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(54) **PROCESS FOR PREPARING A SPRAY DRIED DETERGENT PARTICLE**

VERFAHREN ZUR HERSTELLUNG EINES SPRÜHGETROCKNETEN WASCHMITTELPARTIKELS

PROCÉDÉ DE PRÉPARATION D'UNE PARTICULE DE DÉTERGENT SÉCHÉE PAR ATOMISATION

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**Description****Field of the invention**

5   **[0001]** The present invention relates to a process for preparing a spray-dried detergent particle. Particularly to a spray-dried laundry detergent particle with low pH, low alkalinity and having good particle characteristics. The spray-dried detergent particle is suitable for use as a solid laundry detergent composition or for incorporation into a solid laundry detergent composition.

10   **Background of the invention**

**[0002]** Traditionally, powder detergent compositions have been formulated to provide a pH ranging from 10.5 to 11.5 in a wash solution. A basic pH ensures that the surfactant systems, enzymes, and other ingredients in the composition remain solubilized in the wash water, thereby promoting effective soil release and cleaning performance. Cotton fabric swells at a  
15   pH of 9.5 to 10, which allows the surfactant to penetrate deeply into the fibre. Furthermore, a basic pH ensures effective removal of fatty and particulate stains from soiled clothing.

**[0003]** Despite the above-mentioned advantages, the higher pH also has certain disadvantages. Higher pH in the wash solution tends to be harsh on the skin of consumers and are associated with the problem of colour fading of the laundered fabrics.

20   **[0004]** These days consumers prefer laundry detergent composition which are milder on the skin. Particularly consumers laundering fabrics by hand, prefer composition which deliver good cleaning performance while being mild to the skin and offering a desirable feel while washing. Further consumers also prefer detergent composition which deliver good foam during washing which is easily rinsed off at a later stage.

**[0005]** In the past, such milder compositions have been formulated by lowering the pH of the composition. Formulating a  
25   low pH composition necessitates significantly reducing or completely removing the alkaline ingredients such as sodium carbonate and sodium silicate. The sodium carbonate and sodium silicate not only provide a wash liquor pH of around 10.5, but they also function as effective builders to sequester Ca and Mg ions present in hard water. Beside these benefits, sodium carbonate and sodium silicate contribute to improving the physical properties such as flow and storage behaviours of the spray-dried detergent particle. Past attempts at reducing or eliminating sodium carbonate from the spray-dried  
30   detergent particle were not satisfactory as the resultant spray-dried detergent particle were found to have acceptable powder properties only for a short duration that is immediately after spray drying and showed caking on storage.

**[0006]** Along with sodium carbonate, sodium silicate is generally considered a critical ingredient in spray-dried detergent particle as it serves to provide stability and integrity to the detergent particle formed during the spray-drying operation. Several attempts to reduce or eliminate sodium silicate resulted in deterioration of powder properties with  
35   respect to flow and caking tendency. Sodium silicate also plays a role in the viscosity and flow behaviour of the slurry, a low silicate containing slurry may cause gelation of the slurry and the slurry may not pumpable.

**[0007]** Hence, preparing a low pH spray-dried detergent particle has been a challenge in the past where the particle possesses good powder flow properties especially on prolonged storage.

**[0008]** US3951877A (Lion Fat and Oil Co. Ltd, 1976) discloses a spray-dried heavy-duty granular detergent composition which maintains its granule strength during packaging or transport. The granular detergent composition includes  
40   inorganic aluminium salts, non-soap anionic surfactant and sodium citrate. The inorganic aluminium salt is preferably selected from the group consisting of aluminium salts, aluminium oxide, aluminium hydroxide.

**[0009]** US 3011977 (Bernhard Raecke, 1961) discloses a detergent composition which eliminates or substantially reduces the irritation of the skin by the addition of complex aluminium compounds. The composition includes phosphate  
45   builder.

**[0010]** US 3951877 A (Okumura Osamu et. al., 1976) discloses a heavy-duty granular detergent composition having 10 wt.% to 60 wt.% sodium citrate, 5 wt.% to 40 wt.% non-soap anionic surfactant and 0.1 wt.% to 10 wt.% inorganic aluminium salt.

**[0011]** It is thus an object of the present invention to provide a process for preparing a spray dried detergent particle  
50   having low pH and low alkalinity.

**[0012]** It is yet another object of the present invention to provide a spray-dried detergent particle which ensures good fabric care profile and is gentle to hand while maintaining good cleaning performance at low pH and low alkalinity profile of detergent composition.

**[0013]** It is yet another object of the present invention to provide a spray dried detergent particle with low levels of alkaline builders such as carbonate and silicate and yet having good powder properties over extended storage periods.

55   **[0014]** It is yet another object of the present invention to provide a spray dried detergent particle having low pH and alkalinity which is substantially free of carbonate, zeolite and STPP and which provides good cleaning performance and good powder properties.

**[0015]** It is yet another object of the present invention to provide a spray-dried particle which shows improved anti-ashing properties on multiple washes and deposits low or no insoluble residues on laundered fabrics.

