fine or atomized spray of the coating mixture to achieve complete coverage of the particles. Typically, the droplet size from the atomizer is less than about 100 micrometres. This atomization can be achieved either through a conventional two-fluid nozzle with atomizing air, or alternatively by means of a conventional pressure nozzle. To achieve this type of atomization, the solution or slurry rheology is typically characterized by a viscosity of less than about 500 centipoise, preferably less than about 200 centipoise. To achieve best results, the nozzle location is placed at or above the fluidized height of the particles in the fluid bed. The fluidized height is typically determined by a weir or overflow gate height. The coating zone of the fluid bed is then typically followed by a drying zone and a cooling

zone. Of course, one with ordinary skill in the art will recognize that alternative arrangements are also possible to achieve the resultant coating on the detergent particles of the present invention.

[0043] An alternative embodiment uses an agitated fluid bed, which includes mechanical and/or pneumatic mixing elements in addition to the conventional bed that is fluidized air passing through holes in a distributor plate. The advantage of the agitated bed is that it can be used to apply additional shear as a means to control granular shape and smoothness while performing the coating operation.

Inorganic material and Inorganic salt:

[0044] Preferably the coating material is an inorganic material. The inorganic material is preferably an inorganic salt, more preferably a water soluble inorganic salt. Inorganic salt(s) is/are preferably present as a coating on the detergent particle, the coating is present enveloping the core. The inorganic salt(s) is/are preferably present at a level that reduces the stickiness of the detergent particle to a point where the particles are free flowing.

[0045] It will be appreciated by those skilled in the art that while multiple layered inorganic salt coatings, of the same or different inorganic salt, could be applied, a single coating layer is preferred, for simplicity of operation, and to maximise the thickness of the coating.

[0046] Disclosed detergent particle includes 10 to 40wt% of the coating material. When the coating material is an inorganic salt it is preferably selected from sodium carbonate and/or sodium sulphate of which preferably at least 5 parts of the total inorganic salt is sodium carbonate.

[0047] Preferably the inorganic salt forms a layer covering/enveloping the core and the coating is preferably applied to the surface of the surfactant core, by deposition from an aqueous solution having 25 to 30wt% of the water-soluble inorganic salt. In the alternative coating can be performed using a slurry. The aqueous solution preferably contains greater than 150g/L, more preferably 300 g/L of the salt. An aqueous spray-on of the coating solution in a fluidized bed has been found to give good results and may also generate a slight rounding of the detergent particle during the fluidisation process. Drying and/or cooling may be needed to finish the process.

Perfume

[0048] In addition to the coating material the coating of the detergent particle according to the present invention includes a perfume.

[0049] In the coating, the perfume may be present either within the coating material layer, on the outer surface of the coating material layer or at least covering a portion of the outer surface of the coating layer or a combination of thereof.

[0050] The perfume forms a layer on a surface of the coating material layer. It is preferred that the perfume forms a layer enveloping at least a portion of an outer surface of the coating material, more preferably completely enveloping an outer surface of the coating material.

[0051] Perfume is preferably sprayed onto a detergent particle having a surfactant core pre-coated with a coating material layer, which coating material is preferably an inorganic salt, still preferably a water soluble inorganic salt. The term "pre-coated" as used herein refers to the detergent particle comprising the surfactant core enveloped with a coating material but prior to coating with the perfume. Preferably, the perfume forms a layer on an exposed outer surface of the coating material layer. The perfume layer either covers a portion of the outer surface of the coating material layer, or covers the entire outer surface of the coating material layer. It is believed that presence of perfume layer on the surface of the coating material layer improves perfume delivery and maintains the encapsulated perfume intact. Preferably the spray coating is in a mixer other than a fluid bed drier.

[0052] Preferably the perfume layer includes a free oil perfume, perfume in encapsulated form encapsulating a free oil also termed herein as encapsulated perfume or mixtures thereof.

[0053] Preferably when the perfume is a free oil perfume, the content of perfume molecules in the free oil is in the range from 90 to 100wt% by weight of the free oil composition. The free oil may have small amounts of solvent in the composition, the content of such solvent preferably is in the range from 0 to 10wt% but most preferably not more than 5wt%.

