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## (54) Title: LAUNDRY DETERGENT PARTICLES

(57) **Abstract:** The present invention provides a coated lenticular or disc detergent particle having perpendicular dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8 mm 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 % water soluble inorganic salts; and (iii) from 0.0001 to 0.1 wt % dye, wherein the dye is covalently bound to from 1 to 4 sulphonate groups, wherein the inorganic salts and the dye are present on the detergent particle as a coating and the surfactant is present as a core.



WO 2012/048948 A1

- 1 -

## **Laundry Detergent Particles**

### Field of Invention

- 5     The present invention relates to substantially light coloured particles that provide a perceptible colour when added to a liquor.

### Background of Invention

- 10    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
- 15    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
- 20    in example 2.

US 7,022,660 discloses a process for the preparation of a detergent particle having a coating.

### 25    Summary of the Invention

We have found that it is possible to have a dye containing coating that is substantially colourless but releases the dye colour to the wash liquor. The invention may also increase the photostability of the dye in the product on storage.

- 2 -

We have also found that it is possible to provide a coated laundry detergent particle to provide a wash colour that is different from the perceived colour, other than white, of the coated laundry detergent particle; in this respect the perceived colour is due to a pigment. The perceived colour of the coated laundry detergent particle is preferably white but for example a red or orange coated laundry detergent particle may provide a blue colour to the wash. Alternatively, red coated laundry detergent particle may provide a shading benefit that is perceived as whiteness by the use of a blue or violet shading dye.

- 10 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 %, preferably 20 to 40 wt%, water soluble inorganic salts; and,
  - (iii) from 0.0001 to 0.1 wt % dye, preferably 0.001 to 0.01 wt % dye, wherein the dye is covalently bound to from 1 to 4 sulphonate groups,
- wherein the inorganic salts and the dye are present on the laundry detergent particle as a coating and the surfactant is present as a core.

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

- 25 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

- 3 -

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.

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#### Detailed Description of the Invention

##### SHAPE

Preferably the coated laundry detergent particle is curved.

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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  $y = z$ .

15 The coated laundry detergent particle may be shaped as a disc.

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

20 topologic genus of zero.

##### CORE

##### SURFACTANT

25 The coated laundry detergent particle comprises between 40 to 90 wt%, preferably 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  
30 current edition of "McCutcheon's Emulsifiers and Detergents" published by Manufacturing Confectioners Company or in "Tenside-Taschenbuch", H. Stache,

- 4 -

2nd Edn., Carl Hauser Verlag, 1981. Preferably the surfactants used are saturated.

### Anionic Surfactants

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Suitable anionic detergent compounds which may be used are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher C<sub>8</sub> to C<sub>18</sub> alcohols, produced for example from tallow or coconut oil, sodium and potassium alkyl C<sub>9</sub> to C<sub>20</sub> benzene sulphonates, particularly sodium linear secondary alkyl C<sub>10</sub> to C<sub>15</sub> benzene sulphonates; and sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum. Most preferred anionic surfactants are sodium lauryl ether sulfate (SLES), particularly preferred with 1 to 3 ethoxy groups, sodium C<sub>10</sub> to C<sub>15</sub> alkyl benzene sulphonates and sodium C<sub>12</sub> to C<sub>18</sub> 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.

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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.

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Preferably, at least 50 wt % of the anionic surfactant is selected from: sodium C<sub>11</sub> to C<sub>15</sub> alkyl benzene sulphonates; and, sodium C<sub>12</sub> to C<sub>18</sub> alkyl sulphates. Even

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- 5 -

more preferably, the anionic surfactant is sodium C<sub>11</sub> to C<sub>15</sub> alkyl benzene sulphonates.

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

#### Nonionic Surfactants

10 Suitable nonionic detergent compounds which may be used include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Preferred nonionic detergent compounds are C<sub>6</sub> to C<sub>22</sub> alkyl phenol-  
15 ethylene oxide condensates, generally 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic C<sub>8</sub> to C<sub>18</sub> 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.