## Summary of the invention

**[0016]** The present inventors have found that a spray dried detergent particle formed from an aqueous slurry having one or more ingredients selected from the group consisting of an organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof provides for excellent powder properties and extended shelf life without getting caked. It is also surprisingly found that the spray-dried particle provides desired pH in wash solution required for good stain removal performance without being harsh on the hands or the fabrics.

**[0017]** The spray-dried detergent particle according to the present invention providing one or more of the above-mentioned benefits preferably while incorporating low or no alkaline builders, particularly alkali metal carbonates and alkali metal silicates. Preferably the spray-dried detergent particle includes less than 2 wt.% alkali metal silicate. Preferably the spray-dried detergent particle includes no alkali metal carbonate.

**[0018]** According to a first aspect of the present invention, disclosed is a process for preparing a spray dried laundry detergent particle, said process comprising the steps of:

(i) reacting an organic carboxylic acid with any one of (a) aluminate salt solution (b) aluminium hydroxide solution wherein the aluminium hydroxide solution is formed in-situ by any one of the reaction selected from the group consisting of (i) reacting sodium aluminate solution with an acid; or, (ii) reacting an aluminium salt with an alkaline source; or mixtures thereof to form one or more reaction product selected from the group consisting of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof;

(ii) adding an amount of alkaline source to the aqueous mixture to provide an aqueous slurry having a pH of 4 to 8.5, wherein the aqueous slurry comprises one or more reaction product selected from the group consisting of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof and a deterative surfactant; and,

(iii) spray-drying the aqueous slurry to form a spray-dried detergent particle.

**[0019]** Preferably the aqueous slurry includes an organic carboxylic acid salt of alkali metal.

**[0020]** These and other aspects, features and advantages will become apparent to those of ordinary skill in the art from a reading of the following detailed description and the appended claims. For the avoidance of doubt, any feature of one aspect of the present invention may be utilised in any other aspect of the invention. The word "comprising" is intended to mean "including" but not necessarily "consisting of" or "composed of." In other words, the listed steps or options need not be exhaustive. It is noted that the examples given in the description below are intended to clarify the invention and are not intended to limit the invention to those examples per se. Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts of material or conditions of reaction, physical properties of materials and/or use are to be understood as modified by the word "about". Numerical ranges expressed in the format "from x to y" are understood to include x and y. When for a specific feature multiple preferred ranges are described in the format "from x to y", it is understood that all ranges combining the different endpoints are also contemplated.

## Detailed description of the invention

### Process of making a spray-dried detergent particle

**[0021]** According to a first aspect of the present invention disclosed is a process of preparing a spray-dried detergent particle comprising the steps as described herein below.

### Step (i): Reacting an organic carboxylic acid with any one of (i) aluminate salt solution, (ii) aluminium hydroxide or mixtures thereof

**[0022]** According to the first aspect of the present invention disclosed is a process of reacting an organic carboxylic acid with any one of (a) aluminate salt solution, (b) aluminium hydroxide wherein the aluminium hydroxide solution is formed in-situ by any one of the reaction selected from the group consisting of (i) reacting sodium aluminate solution with an acid; or, (ii) reacting an aluminium salt with an alkaline source, or mixtures thereof in an aqueous mixture.

Organic carboxylic acid:

**[0023]** Preferably the organic carboxylic acid is monomeric organic carboxylic acid, still preferably a monomeric organic polycarboxylic acid. Suitable examples of the organic carboxylic acid include but is not limited to formic acid, acetic acid, propionic acid, butyric acid, caprylic acid and lauric acid, stearic acid, linoleic acid, acrylic acid, methacrylic acid, chloroacetic acid, citric acid, lactic acid, glyoxylic acid, acetoacetic acid, oxalic acid, malonic acid, adipic acid, phenylacetic acid, benzoic acid, salicylic acid, glycine, alanine, valine, aspartic acid, glutamic acid, lysine, phenylalanine, nicotinic acid, picolinic acid, fumaric acid, benzoic acid, succinic acid and glycolic acid. Preferably, the organic acid is selected from the group consisting of citric acid, malic acid, succinic acid, lactic acid, glycolic acid, fumaric acid, tartaric acid, formic acid, and mixtures thereof. More preferably, the acid is citric acid, lactic acid, and tartaric acid. Most preferably the organic carboxylic acid is citric acid. The organic carboxylic acid is preferably added in excess.

**[0024]** Preferably the amount of the organic carboxylic acid is in an amount which changes the pH of the aqueous mixture to less than 4, where the pH is measured using a 1 wt.% solution in distilled water.

**[0025]** The organic carboxylic acid reacts with (i) aluminate salt solution, (ii) aluminium hydroxide solution wherein the aluminium hydroxide solution is formed in-situ by any one of the reaction selected from the group consisting of (i) reacting sodium aluminate solution with an acid; or, (ii) reacting an aluminium salt with an alkaline source, or mixtures thereof in an aqueous mixture to form one or more of the reaction products selected from an organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof.