[0054] Preferably when the perfume is an encapsulated perfume, the encapsulated perfume is in the form of an slurry suspending the encapsulated perfume droplets/particles in a polar solvent, preferably the slurry is an aqueous slurry. The encapsulated perfume composition in this form comprises from 30 to 60wt% moisture and between 30 to 50wt% perfume oil. Preferably the encapsulated perfume composition includes a polymeric structuring system to increase the viscosity of the aqueous phase. The inclusion of the polymeric structuring system ensures that the encapsulated perfume is uniformly dispersed in the composition and does not separate out from the mixture. Any commercially available encapsulated perfume may be used for the present invention. A preferred kind of the encapsulated perfume is shear sensitive encapsulated perfume.

[0055] The preferred ratio in which the free oil to encapsulated perfume is present is between 5:1 to 1:5, more preferably 2:1 to 1:2 and the most optimum ratio is 1:1.

[0056] Preferably the perfume layer is multi-layered. The multi-layered perfume includes a layer of perfume in encapsulate form and a layer of free oil. Preferably the multi-layered perfume includes a layer of perfume in encapsulate form enveloping a layer of free oil. The layer of perfume in the encapsulate form preferably envelops a portion of the outer surface of free oil layer, or more preferably envelops the entire outer surface of the free oil layer. When the perfume layer is multi-layered it is preferred that the free oil layer is applied first followed by the deposition of a layer of perfume in encapsulated form.

[0057] Thus when the perfume layer is multi-layered the layer of free oil is proximal to an outer surface of the coating material layer of the detergent particle.

[0058] Preferably after the core of the detergent particle is coated with the coating material which is preferably an inorganic salt layer, the particles are then treated to provide a perfume layer on the coating material layer. The perfume layer is preferably provided by spraying the perfume oil or the perfume in encapsulate form in a coating mixer onto the pre-coated detergent particle. The coating mixer may be any of a number of mixers including low speed mixers and drum mixers.

[0059] The detergent particle includes perfume preferably in the range from 0.001 to 3 wt %, most preferably from 0.1 to 2 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.

[0060] 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.

[0061] Preferably the perfume may be in the form of a solid, liquid solution having an organic solvent, or in the form of a slurry having a polar solvent.

[0062] 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.

[0063] It is preferred that the detergent particle does not contain a peroxygen bleach, e.g., sodium percarbonate, sodium perborate, and peracid.

Water content

[0064] The detergent particle preferably includes from 0 to 15 wt% water, more preferably 0 to 10 wt%, most preferably from 1 to 5wt % water, at 293K and 50% relative humidity. This facilitates the storage stability of the detergent particle and its mechanical properties.

Other ingredients

[0065] The detergent particle according to the present invention may include further ingredients as described below which may be present in the coating or the core of the particle or in both the core as well as the coating.

Fluorescent Agent

[0066] The 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.

[0067] 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.

[0068] 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-sulphophenyl)-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)]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.

Polymers

[0069] The detergent particle may preferably include one or more 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

[0070] Preferably the detergent particle includes one or more enzymes. Preferably the level of each enzyme is from 0.0001 wt% to 0.5 wt% protein on product. Especially contemplated enzymes include proteases, alpha-amylases, cellulases, lipases, peroxidases/oxidases, pectate lyases, laccases and mannanases, or mixtures thereof.

[0071] 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. stearotheophilus* (JP 64/744992) or *B. pumilus* (WO 91/16422).

[0072] 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.

[0073] Preferred commercially available lipase enzymes include Lipolase™ and Lipolase Ultra™, Lipex™ (Novozymes A/S) and Lipoclean™.

[0074] 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.

[0075] 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.

[0076] 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™, Dyrasym™, Esperase™, Everlase™, Polarzyme™, and Kan-nase™, (Novozymes A/S), Maxatase™, Maxacal™, Maxapem™, Properase™, Purafect™, Purafect Oxp™, FN2™, and FN3™ (Genencor International Inc.).

[0077] 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.

[0078] 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.).

[0079] 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).

[0080] 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).