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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.

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

Preferably all the surfactants are mixed together before being dried. Conventional mixing equipment may be used. The surfactant core of the laundry detergent

- 6 -

particle may be formed by extrusion or roller compaction and subsequently coated with an inorganic salt.

#### Calcium Tolerant Surfactant System

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

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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.

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

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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 ( $4 \times 10^{-3}$  Molar  $\text{Ca}^{2+}$ ). 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.

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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

- 7 -

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.

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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.

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#### Water Soluble Inorganic Salts

The water-soluble inorganic salts are preferably selected from sodium carbonate, sodium chloride, sodium silicate and sodium sulphate, or mixtures thereof, most preferably, 70 to 100 wt% sodium carbonate on total water-soluble inorganic salts. The water-soluble inorganic salt is present as a coating on the particle. The water-soluble inorganic salt is preferably present at a level that reduces the stickiness of the laundry detergent particle to a point where the particles are free flowing.

20 It will be appreciated by those skilled in the art that while multiple layered coatings, of the same or different coating materials, could be applied, a single coating layer is preferred, for simplicity of operation, and to maximise the thickness of the coating. The amount of coating should lay in the range 1 to 40 wt% of the particle, preferably 20 to 40 wt%, more preferably 25 to 35 wt% for the best results in terms of anti-caking properties of the detergent particles.

25

The coating is preferably applied to the surface of the surfactant core, by deposition from an aqueous solution of the water soluble inorganic salt. In the alternative coating can be performed using a slurry. The aqueous solution preferably contains greater than 50g/L, more preferably 200 g/L of the salt. An

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- 8 -

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.

5

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.

## 10 DYE

The dye is added to the coating 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.

15

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

The dyes for use in the current invention are covalently bound to from 1 to 4  
20 sulphonate groups, preferably 1 to 2 sulphonate groups.

Suitable dye may be selected from acid and direct dyes listed in the color index (Society of Dyers and Colourists and American Association of Textile Chemists and Colorists). Preferably the dye is an acid dye.

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The dye may be any colour, preferable the dye is blue, violet, green or red. Most preferably the dye is blue or violet.

The dye is preferably selected from those having: anthraquinone; mono-azo; bis-  
30 azo; xanthene; phthalocyanine; and, phenazine chromophores.

- 9 -

More preferably the dye is selected from those having: anthraquinone; mono-azo; and, phenazine chromophores. Most preferably the dye is selected from anthraquinone and phenazine chromophores.

- 5 Preferably the dye is selected from: acid blue 80, acid blue 62, acid violet 43, acid green 25, direct blue 86, acid blue 59, acid blue 98, direct violet 9, direct violet 99, direct violet 35, direct violet 51, acid violet 50, acid yellow 3, acid red 94, acid red 51, acid red 95, acid red 92, acid red 98, acid red 87, acid yellow 73, acid red 50, acid violet 9, acid red 52, food black 1, food black 2, acid red 163, acid black 1,
- 10 acid orange 24, acid yellow 23, acid yellow 40, acid yellow 11, acid red 180, acid red 155, acid red 1, acid red 33, acid red 41, acid red 19, acid orange 10, acid red 27, acid red 26, acid orange 20, acid orange 6, sulphonated Al and Zn phthalocyanines.
- 15 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
- 20 violet to blue-green.

A combination of dyes may be used.

- The dye is preferably present in the coating as an agglomerate. An agglomerate is
- 25 a distinct particle containing 4 or more dye molecules. Each distinct agglomerate preferably consists of more than 16 dye molecules.

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### The coated laundry detergent particle

Preferably, the coated laundry detergent particle comprises 0 to 10 wt % water, more preferably, from 1 to 5 wt % water at 293K and 50% relative humidity.

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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.

- 10 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.