Aluminate salt solution:

**[0026]** According to the first aspect of the present invention, the organic carboxylic acid may be reacted with an aluminate salt solution. Preferably the aluminate salt solution is an alkaline solution. Preferably the aluminate salt is an alkali metal salt of aluminate, alkaline earth metal salt of aluminate or mixtures thereof. More preferably an alkali metal aluminate. Preferably the aluminate salt is a sodium aluminate, a magnesium aluminate or mixtures thereof. Preferably the aluminate salt solution is sodium aluminate. Aluminate salt is preferably in a liquid form, solid form or formed in-situ. Sodium aluminate is commercially available in anhydrous form. Commercially available sodium aluminate preferably includes from 50 wt.% to 60 wt.% aluminium oxide ( $\text{Al}_2\text{O}_3$ ) and 35 wt.% to 45 wt.% sodium oxide ( $\text{Na}_2\text{O}$ ). The sodium aluminate is used in the present invention in the form of an aqueous solution. Available sodium aluminate are alkaline and have an  $\text{Al}_2\text{O}_3$  to  $\text{Na}_2\text{O}$  ratio in the preferred ratio ranges of 0.5:1 to 1.64:1, preferably 0.8:1 to 1.6:1, more preferably 1:1 to 1.6:1, further preferably 1.1:1 to 1.6:1, more preferably 1.15:1 to 1.5:1, still more preferably 1.19:1 to 1.35 :1. A commercially available sodium aluminate from ex. Sigma Aldrich has a  $\text{Al}_2\text{O}_3$  to  $\text{Na}_2\text{O}$  ratio of 0.5:1 to 1.64:1, preferably 0.8:1 to 1.55:1, more preferably 1:1 to 1.5:1.

**[0027]** In a preferred embodiment of the inventive process the aluminate salt is formed by digesting an aluminium compound with an alkaline source to form the aluminate salt. Preferably the aluminium compound is aluminium sulphate which is digested in presence of sodium hydroxide solution (alkaline source) to form sodium aluminate.

Aluminium hydroxide:

**[0028]** According to the first aspect of the present invention, the organic carboxylic acid may be reacted with an aluminium hydroxide. Preferably the aluminium hydroxide is solubilized in an alkaline solution. The aluminium hydroxide is formed in-situ.

In-situ formed aluminium hydroxide:

**[0029]** The aluminium hydroxide is in-situ formed. Preferably an aluminium salt reacts with an alkaline source to form in-situ aluminium hydroxide. Preferably the aluminium salt is selected from the group consisting of aluminium sulphate, aluminium chloride, or mixtures thereof. Preferably the alkaline source is selected from an alkali metal hydroxide, alkali metal silicate or mixtures thereof. Preferably the alkali metal hydroxide is the alkaline source, still preferably the alkaline source is sodium hydroxide. Preferably the in-situ formed aluminium hydroxide reacts with the organic carboxylic acid. Preferably the aluminium salt is aluminium sulphate which is added to the aqueous mixture having sodium hydroxide where the aluminium sulphate reacts with the sodium hydroxide to form in-situ aluminium hydroxide. The aluminium hydroxide thereafter reacts with the organic carboxylic acid. The aqueous mixture may preferably include a deterative surfactant. Preferably when the aqueous mixtures include a deterative surfactant in an acid form, it is preferred that the acid form is completely neutralized with the alkaline source (alkali metal hydroxide, preferably sodium hydroxide) before the addition of the aluminium salt. This is to avoid the possibility of formation of Aluminium salt of the deterative surfactant (AILAS).

**[0030]** In another preferred embodiment the in-situ aluminium hydroxide is formed by reacting sodium aluminate

solution with an acid. Preferably the acid is an inorganic acid. Preferably the acid is an acid form of LAS.

Organic carboxylic acid salt of aluminium:

**[0031]** The organic carboxylic acid reacts with (i) aluminate salt solution, (ii) aluminium hydroxide wherein the aluminium hydroxide solution is formed in-situ by any one of the reactions selected from the group consisting of (i) reacting sodium aluminate solution with an acid; or, (ii) reacting an aluminium salt with an alkaline source, or mixtures thereof in an aqueous mixture to form an organic carboxylic acid salts of aluminium. Preferably the organic carboxylic acid is citric acid which reacts with any one of (i) aluminate salt solution (ii) aluminium hydroxide wherein the aluminium hydroxide solution is formed in-situ by any one of the reactions selected from the group consisting of (i) reacting sodium aluminate solution with an acid; or, (ii) reacting an aluminium salt with an alkaline source or mixtures thereof to form aluminium citrates.

**[0032]** Preferably when the organic carboxylic acid is reacted with alkaline sodium aluminate salt solution, in addition to the organic carboxylic acid salt of aluminium, the reaction may also preferably result in the formation of one or more additional reaction products selected from the group consisting of aluminium hydroxide, alkali metal salt of organic carboxylic acid or mixtures thereof. Preferably in the aqueous mixture alkali metal salt of organic carboxylic acid is an organic carboxylic acid salt of sodium. Preferably the organic carboxylic acid salt of aluminium is aluminium citrates and has one or more of aluminium mono-citrate, aluminium dicitrate, aluminium tricitrate or mixtures thereof.

An aluminium complex of organic carboxylic acid:

**[0033]** The organic carboxylic acid may react with any one of (i) aluminate salt solution, (ii) aluminium hydroxide wherein the aluminium hydroxide solution is formed in-situ by any one of the reactions selected from the group consisting of (i) reacting sodium aluminate solution with an acid; or, (ii) reacting an aluminium salt with an alkaline source, or (iii) mixtures thereof in an aqueous mixture to form an aluminium complex of organic carboxylic acid. Preferably the organic carboxylic acid is citric acid.