[0081] Further enzymes suitable for use are disclosed in WO2009/087524, WO2009/090576, WO2009/148983 and WO2008/007318.

Enzyme Stabilizers

[0082] Any enzyme present in the detergent particle 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.

[0083] 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.

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

PROCESS FOR PREPARING THE DETERGENT PARTICLE

[0085] In a second aspect of the present invention disclosed is a process for preparing the detergent particle according to the first aspect preferably including the steps of:

a) feeding a surfactant to an extruder and extruding it to form an extrudate with a diameter of at least 3 mm, preferably at least 4 mm;

b) cutting the extrudate to form the core of the detergent particle having a thickness less than their diameter wherein the thickness is at least 0.2 mm,

c) coating the core by applying 5 to 40 wt% of a coating material to form a coating material layer and preferably drying it.

d) applying a perfume to form a layer on a surface of the coating material layer.

[0086] Preferably, the coating material is an inorganic salt more preferably selected from sodium carbonate and/or sodium sulphate. The perfume layer is preferably applied by spraying the perfume oil or the perfume in encapsulate form in a coating mixer onto the pre-coated detergent particle. The coating mixer may be any of a number of mixers including low speed mixers and drum mixers.

[0087] The extruder provides further opportunities to blend in ingredients other than surfactants, or even to add further surfactants. However, it is generally preferred that all of the anionic surfactant, or other surfactant supplied in admixture with water; i.e. as paste or as solution, is added into the drier to ensure that the water content can then be reduced and the material fed to and through the extruder is sufficiently dry. Additional materials that can be blended into the extruder are thus mainly those that are used at very low levels in a detergent composition: such as fluorescer, shading dye, enzymes, perfume, silicone antifoams, polymeric additives and preservatives. The limit on such additional materials blended in the extruder has been found to be about 10 wt%, but it is preferred for product quality to be ideal to keep it to a maximum of 5 wt%. Solid additives are generally preferred. Solid particulate structuring (liquid absorbing) materials or builders, such as zeolite, carbonate, silicate are preferably not added to the blend being extruded. These materials are not needed due to the self-structuring properties of the very dry LAS-based feed material. If any is used the total amount should be less than 5 wt%, preferably less than 4 wt%, most preferably less than 3 wt%. At such levels no significant structuring occurs and the inorganic particulate material is added for a different purpose, for instance as a flow aid to improve the feed of particles to the extruder. The output from the extruder is shaped by the die plate used. The extruded material has a tendency to swell up in the centre relative to the periphery. We have found that if a cylindrical extrudate is regularly sliced as it exits the extruder the resulting shapes are short cylinders with two convex ends. These particles are herein described as oblate spheroids, or lentils. This shape is pleasing visually.

Coating

[0088] The next step involves coating the extruded core by applying 5 to 40 wt% of a coating material to form a coating material layer and preferably drying it. Coating allows the particles to be coloured easily. Coating makes the particles more suitable for use in detergent compositions that may be exposed to high humidity for long periods.

[0089] The extruded particles can be considered as oblate spheroids with a major radius "a" and minor radius "b". Hence, the surface area(S) to volume (V) ratio can be calculated as:

$$\frac{S}{V} = \frac{3}{2b} + \frac{3b}{4\epsilon a^2} \ln\left(\frac{1+\epsilon}{1-\epsilon}\right) \text{ mm}^{-1}$$

when ϵ is the eccentricity of the particle.

DETERGENT FORMULATION HAVING THE DISCLOSED DETERGENT PARTICLE

[0090] According to another aspect of the present invention disclosed is a detergent formulation including 50 to 100wt% detergent particle according to the present invention. The detergent formulation is preferably present in a package.

[0091] Preferably the package for commercial sale includes 0.01 kg to 5 kg, preferably 0.02 kg to 2 kg and most preferably 0.5 kg to 2 kg of the detergent formulation.