- 15 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.

- 20 With regard to the optical properties of the white laundry detergent particle, these are perceived as white but with the following parameters  $L^* > 70$ ,  $a^*$  and  $b^*$  from -5 to +5, preferably  $b^*$  from -1 to +5, preferably  $a^*$  from -2 to +2.

### Water content

- 25 The particle preferably comprises from 0 to 15 wt % water, more preferably 0 to 10 wt %, most preferably from 1 to 5 wt % water. This facilitates the storage stability of the particle and its mechanical properties.

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- 11 -

### Other Adjuncts

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

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### Fluorescent Agent

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.

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.

Preferred classes of fluorescer are: Di-styryl biphenyl compounds, e.g. Tinopal (Trade Mark) CBS-X, Di-amine stilbene di-sulphonic acid compounds, e.g. Tinopal DMS pure Xtra and Blankophor (Trade Mark) HRH, and Pyrazoline compounds, e.g. Blankophor SN. Preferred fluorescers are: sodium 2 (4-styryl-3-sulfophenyl)-2H-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.

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- 12 -

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.

## 5 Perfume

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  
10 Fragrance Association) 1992 International Buyers Guide, published by CFTA Publications and OPD 1993 Chemicals Buyers Directory 80th Annual Edition, published by Schnell Publishing Co.

It is commonplace for a plurality of perfume components to be present in a  
15 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.

In perfume mixtures preferably 15 to 25 wt% are top notes. Top notes are defined  
20 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.

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

## Pigments

The coated laundry detergent particle may contain one or more Organic or  
30 Inorganic pigments, to provide a colour. The colour is preferably different to the

- 13 -

dye, so that on dissolution the liquor is a different colour to the coated laundry detergent particle. For example a coated laundry detergent particle coloured red with a pigment and containing a blue dye, to give a blue liquor colour. Suitable Inorganic Pigments are described in Industrial Inorganic Pigments edited by G. Buxbaum and G. Pfaff (3<sup>rd</sup> edition Wiley-VCH 2005). Suitable organic pigments are described in Industrial Organic Pigments edited by W. Herbst and K. Hunger (3<sup>rd</sup> 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.

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Pigments are 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.

15

Suitable organic pigments are preferably selected from monoazo pigments, betanaphthol 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.

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25 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

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- 14 -

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.

10 Pigments are preferably present from 0.001 to 0.1wt%.

#### Polymers

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.

#### 20 Enzymes

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

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, and mannanases, or mixtures thereof.

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- 15 -

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  
5 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.*  
10 *stearothermophilus* (JP 64/744992) or *B. pumilus* (WO 91/16422).

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,  
15 WO 00/60063, WO 09/107091 and WO09/111258.

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

20 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.

Phospholipids, such as lecithin or phosphatidylcholine, consist of glycerol  
25 esterified with two fatty acids in an outer (sn-1) and the middle (sn-2) positions and esterified with phosphoric acid in the third position; the phosphoric acid, in turn, may be esterified to an amino-alcohol. Phospholipases are enzymes which participate in the hydrolysis of phospholipids. Several types of phospholipase activity can be distinguished, including phospholipases A<sub>1</sub> and A<sub>2</sub> which hydrolyze  
30 one fatty acyl group (in the sn-1 and sn-2 position, respectively) to form



- 16 -

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.

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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 Kannase™, (Novozymes A/S), Maxatase™, Maxacal™, Maxapem™, Properase™, Purafect™, Purafect OxP™, FN2™, and FN3™ (Genencor International Inc.).

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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.

20

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

25

amylases are Duramyl™, Termamyl™, Termamyl Ultra™, Natalase™, Stainzyme™, Fungamyl™ and BAN™ (Novozymes A/S), Rapidase™ and Purastar™ (from Genencor International Inc.).

30

Suitable cellulases include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Suitable cellulases include cellulases

- 17 -

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  
5 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).