#### Aqueous mixture

**[0034]** The aqueous mixture preferably includes a deterative surfactant. Suitable deterative surfactant includes anionic, nonionic, cationic, amphoteric, zwitterionic deterative surfactant or mixtures thereof. Preferably the deterative surfactant is anionic, nonionic or mixtures thereof. More preferably the aqueous mixture includes an anionic surfactant. Suitable anionic deterative surfactant is an alkyl sulphonate surfactant, alkyl sulphate surfactant or mixtures thereof. The deterative surfactant may be linear or branched, substituted or unsubstituted. The deterative surfactant may be derived from petrochemical material or is bioderived.

**[0035]** Anionic deterative surfactant: The aqueous mixture preferably includes a deterative surfactant. The deterative surfactant is preferably an anionic surfactant. Preferably the anionic surfactant is selected from the group consisting of alkyl benzene sulphonate, alkyl ether sulphate, alkyl sulphate or mixtures thereof. The deterative anionic surfactant is either pre-neutralized and added into the aqueous mixture or a liquid acid form of the anionic surfactant is added to the aqueous mixture and neutralized in-situ. Alternately, the acid form of the anionic surfactant may be partly neutralized and thereafter added into the aqueous mixture such that the remaining unneutralized part of the liquid acid form of the anionic surfactant is neutralized in-situ in the aqueous mixture. Pre-neutralized surfactant is commercially available in solid form or in the form of paste.

**[0036]** Preferably the deterative surfactant is added to the aqueous mixture before addition of the aluminate salt solution and/or aluminium hydroxide formed in-situ. In some embodiments the deterative surfactant in the acid form is in-situ neutralized using alkali metal hydroxide to form the neutralized salt form. Preferably the alkali metal hydroxide is sodium hydroxide. This is followed by addition of the aluminate salt solution and/or aluminium hydroxide formed in-situ. When the sodium aluminate salt is used, the acid form of the deterative surfactant reacts with the excess of alkali metal hydroxide to fully neutralize and thereafter the alkali metal hydroxide further reacts with the sodium aluminate salt to form the aluminium hydroxide.

**[0037]** In some other embodiments of the present invention the deterative surfactant in the neutralized salt form or acid form may be added to the aqueous mixture after the formation of the organic carboxylic acid salt of aluminium. When added in the acid form the deterative surfactant is preferably neutralized using alkali metal hydroxide to form the salt form. The alkali metal hydroxide is preferably sodium hydroxide.

**[0038]** When the deterative surfactant is added into the aqueous mixture in the form of a partly neutralized surfactant, the partly neutralized anionic surfactant is preferably prepared by a neutralization process which involves the step of (i) mixing a liquid acid form of the anionic surfactant and a neutralizing agent to form a partially neutralized solution; preferably the neutralizing agent is an alkali metal hydroxide, wherein the amount of alkali metal hydroxide neutralizing agent is sufficient to react with a portion of liquid acid anionic surfactant precursor to form in-situ anionic surfactant salt. The neutralized

anionic surfactant formed by neutralizing the acid form with the alkali metal hydroxide neutralizing agent contributes from 28 parts to 98 parts of the total anionic surfactant by weight present in the spray-dried particle.

**[0039]** On addition of alkaline source, to the partly neutralized anionic surfactant in the aqueous mixture, the remaining unreacted acid form of the anionic surfactant reacts with the alkali metal silicate salt to form fully neutralized salt form of the anionic surfactant. The alkaline source may be the excess of alkali metal aluminate salt, alkali metal silicate salt or mixtures thereof.

**[0040]** Depending on the whether the organic carboxylic acid reacted with aluminate salt or the in-situ aluminium hydroxide, the order of addition may be changed to ensure formation of aluminium Citrate in the process and to ensure complete neutralisation of LAS acid.

**[0041]** In one embodiment of the present invention a fully neutralized anionic surfactant is added to the aqueous mixture. In this embodiment the liquid acid anionic surfactant precursor is reacted with an alkali metal hydroxide to form fully neutralized anionic surfactant salt before addition to the aqueous mixture. More preferably the liquid acid precursor of the anionic surfactant is partly or fully neutralized in-situ.

**[0042]** Typically, the deterative surfactant is present in the aqueous mixture when the aluminate salt and/or aluminium hydroxide is contacted with the organic carboxylic acid. Preferably when the aluminium hydroxide is formed in-situ by reacting an aluminium salt with an alkaline source (preferably alkali metal hydroxide), the aqueous mixture has deterative surfactant which is neutralized before the addition of the aluminium salt. Preferably the aluminium salt is aluminium sulphate. The order of addition is preferably to neutralize the acid form of the deterative surfactant by contacting the acid form of the deterative surfactant with an aqueous solution of alkali metal hydroxide. Following the neutralization, the next step is to add the aluminium salt which reacts with the excess of alkali metal hydroxide to form aluminium hydroxide. Thereafter the organic carboxylic acid is added to form the organic carboxylic acid salt of aluminium.