[0092] Preferably the detergent particle of the present invention includes all the ingredients required in a fully formulated detergent formulation in which case the detergent formulation includes 100wt% of the detergent particle. In other preferred embodiments the detergent particle of the present invention may be admixed with additional ingredients, such as bleaching agents, enzymes, perfumes, non-coated detergent particles, and various other ingredients to produce a fully formulated detergent composition. In such embodiments at least 50wt% the detergent particle more preferably at least 60wt%, still preferably at least 70wt% and further preferably at least 80wt% of the detergent particle is present the detergent formulation.

Examples

Example 1: Comparative detergent particle having perfume incorporated in the core

[0093] Core manufacture: Surfactant raw materials were dried in a spray-drier to produce a spray-dried powder having anionic surfactant and non-ionic surfactant. The spray-dried powder was fed to a twin-screw co-rotating extruder fitted with a shaped orifice plate and cutter blade to extrude the core particle. During the extruding process, perfume at a level of 0.527% was added to achieve a final perfume level of 0.4 % by weight in the full formulation, including coating and additional materials. The perfume included a mixture of free oil and perfume in encapsulated form.

[0094] In addition to perfume, during the extrusion process other ingredients like enzymes, dyes were also dosed into the extrudable mass.

[0095] Applying the coating: The core particle obtained in the previous step was then coated with the coating material by spray deposition. Sodium carbonate was used as the coating material. The core particle was charged to the fluidizing chamber of a Strea 1 laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated with a solution of sodium carbonate using a top-spray configuration to form a coating material layer enveloping the surfactant core. The sodium carbonate solution was fed to the spray nozzle of the Strea 1 via a peristaltic pump (Watson-marlow model 101U/R). The composition of the coating material is given below.

[0096] The detergent particle obtained by the above process involved the following composition:

Core particle: 1000 grams

Coating solution: 110 grams sodium carbonate dissolved in 293 grams water.

[0097] The perfume concentration in the prepared detergent particle (comparative example 1) was measured immediately after coating using the Gas Chromatography method, the measurement were repeated for 5 different detergent particles prepared in the same batch and the results are tabulated in Table 1. The error in the perfume oil measurement was $\pm 0.03\%$. The total loss during the preparation of the detergent particle were calculated by determining the actual theoretical level of perfume in the coated particle given the coating level achieved and then calculating the percentage remaining by dividing the measured value by the theoretical value and the loss is then calculated. The data obtained are provided in Table 1.

Table 1

Samples	Measured total perfume wt% present in detergent particle according to Example 1	Total perfume (wt%) added in each detergent particle according to Example 1	Measured coating level for sample	Total perfume Losses (wt%)
1	0.33	0.527	11.55	41.25

(continued)

Samples	Measured total perfume wt% present in detergent particle according to Example 1	Total perfume (wt%) added in each detergent particle according to Example 1	Measured coating level for sample	Total perfume Losses (wt%)
2	0.33	0.527	14.69	39.08
3	0.35	0.527	13.32	36.41
4	0.32	0.527	15.53	40.34
5	0.33	0.527	14.09	39.51
Average loss				39.32

[0098] The data provided in table 1 shows that when the perfume is incorporated into the core (as in comparative example 1) which involves the process of extrusion and fluid bed coating post perfume addition, there is an average perfume loss of 39% in the detergent particle, further it was observed that the perfume added in the encapsulated form were ruptured in the detergent particle.

Example 2: Detergent particle prepared according to the present invention having a coating with perfume.

[0099] Core manufacture: A core particle was prepared using a process similar to that disclosed in Example 1. The only difference in the process was that in the detergent particle prepared in Example 2 no perfume was added to the extruder during the extrusion process. Therefore there was no perfume in the core of Example 2.

[0100] Applying the coating: The core particle obtained in the previous step was then coated with sodium carbonate solution by spray deposition similar to as explained in Example 1. The intra particle porosity of the detergent particle after the application of the coating was determined using a Quantachrom Helium Pycnometer, Model Ultrapycnometer 1000 and was found to be 0.06 volume fraction.