- 10 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  
15 Novozym™ 51004 (Novozymes A/S).

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

## 20 Enzyme Stabilizers

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  
25 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.

- 18 -

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.

- 5 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.

Sequesterants may be present in the coated laundry detergent particles.

10

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.

15 EXPERIMENTAL

LAS refers to linear alkyl benzene sulphonate. PAS refers to primary alkyl sulphate. NI refers to an ethoylated alcohol non-ionic surfactant having an average of 30 ethoylated units and an alkyl chain of C12-14. Specifically the following were used: LAS – UFASAN 65 ex Unger, PAS – Stepanol CFAS70 ex Stepan and NI – Leutensol AO 30 ex BASF.

20

Example 1: (particle manufacture)

- 25 Two coated laundry detergent particle colour were created containing Acid Violet 50 such that:

Particle 1 contain Acid Violet 50 in the core (reference)

Particle 2 contains Acid Violet 50 in a carbonate coating

30

- 19 -

The particles were oblate ellipsoids which had the following dimension x= 1.1 mm  
y= 4.0 mm z= 5.0 mm.

The particles weighed ~0.013g each.

5

Particle 1 appeared violet to the eye, particle 2 appear off-white to the eye.

#### Preparation of core of particle 1

10 1962.5g of dried, milled surfactant blend (LAS/PAS/NI 68/17/15 by weight) was  
thoroughly mixed with 37.38g of perfume oil and 0.124g of Acid Violet 50 dye. 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  
15 circular orifices of 5mm diameter.

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.

#### 20 Coating of Particle 1

764g of the extrudates above were charged to the fluidising chamber of a Strea 1  
laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated using 1069g of  
a solution containing 320.7g of sodium carbonate in 748.3g of water, using a top-  
25 spray configuration.

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

30

- 20 -

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.

5 Preparation of core of Particle 2

1962.9g of dried, milled surfactant blend (LAS/PAS/NI 68/17/15 by weight) was mixed with 37.38g of perfume oil and the mixture extruded using a ThermoFisher 24HC twin screw extruder, operated at a rate of 8kg/hr. Inlet temperature of the  
10 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.

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.

15

Coating of Particle 2

715g of the extrudates above were charged to the fluidising chamber of a Strea 1 laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated using 1000g of  
20 a solution containing of 300g sodium carbonate in 0.09g Acid Violet 50 and 669.91 of water using a top-spray configuration.

The coating solution was fed to the spray nozzle of the Strea 1 via a peristaltic pump (Watson-Marlow model 101U/R) at an initial rate of 2.7g/min, rising to  
25 25g/min during the course of the coating trial.

The Fluid bed coater was operated with an initial air inlet air temperature of 60°C increasing to 75 °C during the course of the coating trial whilst maintaining the outlet temperature in the range 47-52°C throughout the coating process.

30

- 21 -

**Example 2:** (Coated laundry detergent particle colour)

The colour of the particles of example 1 was measured using a reflectometer (UV-excluded) and expressed as the CIE L\*a\*b\* value. The results are shown below:

5

	L*	a*	b*
Particle 1: Dye in the Core (Reference)	76.2	2.9	-7.9
Particle 2: Dye in the Coating	85.8	0.5	4.4

L\* is the lightness, as objects become coloured L\* drops

a\* is the red-green axis with +ve values indicating a red colour and –ve a green colour

10

b\* is the yellow-blue axis with +ve values indicating a yellow colour and –ve a blue colour

Particle 1 is clearly violet with a negative b\* value.

15

Particle 2 is a slightly yellow-white colour. The yellowness comes from the slightly yellow colour of the surfactants.