**[0043]** When the aluminate salt is used, preferably where the aluminate salt is sodium aluminate, the order of addition is preferably to contact water with the aluminate salt solution, followed by the addition of organic carboxylic acid to form the carboxylic acid salt of aluminium. Next the deterative surfactant is added in neutralized salt form or an acid form. When the deterative surfactant is added in the acid form, the alkali metal hydroxide solution is added to neutralize the acid form to the corresponding salt form of the deterative surfactant.

**[0044]** When the deterative surfactant is pH sensitive, that is those deterative surfactants which hydrolysis at low pH conditions, then it is preferred that the deterative surfactant is added to the aqueous slurry after the pH is raised to 7 or above. Non-limiting examples of the deterative surfactant is primary alkyl sulphate (PAS) surfactant. Preferably PAS has an alkyl chain length of C<sub>8</sub> to C<sub>18</sub>, more preferably with an alkyl chain length of C<sub>12</sub> to C<sub>14</sub>. Preferably the primary alkyl sulphate surfactant is linear or branched, preferably linear. Preferably the primary alkyl sulphate surfactant is substituted or unsubstituted.

**[0045]** Preferably the deterative surfactant is an anionic surfactant. Suitable anionic deterative surfactant includes sulphonate and sulphate surfactant. Suitable sulphonate surfactant include methyl ester sulphonate, alpha olefin sulphonate, alkyl benzene sulphonate, especially alkyl benzene sulphonate, preferably C<sub>10</sub> to C<sub>13</sub> alkyl benzene sulphonate. A preferred deterative anionic surfactant is linear alkyl benzene sulphonate, where the alkyl chain has 5 to 20 carbon atoms, more preferably the linear alkylbenzene sulphonate surfactant has a C<sub>10</sub> to C<sub>18</sub> alkyl group, still preferably C<sub>10</sub> to C<sub>14</sub> alkyl group. Suitable alkyl benzene sulphonate (LAS) is obtainable, preferably obtained, by sulphonating commercially available linear alkyl benzene (LAB); suitable LAB includes low 2-phenyl LAB, other suitable LAB includes high 2-phenyl LAB, such as those supplied by Sasol under the tradename Hyblene®. Suitable sulphate surfactants include alkyl sulphate, preferably C<sub>8</sub> to C<sub>18</sub> alkyl sulphate, or predominantly C<sub>12</sub> to C<sub>18</sub> alkyl sulphate. One or more anionic surfactant may be present in the spray-dried detergent particle.

**[0046]** Suitable sulphate surfactant includes alkyl sulphate, preferably C<sub>8</sub> to C<sub>18</sub> alkyl sulphate, or predominantly C<sub>12</sub> to C<sub>18</sub> alkyl sulphate. A preferred sulphate deterative surfactant is alkyl alkoxyated sulphate, preferably alkyl ethoxyated sulphate, preferably a C<sub>8</sub> to C<sub>18</sub> alkyl alkoxyated sulphate, preferably a C<sub>8</sub> to C<sub>18</sub> alkyl ethoxyated sulphate, preferably the alkyl alkoxyated sulphate has an average degree of alkoxylation of from 0.5 to 20, preferably from 0.5 to 10, preferably the alkyl alkoxyated sulphate is a C<sub>8</sub> to C<sub>18</sub> alkyl ethoxyated sulphate having an average degree of ethoxylation of from 0.5 to 10, preferably from 0.5 to 5, more preferably from 0.5 to 3 and most preferably from 0.5 to 1.5. The alkyl sulphate, alkyl alkoxyated sulphate and alkyl benzene sulphonate may be linear or branched, substituted or un-substituted and may be derived from petrochemical material or biomaterial. Other suitable anionic deterative surfactant include, soaps, alkyl ether carboxylates. Suitable anionic deterative surfactant may be in salt form, suitable counter-ions include sodium, calcium, magnesium, amino alcohols, and any combinations thereof. A preferred counterion is sodium.

**[0047]** Suitable non-ionic deterative surfactant are selected from the group consisting of: C<sub>8</sub> to C<sub>18</sub> alkyl ethoxylates, such as, NEODOL® non-ionic surfactants from Shell; C<sub>6</sub> to C<sub>12</sub> alkyl phenol alkoxyates wherein preferably the alkoxyate units are ethyleneoxy units, propyleneoxy units or a mixture thereof; C<sub>12</sub> to C<sub>18</sub> alcohol and C<sub>6</sub> to C<sub>12</sub> alkyl phenol condensates with ethylene oxide/propylene oxide block polymers such as Pluronic® from BASF; alkyl polysaccharides, preferably alkyl polyglycosides; methyl ester ethoxylates; polyhydroxy fatty acid amides; ether capped poly(oxyalkylated) alcohol surfactants and mixtures thereof.

**[0048]** Suitable non-ionic deterative surfactants are alkyl polyglucoside and/or an alkyl alkoxyated alcohol. Suitable non-ionic deterative surfactants include alkyl alkoxyated alcohols, preferably C<sub>8</sub> to C<sub>18</sub> alkyl alkoxyated alcohol, preferably a C<sub>8</sub> to C<sub>18</sub> alkyl ethoxyated alcohol, preferably the alkyl alkoxyated alcohol has an average degree of alkoxylation of from 1 to 50, preferably from 1 to 30, or from 1 to 20, or from 1 to 10, preferably the alkyl alkoxyated alcohol is a C<sub>8</sub> to C<sub>18</sub> alkyl ethoxyated alcohol having an average degree of ethoxylation of from 1 to 10, preferably from 1 to 7, more preferably from 1 to 5 and most preferably from 3 to 7. The alkyl alkoxyated alcohol can be linear or branched and substituted or unsubstituted. Suitable nonionic deterative surfactants include secondary alcohol-based deterative surfactants.