[0101] Applying perfume layer: The detergent particle pre-coated with the sodium carbonate layer was then transferred to a drum mixer. To a batch of 300 kg detergent particle in the drum mixer, 2.73 Kg of free oil was sprayed on the detergent particle through a single phase Nozzle 4012 (from Spray systems) at a maximum pressure of 0.5 bar for a period of around 90 seconds and thereafter 1.06 Kg of perfume in encapsulated form was sprayed through an internal atomised twin phase nozzle, SU43 (from spray systems) at an atomizing pressure of 0.8 bar for a period of around 70 seconds. Thereafter mixing was continued for a further 90 seconds before discharging the detergent particle having a coating including sodium carbonate layer and a perfume layer.

[0102] The perfume content of the detergent particle produced by Example 2 was measured analytically using Gas Chromatography. The perfume content and loss percentage were determined for a sample of 5 different detergent particle prepared in the same batch and the results are provided in Table 2.

Table 2

Samples	Measured total perfume (wt%) present in detergent particle according to Example 2	Total perfume (wt%) added in each detergent particle according to Example 2	Total perfume Losses (%)
1	1.1 $\pm$ 4%	1.04	0
2	1.0 $\pm$ 4%	1.04	4%
3	1.1 $\pm$ 4%	1.04	0
4	1.1 $\pm$ 4%	1.04	0

[0103] As seen in Table 2, detergent particle having a composition according to the present invention (Example 2) having a coating with free oil and perfume in encapsulated form did not shown significant perfume loss and SEM analysis showed that the perfume in encapsulated form was predominantly intact.

Example 3: Determining the flow rate of the inventive detergent particle:

[0104] The flow rate (or CDFR) of the detergent particle of Example 2 was determined by the following method. A sample of the detergent particle was placed in a glass tube having a diameter of 59.5 millimetre and the time taken for a fixed volume (718.78 ml) to flow out of an orifice having a diameter of 40 millimeters was measured and the flow rate was then calculated in mL/s and provided in Table 3.

Table 3

Trial No	Day 0 CDFR (ml/s)	Week 12 CDFR (ml/s) at		
		20°C	37°C	45°C
1	465	480	498	477
2	470	497	488	477
3	493	475	505	486
4	476	499	505	486

[0105] The data above showing the flow rate for the detergent particle of Example 2 immediately after applying the perfume (Day 0) and the change in flow rate after 12 weeks storage reflects very little change in flow over this period, indicating that the particles were free flowing upon storage.

Claims

1. A detergent particle having a core and a coating enveloping the core, said detergent particle having perpendicular dimensions x, y and z, wherein x is from 0.2 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm and the particle comprises:

- i. from 20 to 39 wt% surfactant;
- ii. from 5 to 40 wt% coating material; and,
- iii. perfume;

wherein the core comprises 95 parts to 100 parts of the total surfactant content and the coating comprises 95 parts to 100 parts of the coating material and the perfume and wherein the detergent particle has an intra-particle porosity in the range from 0 to 0.2 volume fraction.

2. A detergent particle according to claim 1 wherein the coating material is an inorganic material, preferably an inorganic salt selected from sodium carbonate, sodium sulphate or mixtures thereof.

3. A detergent particle according to claim 1 or 2 wherein the coating material forms a layer covering the core and the perfume forms a layer covering at least a portion of an outer surface of the coating material layer.

4. A detergent particle according to any one of the preceding claims, wherein the perfume layer comprises free oil, perfume in encapsulate form or mixtures thereof.

5. A detergent particle according to claim 4 wherein the perfume layer is multi-layered comprising a layer of perfume in encapsulate form covering a layer of free oil.

6. A detergent particle according to claim 5 wherein the free oil layer of the multi-layered perfume layer is proximal and is covering an outer surface of said coating material layer.

7. A detergent particle according to any one of the preceding claims wherein the surfactant is selected from anionic surfactant, non-ionic surfactant or mixtures thereof.

8. A detergent particle according to claim 7 wherein the total surfactant content of the detergent particle comprises from 15 to 85 parts anionic surfactant and from 5 to 75 parts non-ionic surfactant.