- 22 -

**Example 3:** (Dye in Coating – 2<sup>nd</sup> composition including polymer and other minor ingredients in the core)

Preparation of core of Particle 3

5

1000g of dried, milled surfactant blend (LAS/PAS/NI 68/17/15 by weight) was thoroughly mixed with 35g of 1-Hydroxyethylidene-1,1-Diphosphonic Acid Tetra Sodium Salt (Dequest 2016D ex thermPhos), 50g of polymer (Texcare SRA 300 F ex Clariant) and 140g of granular Anhydrous Citric Acid (particle size 250 – 710  
10 microns). 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.

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

Coating of Particle 3

20 750g of the extrudates above were charged to the fluidising chamber of a Strea 1 laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated using 1050g of a solution containing of 315g sodium carbonate in 0.095g Acid Violet 50 and 734.91g of water using a top-spray configuration.

25 The coating solution was fed to the spray nozzle of the Strea 1 via a peristaltic pump (Watson-Marlow model 101U/R) at an initial rate of 4.7g/min, rising to 30g/min during the course of the coating trial.

- 23 -

The Fluid bed coater was operated with an initial air inlet air temperature of 55°C increasing to 72 °C during the course of the coating trial whilst maintaining the outlet temperature in the range 47-49°C throughout the coating process.

5 Particle 3 appear white to the eye.

The colour of the particles of example 3 was measured using a reflectometer (UV-excluded) and expressed as the CIE L\*a\*b\* value. The results are shown below

	L*	a*	b*
Particle 3	90.15	-0.18	3.39

10

**Example 4: (Wash liquor colour)**

2.04 g of Particle 2 and 2.25 of Particle 1 were separately dissolved in 100ml of demineralised water. The solutions were centrifuged at 15 minutes for 11000

15 RPM and the colour of the liquid measured on A UV-VIS absorption spectrometer. Both liquid appeared violet to the eye.

The UV-VIS spectrum gave the spectrum of Acid Violet 50 for both solutions with a maximum absorption at 570nm. The optical densities are given in the table

20 below

	Optical density (5cm) at 570nm
Particle 1: Dye in Core (Reference)	0.175
Particle 2: Dye in Coating	0.155

Both particles effectively deliver Acid Violet 50 to solution.

25



- 24 -

**Example 5: (Staining)**

25 of each particle were scattered on to a 20 by 20 cm piece of white woven cotton which was submerged in 500ml of demineralised water such that the cloth  
5 were covered by 2cm of water. The particles were left for 40 minutes then the cloth washed, rinsed and dried. The number of stains on each cloth was counted and the % staining calculated. % staining is the fraction of particles that give rise to stains:

$$\% \text{staining} = 100 \times (\text{number of stains}) / (\text{number of particles})$$

10 The results are given in the table below:

	%staining
Particle 1 Dye in Core (Reference)	12
Particle 2 Dye in Coating	4

Surprisingly the particles show very low staining. Surprisingly the particle with the dye in the coating shows lowest staining.

15

**Example 6: (particle manufacture)**

Three coated laundry detergent particle colour were created containing the mono sulfonated dye Acid Blue 80 such that:

20

Particle 3 contain Acid Blue 80 in the core (reference) without perfume

Particle 4 contains Acid Blue 80 in a carbonate coating

Particle 5 contained Acid Blue 80 in a carbonate coating without perfume

25 The particles were oblate ellipsoids which had the following dimension x= 1.1 mm  
y= 4.0 mm z= 5.0 mm.

- 25 -

The particles weighed ~0.013g each.

#### Preparation of core of particle 3

5 2000g of dried, milled surfactant blend (LAS/PAS/NI 68/17/15 by weight) was thoroughly mixed with 0.124g of Acid Blue 80 dye. 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.

10

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 3

15

764g of the extrudates above were charged to the fluidising chamber of a Strea 1 laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated using 1069g of a solution containing 320.7g of sodium carbonate in 748.3g of water, using a top-spray configuration.

20

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

25

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.

30

- 26 -

#### Preparation of core of Particle 4

1962.9g of dried, milled surfactant blend (LAS/PAS/NI 68/17/15 by weight) was mixed with 37.38g of perfume oil and the mixture extruded using a ThermoFisher  
5 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.