**[0049]** Zwitterionic surfactant: Suitable zwitterionic deterative surfactants include amine oxides and/or betaines.

**[0050]** One or more deterative surfactant may be present in the spray-dried detergent particle according to the present invention. The surfactant is preferably those which are thermally stable during spray-drying processing conditions in a spray-drying tower with inlet air temperature ranging from 250°C to 500°C and those which are chemically stable at the pH conditions of the spray-drying slurry. Non-limiting examples of the anionic surfactant includes the ones mentioned above.

**[0051]** The pH of the aqueous mixture is preferably less than 4, more preferably the pH of the base mixture is from 2 to 3.5. The aqueous mixture preferably has 0 wt.% zeolite. The present inventors have found that aqueous mixture when spray dried directly provides a spray dried detergent particle which does not have prolonged shelf life. They further found that such spray dried detergent particle has an unpleasant odour. Without being bound by any theory, it is believed that the extremely low pH of the aqueous mixture makes the resultant spray-dried detergent particle prone to caking upon extended storage periods.

#### Step (ii): Forming an aqueous slurry:

**[0052]** In the next step, an aqueous slurry is formed by adding an amount of alkaline source to the aqueous mixture. The aqueous slurry has a pH of 4 to 8.5. The alkaline source may include any salt which enables the pH to be adjusted to 4 to 8.5. More preferably the alkaline source is selected from the group consisting of alkali metal silicate, alkali metal hydroxide or mixtures thereof. Preferably sodium silicate, potassium silicate, sodium hydroxide, potassium hydroxide or mixtures thereof. Preferably the alkaline source is selected from sodium silicate or sodium hydroxide or mixtures thereof. Preferably the alkali metal silicate salt employed is sodium silicate. The alkali metal silicate salt preferably has a weight ratio of SiO<sub>2</sub>:M<sub>2</sub>O where M is an alkali metal, within the range of 1.6 to 3.3 more preferably 1.6 to 2.4, and most preferably 2.0 to 2.85. The alkali metal silicate salt employed is in the form of an aqueous solution, generally having 30 wt.% to 45 wt.% solid content.

**[0053]** Preferably the alkaline source is alkali metal hydroxide which is added to the aqueous mixture to raise the pH upto 6.5, thereafter the pH is raised from 6.5 to 8.5 using alkali metal silicate. Preferably the alkali metal hydroxide is sodium hydroxide. Preferably the alkali metal silicate is sodium silicate. Alternately the alkaline source may include only alkali metal hydroxide to raise the pH of the aqueous mixture to 8.5, still preferably upto 7.

**[0054]** The aqueous mixture typically has some amount of unreacted organic carboxylic acid, which upon addition of the alkaline source reacts with the alkaline source to form organic carboxylic acid salt of an alkali metal in the aqueous slurry. The organic carboxylic acid salt of an alkali metal is preferably selected from the group consisting of mono alkali metal, dialkali metal, tri alkali metal salt of organic carboxylic acid salt or mixtures thereof.

**[0055]** Preferably when the alkaline source is alkaline silicate it is added in an amount to neutralize the organic carboxylic acid to form a citrate salt and silica, in the aqueous slurry the alkaline silicate is completely neutralized, and no residual alkaline silicate exists.

**[0056]** Preferably the aqueous slurry has from 0 wt.% to 10 wt.% organic carboxylic acid salt of an alkali metal, preferably a mixture of organic carboxylic acid salt of a dialkali metal and an organic carboxylic acid salt of a trialkali metal. The aqueous slurry preferably includes from 0 wt.% to 5 wt.% organic carboxylic acid salt of a dialkali metal, still preferably from 0 wt.% to 4.5 wt.% of the organic carboxylic acid salt of a dialkali metal. The aqueous slurry preferably includes from 0 wt.% to 3.5 wt.% organic carboxylic acid salt of a trialkali metal, still preferably from 0 wt.% to 3 wt.%, still more preferably 0 to 2.9 wt.% of the organic carboxylic acid salt of a trialkali metal. Preferably the organic carboxylic acid salt of an alkali metal includes disodium citrate, trisodium citrate, mono sodium citrate and mixtures thereof. Preferably the aqueous slurry comprises aluminium hydroxide. More preferably from 0 wt.% to 1 wt.% aluminium hydroxide.

Filler:

**[0057]** Preferably the aqueous slurry includes from a filler selected from the group consisting of sodium sulphate, sodium chloride, calcium carbonate, magnesium carbonate, calcite, dolomite, or mixtures thereof. The filler acts as a balancing ingredient and can be a neutral inorganic salt or mineral, preferably sodium sulphate or sodium chloride. In one preferred embodiment, the filler is sodium chloride. In another embodiment the filler is a mixture of sodium chloride and sodium sulphate. More preferably the filler is sodium sulphate.