9. A detergent particle according to any one of the preceding claims wherein the particle comprises from 0.001 wt% to 3 wt% perfume.
10. A detergent particle according to any one of the preceding claims wherein the detergent particle are oblate spheroids.
11. A detergent particle according to any one of the preceding claims wherein the amount of coating on each particle is from 5 wt% to 45wt%, preferably 5wt% to 15wt% by weight of the particle.
12. A detergent particle according to any one of the preceding claims, wherein the particle comprises from 0 to 15 wt % water, preferably from 1 to 5 wt % water.
13. A detergent particle according to any one of the preceding claims wherein the core of the detergent particle is an extrudate.
14. A detergent formulation comprising the detergent particle according to any one of the preceding claims 1 to 12, wherein the detergent particle comprises from 50 to 100 wt % of the detergent formulation in a package.
15. A detergent formulation according to claim 14 wherein at least 90 to 100 % of the detergent particles in the x, y and z dimensions are within a 20 % variable from the largest to the smallest coated detergent particle.

Patentansprüche

1. Reinigungsmittelteilchen mit einem Kern und einer den Kern umhüllenden Beschichtung, wobei das Reinigungsmittelteilchen senkrechte Abmessungen x, y und z aufweist, wobei x 0,2 bis 2 mm, y 2 bis 8 mm und z 2 bis 8 mm beträgt, und die Teilchen umfassen:
 - i. 20 bis 39 Gew.-% Tensid;
 - ii. 5 bis 40 Gew.-% Beschichtungsmaterial; und
 - iii. Duftstoff;

wobei der Kern 95 Teile bis 100 Teile des gesamten Tensidgehalts umfasst und die Beschichtung 95 Teile bis 100 Teile des Beschichtungsmaterials und des Duftstoffs umfasst und wobei das Reinigungsmittelteilchen eine intrapartikuläre Porosität im Bereich von 0 bis 0,2 Volumenanteilen aufweist.
2. Reinigungsmittelteilchen nach Anspruch 1, wobei das Beschichtungsmaterial ein anorganisches Material ist, bevorzugt ein anorganisches Salz, ausgewählt aus Natriumcarbonat, Natriumsulfat oder Mischungen davon.
3. Reinigungsmittelteilchen nach Anspruch 1 oder 2, wobei das Beschichtungsmaterial eine Schicht bildet, die den Kern bedeckt, und der Duftstoff eine Schicht bildet, die mindestens einen Teil einer Außenfläche der Beschichtungsmaterialschicht bedeckt.
4. Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche, wobei die Duftstoffschicht freies Öl, Duftstoff in eingekapselter Form oder Mischungen davon umfasst.
5. Reinigungsmittelteilchen nach Anspruch 4, wobei die Duftstoffschicht mehrschichtig ist, umfassend eine Schicht von Duftstoff in eingekapselter Form, die eine Schicht von freiem Öl bedeckt.
6. Reinigungsmittelteilchen nach Anspruch 5, wobei die freie Ölschicht der mehrschichtigen Duftstoffschicht proximal ist und eine Außenfläche der Beschichtungsmaterialschicht bedeckt.
7. Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche, wobei das Tensid ausgewählt ist aus anionischem Tensid, nichtionischem Tensid oder Mischungen davon.
8. Reinigungsmittelteilchen nach Anspruch 7, wobei der gesamte Tensidgehalt des Reinigungsmittelteilchens 15 bis 85 Teile anionisches Tensid und 5 bis 75 Teile nichtionisches Tensid umfasst.
9. Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche, wobei das Teilchen 0,001 Gew.-% bis

3 Gew.-% Duftstoff umfasst.

10. Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche, wobei die Reinigungsmittelteilchen abgeflachte Sphäroide sind.

11. Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche, wobei die Menge der Beschichtung auf jedem Teilchen 5 Gew.-% bis 45 Gew.-%, bevorzugt 5 Gew.-% bis 15 Gew.-%, bezogen auf das Gewicht des Teilchens, beträgt.

12. Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche, wobei das Teilchen 0 bis 15 Gew.-% Wasser, bevorzugt 1 bis 5 Gew.-% Wasser, umfasst.

13. Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche, wobei der Kern des Reinigungsmittelteilchens ein Extrudat ist.