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

#### Coating of Particle 4

715g of the extrudates above were charged to the fluidising chamber of a Strea 1  
15 laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated using 1000g of a solution containing of 300g sodium carbonate in 0.09g Acid Blue 80 and 669.91 of water using a top-spray configuration.

The coating solution was fed to the spray nozzle of the Strea 1 via a peristaltic  
20 pump (Watson-Marlow model 101U/R) at an initial rate of 2.7g/min, rising to 25g/min during the course of the coating trial.

The Fluid bed coater was operated with an initial air inlet air temperature of 60°C increasing to 75 °C during the course of the coating trial whilst maintaining the  
25 outlet temperature in the range 47-52°C throughout the coating process.

#### Preparation of core of Particle 5

2000g of dried, milled surfactant blend (LAS/PAS/NI 68/17/15 by weight) was  
30 extruded using a ThermoFisher 24HC twin screw extruder, operated at a rate of

- 27 -

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.

- 5 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 5

- 10 715g of the extrudates above were charged to the fluidising chamber of a Strea 1 laboratory fluid bed drier (Aeromatic-Fielder AG) and spray coated using 1000g of a solution containing of 300g sodium carbonate in 0.09g Acid Blue 80 and 669.91 of water using a top-spray configuration.
- 15 The coating solution was fed to the spray nozzle of the Strea 1 via a peristaltic pump (Watson-Marlow model 101U/R) at an initial rate of 2.7g/min, rising to 25g/min during the course of the coating trial.

- The Fluid bed coater was operated with an initial air inlet air temperature of 60°C
- 20 increasing to 75 °C during the course of the coating trial whilst maintaining the outlet temperature in the range 47-52°C throughout the coating process.

#### Example 7: (Coated laundry detergent particle colour)

- 25 The colour of the particles of example 1 was measured using a reflectometer (UV-excluded) and expressed as the CIE L\*a\*b\* value. The results are shown below:

- 28 -

	L*	a*	b*
Particle 3: Dye in the Core (Reference)	87.8	-4.9	-2.1
Particle 4: Dye in the Coating with perfume	91.0	-1.6	2.9
Particle 5: Dye in the Coating	90.4	-1.8	3.3

L\* is the lightness, as objects become coloured L\* drops

a\* is the red-green axis with +ve values indicating a red colour and -ve a green colour

- 5 b\* is the yellow-blue axis with +ve values indicating a yellow colour and -ve a blue colour

Particle 3 is clearly blue-green with a negative a\* and b\* value.

Particle 4 and 5 are a slightly yellow-white colour. The yellowness comes from the slightly yellow colour of the surfactants.

- 29 -

**We claim:**

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  
5 the particle comprises:  
(i) from 40 to 90 wt % surfactant selected from: anionic surfactant; and, non-ionic surfactant;  
(ii) from 1 to 40 wt % water soluble inorganic salts; and,  
(iii) from 0.0001 to 0.1 wt % dye, wherein the dye is covalently bound to from 1 to  
10 4 sulphonate groups,  
wherein the inorganic salts and the dye are present on the detergent particle as a coating and the surfactant is present as a core.
2. A coated detergent particle according to claim 1, wherein the dye is an acid  
15 dye.
3. A coated detergent particle according to claim 1 or 2, wherein the dye is selected from those having: anthraquinone; mono-azo; bis-azo; xanthene; phthalocyanine; and, phenazine chromophores.  
20
4. A coated detergent particle according to claim 3, wherein the dye is selected from those having: anthraquinone; mono-azo; and, phenazine chromophores.
- 25 5. A coated detergent particle according to claim 1, Wherein the dye is selected from: acid blue 80, acid blue 62, acid violet 43, acid green 25, direct blue 86, acid blue 59, acid blue 98, direct violet 9, direct violet 99, direct violet 35, direct violet 51, acid violet 50, acid yellow 3, acid red 94, acid red 51, acid red 95, acid red 92, acid red 98, acid red 87, acid yellow 73, acid red 50, acid violet 9,  
30 acid red 52, food black 1, food black 2, acid red 163, acid black 1, acid orange 24,