**[0058]** The aqueous slurry prepared according to the process of the first aspect of the present invention preferably

comprises:

- (i) from 2 wt.% to 35 wt.% deterative surfactant;
- (ii) from 0.2 wt.% to 8 wt.% of one or more of organic carboxylic acid salts of aluminium, aluminium complex of organic carboxylic acid or mixtures thereof;
- (iii) preferably from 0 to 10 wt.% organic carboxylic acid salt of alkali metal;
- (iv) preferably 0 wt.% to 2wt.% silicate salt of alkali metal;
- (v) preferably from 0 wt.% to 1 wt.% aluminium hydroxide;
- (vi) preferably from 15 wt.% to 70 wt.% filler; and,
- (vii) from 20 to 40 wt.% water.

**[0059]** Preferably the amount of deterative surfactant in the aqueous slurry is not less than 2.5 wt.%, still preferably not less than 5 wt.%, more preferably not less than 8 wt.%, still more preferably not less than 10 wt.%, but typically not more than 35 wt.%, preferably not more than 34 wt.% or still preferably not more than 32 wt.%.

**[0060]** Preferably the amount of one or more of organic carboxylic acid salts of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof in the aqueous slurry is not less than 0.25 wt.%, still preferably not less than 0.3 wt.%, more preferably not less than 0.35 wt.%, still more preferably not less than 0.4 wt.%, but typically not more than 9 wt.%, preferably not more than 8 wt.% or still preferably not more than 7 most preferably not more than 6.5 wt.%.

**[0061]** Preferably the amount of organic carboxylic acid salt of alkali metal in the aqueous slurry is not less than 1 wt.%, still preferably not less than 1.5 wt.%, more preferably not less than 2, still more preferably not less than 2.5 wt.%, furthermore preferably not less than 3 wt.%, but typically not more than 9.5 wt.%, preferably not more than 9 wt.% or still preferably not more than 8.5 wt.%, more preferably not more than 7.5 wt.%.

**[0062]** Preferably the amount of silicate salt of alkali metal in the aqueous slurry is not less than 0.1 wt.%, still preferably not less than 0.2 wt.%, more preferably not less than 0.25 wt.%, still more preferably not less than 0.5 wt.%, but typically not more than 2 wt.%, preferably not more than 1.8 wt.% or still preferably not more than 1 wt.%. Preferably the aqueous slurry has less than 2 wt.% alkali metal silicate, still preferably less than 1 wt.%, further preferably 0 wt.% alkali metal silicate.

**[0063]** Preferably the amount of aluminium hydroxide is not less than 0.02 wt.%, still preferably not less than 0.04 wt.%, more preferably not less than 0.05 wt.%, still more preferably not less than 0.1 wt.%, but typically not more than 0.4 wt.%, preferably not more than 0.5 wt.% or still preferably not more than 0.9 wt.%.

**[0064]** Preferably the amount of water is not less than 22 wt.%, still preferably not less than 23 wt.%, more preferably not less than 24 wt.%, still more preferably not less than 25 wt.%, but typically not more than 40 wt.%, preferably not more than 37 wt.% or still preferably not more than 35 wt.%.

**[0065]** Preferably the filler is present in an amount ranging from 15 wt.% to 70 wt.% in the slurry. Preferably the amount of filler is not less than 16 wt.%, still preferably not less than 18wt.%, more preferably not less than 20 wt.%, still more preferably not less than 22 wt.%, but typically not more than 70 wt.%, preferably not more than 68 wt.% or still preferably not more than 65 wt.%.

**[0066]** Additionally, one or more of optional ingredients may be present in the aqueous slurry. The optional ingredients may include but it not limited to polymer, optical brighteners which is preferably selected from fluorescers, colourants, shading dye, pigments, hydrotropes, or mixtures thereof and antifoams.

**[0067]** Optionally the aqueous slurry includes silica. Preferably the silica is present in an amount ranging from 0 wt.% to 1 wt.%, still preferably from 0 wt.% to 0.8 wt.%. The silica may be generated in-situ. Although not essential, when alkaline silicate source is used for providing alkalinity in the process, silica may be formed in-situ.

**[0068]** Preferably the aqueous slurry has 0 wt.% to 2 wt.% alkali metal silicate. Preferably the aqueous slurry has less than 2 wt.% alkali metal silicate, still preferably less than 1 wt.%, further preferably 0 wt.% alkali metal silicate.

**[0069]** Preferably the aqueous slurry has less than 2 wt.% alkali metal carbonate builder, still preferably less than 1 wt.%, further preferably 0 wt.% carbonate builder. Preferably the aqueous slurry has 0 wt.% to 2 wt.% alkali metal carbonate builder. Examples of the carbonate builder salt includes alkaline earth metal and alkali metal carbonates or mixtures thereof. Typically, the alkali metal carbonates are sodium and/or potassium carbonate of which sodium carbonate is most preferred. Alkali metal carbonate according to the invention refers to carbonates, bicarbonates, sesquicarbonates or mixtures thereof. Preferably the alkali metal carbonate includes sodium carbonate. The amount of alkali metal carbonate builder is kept low to avoid potential release of the carbon dioxide in the slurry. Further it was found that addition of the alkali metal carbonate to the slurry increases the pH above 8.5 and at these pH levels the slurry becomes difficult to process.