14. Reinigungsmittelformulierung, umfassend das Reinigungsmittelteilchen nach irgendeinem der vorhergehenden Ansprüche 1 bis 12, wobei das Reinigungsmittelteilchen 50 bis 100 Gew.-% der Reinigungsmittelformulierung in einer Verpackung umfasst.

15. Reinigungsmittelformulierung nach Anspruch 14, wobei mindestens 90 bis 100% der Reinigungsmittelteilchen in den Abmessungen x, y und z innerhalb einer 20%-Variablen von dem größten zum kleinsten beschichteten Reinigungsmittelteilchen liegen.

Revendications

1. Particule de détergent présentant un noyau et un revêtement enveloppant le noyau, ladite particule de détergent présentant des dimensions perpendiculaires x, y et z, dans laquelle x est de 0,2 à 2 mm, y est de 2 à 8 mm, et z est de 2 à 8 mm et la particule comprend :

- i. de 20 à 39 % en masse de tensioactif ;
- ii. de 5 à 40 % en masse de matériau de revêtement ; et,
- iii. du parfum ;

dans laquelle le noyau comprend de 95 parties à 100 parties de la teneur totale en tensioactif et le revêtement comprend de 95 parties à 100 parties du matériau de revêtement et du parfum et dans laquelle la particule de détergent présente une porosité intra-particule dans l'intervalle de 0 à 0,2 de fraction de volume.

2. Particule de détergent selon la revendication 1, dans laquelle le matériau de revêtement est un matériau inorganique, de préférence un sel inorganique choisi parmi le carbonate de sodium, sulfate de sodium ou des mélanges de ceux-ci.

3. Particule de détergent selon la revendication 1 ou 2, dans laquelle le matériau de revêtement forme une couche recouvrant le noyau et le parfum forme une couche recouvrant au moins une portion d'une surface externe de la couche de matériau de revêtement.

4. Particule de détergent selon l'une quelconque des revendications précédentes, dans laquelle la couche de parfum comprend de l'huile libre, du parfum dans une forme encapsulée ou des mélanges de ceux-ci.

5. Particule de détergent selon la revendication 4, dans laquelle la couche de parfum est multicouche comprenant une couche de parfum dans une forme encapsulée recouvrant une couche d'huile libre.

6. Particule de détergent selon la revendication 5, dans laquelle la couche d'huile libre de la couche de parfum multicouche est proximale et recouvre une surface externe de ladite couche de matériau de revêtement.

7. Particule de détergent selon l'une quelconque des revendications précédentes, dans laquelle le tensioactif est choisi parmi un tensioactif anionique, tensioactif non-ionique ou des mélanges de ceux-ci.

8. Particule de détergent selon la revendication 7, dans laquelle la teneur totale en tensioactif de la particule de

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détergent comprend de 15 à 85 parties de tensioactif anionique et de 5 à 75 parties de tensioactif non-ionique.

9. Particule de détergent selon l'une quelconque des revendications précédentes, dans laquelle la particule comprend de 0,001 % en masse à 3 % en masse de parfum.

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10. Particule de détergent selon l'une quelconque des revendications précédentes, dans laquelle les particules de détergent sont des sphéroïdes aplatis.

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11. Particule de détergent selon l'une quelconque des revendications précédentes, dans laquelle la quantité de revêtement sur chaque particule est de 5 % en masse à 45 % en masse, de préférence de 5 % en masse à 15 % en masse de la particule.

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12. Particule de détergent selon l'une quelconque des revendications précédentes, dans laquelle la particule comprend de 0 à 15 % en masse d'eau, de préférence de 1 à 5 % en masse d'eau.

13. Particule de détergent selon l'une quelconque des revendications précédentes, dans laquelle le noyau de la particule de détergent est un extrudé.

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14. Formulation de détergent comprenant la particule de détergent selon l'une quelconque des revendications 1 à 12 précédentes, dans laquelle la particule de détergent comprend de 50 à 100 % en masse de la formulation de détergent dans un emballage.

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15. Formulation de détergent selon la revendication 14, dans laquelle d'au moins 90 à 100 % des particules de détergent dans les dimensions x, y et z se trouvent dans une variable de 20 % de la particule de détergent revêtue la plus large à la plus petite.

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