- 30 -

acid yellow 23, acid yellow 40, acid yellow 11, acid red 180, acid red 155, acid red 1, acid red 33, acid red 41, acid red 19, acid orange 10, acid red 27, acid red 26, acid orange 20, acid orange 6, sulphonated Al and Zn phthalocyanines.

5 6. A coated detergent particle according to any one of the preceding claims, wherein the inorganic salts act as a builder.

7. A coated detergent particle according to claim 6, wherein the inorganic salts comprises sodium carbonate.

10

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

15 9. 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.

10. A coated detergent particle according to claim 9, wherein the anionic  
20 surfactant is selected from sodium lauryl ether sulfate with 1 to 3 ethoxy groups, sodium C<sub>10</sub> to C<sub>15</sub> alkyl benzene sulphonates and sodium C<sub>12</sub> to C<sub>18</sub> alkyl sulphates.

11. A coated detergent particle according to any one of the preceding claims,  
25 wherein the non-ionic surfactant is a 10 to 50 EO non-ionic surfactant.

12. A coated detergent particle according to claim 11, wherein the non-ionic surfactant is the condensation products of aliphatic C<sub>8</sub> to C<sub>18</sub> primary or secondary linear or branched alcohols with 20 to 35 ethylene oxide groups.

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- 31 -

13. A coated detergent particle according to any one of the preceding claims, wherein the coated detergent particle comprises 20 to 40 wt % of inorganic builder salts as a coating.

5 14. A coated detergent particle according to claim 13, wherein the coated detergent particle comprises 25 to 35 wt % of inorganic builder salts as a coating.

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

10

16. A coated detergent particle according to claim 15, wherein the particle comprises from 1 to 5 wt % water.

17. A coated detergent particle according to any one of the preceding claims,  
15 wherein the coated detergent particle comprises from 10 to 100 wt % of a detergent formulation in a package.

18. A coated detergent particle according to claim 17, wherein the coated  
20 detergent particle comprises from 50 to 100 wt % of a detergent formulation in a package.

19. A coated detergent particle according to claim 18, wherein the coated  
detergent particle comprises from 80 to 100 wt % of a detergent formulation in a  
package.

25

20. A coated detergent particle according to claim 19, wherein the coated  
detergent particle comprises from 90 to 100 wt % of a detergent formulation in a  
package.



- 32 -

21. 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.

# INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2011/065150

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> INV. C11D17/00 C11D3/40 C11D3/04 C11D3/08 C11D3/10 ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) C11D		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 2 166 077 A1 (PROCTER & GAMBLE [US]) 24 March 2010 (2010-03-24) paragraphs [0021], [0030] - [0033], [0038]; claims; examples -----	1-21
A	US 2 480 579 A (JOSEPH HOLUBA STANLEY) 30 August 1949 (1949-08-30) column 10, line 49 - column 11, line 6; claims -----	1-21
<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="display: flex; align-items: center;"> <input type="checkbox"/> Further documents are listed in the continuation of Box C.         </div> <div style="display: flex; align-items: center;"> <input checked="" type="checkbox"/> See patent family annex.         </div> </div>		
* Special categories of cited documents :		
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&amp;" document member of the same patent family</p> </div> </div>		
Date of the actual completion of the international search  <div style="text-align: center; font-size: 1.2em;">29 November 2011</div>		Date of mailing of the international search report  <div style="text-align: center; font-size: 1.2em;">16/12/2011</div>
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer  <div style="text-align: center; font-size: 1.2em;">Pfannenstein, Heide</div>

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2011/065150

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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