**[0070]** Preferably the aqueous slurry has less than 2 wt.% inorganic phosphate builder, still preferably less than 1 wt.%, further preferably 0 wt.% inorganic phosphate builder. Preferably the aqueous slurry has 0 wt.% to 2 wt.% phosphate builder. Examples of inorganic phosphate builder includes sodium orthophosphate, pyrophosphate and tripolyphosphate.

**[0071]** Preferably the aqueous slurry has less than 2 wt.% zeolite builder, still preferably less than 1 wt.%, further preferably 0 wt.% zeolite builder. Preferably the aqueous slurry has 0 wt.% to 2 wt.% zeolite builder. Examples of the zeolite builder includes zeolite A, zeolite 4A, aluminium zeolite P (zeolite MAP) described and claimed in EP 384 070A (Unilever).



Zeolite MAP is an alkali metal aluminosilicate of the P type having a silicon to aluminium ratio not exceeding 1.33, preferably not exceeding 1.15, and more preferably not exceeding 1.07.

**[0072]** The aqueous slurry may optionally include a polymer. Suitable polymers include carboxylate polymers, soil release polymers, anti-redeposition polymers, cellulosic polymers, care polymers and any combination thereof. Preferably the polymer is a carboxylate polymer. Preferably the carboxylate polymer is a homopolymer or a copolymer. Preferably the copolymer is a maleate/acrylate random copolymer. Preferably the maleate/acrylate random copolymer has a molecular weight ranging from 1,000 Da to 100,000 Da, more preferably from 2,000 Da to 100,000 Da, still preferably 30,000 Da to 100,000 Da, or from 50,000 Da to 100,000 Da, or from 60,000 Da to 80,000 Da. Preferably the homopolymer is a polyacrylate. Preferably the polyacrylate homopolymers has a molecular weight ranging from 4,000 Da to 9,000 Da.

**[0073]** The aqueous slurry may preferably include powder structuring agents. Non-limiting examples of the powder structuring agents includes a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate polymer. the powder structuring agent is present in an amount of 1 wt.% to 5 wt.% in the aqueous slurry.

**[0074]** The aqueous slurry preferably includes less than 5 wt.% organic carboxylic acid, still preferably less than 4 wt.%, further preferably less than 3 wt.%, more preferably less than 1 wt.%, still more preferably the aqueous slurry has 0 wt.% organic carboxylic acid.

**[0075]** Further optional ingredients may be added to the aqueous slurry which includes but are not limited to, any one or more of the following: soap, sequestrants, calcium chloride, other inorganic salts, fluorescers, foam controllers, foam boosters, dyes, anti-redeposition agents, colourants, shading dyes, hydrotrope, viscosity modifiers, dispersants, and combinations thereof. Non-limiting examples of hydrotropes are preferably selected from the group consisting of sodium toluene sulphonate, sodium cumene sulphonate, sodium xylene sulphonate or mixtures thereof. Preferably these optional ingredients must have the ability to withstand the temperature conditions in a spray-drying process. Preferably a filler may be added to the aqueous slurry before spray-drying. Preferably the antiredeposition agent is sodium carboxymethyl cellulose.

#### Step (iv): Forming the spray-dried detergent particle

**[0076]** In the next step, the aqueous slurry is spray dried to form a spray-dried detergent particle.

**[0077]** The spray-drying is carried out using any of the conventional spray drying system known in the art. Preferably in the spray drying system the aqueous slurry is transferred through a pipe system to a pump system consisting of one or more pump and then further to a spray nozzle through which the slurry is released under pressure into a drying tower.

**[0078]** A typical spray-drying process involves the step of transferring the aqueous slurry through a pipe system leading to a first pump and then through a second pump and from a second pump to a plurality of spray nozzles. The first pump is typically a low-pressure pump, such as a pump that can generate a pressure of from  $1 \times 10^5 \text{ Nm}^{-2}$  to  $1 \times 10^6 \text{ Nm}^{-2}$ , which ensures proper flooding of the second pump. Typically, the second pump is a high-pressure pump, such as a pump can generate a pressure ranging from  $2 \times 10^6 \text{ Nm}^{-2}$  to  $2 \times 10^7 \text{ Nm}^{-2}$ . Optionally, the aqueous slurry may be transferred through bolt catchers, magnetic filters, lump breakers, disintegrators such as the Ritz Mill, during the transfer of the aqueous slurry through the pipe system downstream the pump system or the mixer in which the aqueous slurry is formed. The disintegrator is preferably positioned between the pumps. The flow rate of the aqueous slurry along the pipes is typically in the range from 800 Kg/hour to more than 75,000 Kg/hour.

**[0079]** Optionally, the spray drying system may include a deaeration system. The deaeration system is preferably a vacuum assisted de-aerator, which is preferably fed by a transfer pump. The deaeration system remove air bubbles formed during the slurry preparation, thus increasing the bulk density of the spray-dried detergent particle. De-aeration of the slurry may also be carried out by other mechanical means or chemical de-aeration means using antifoams or de-foamers.

**[0080]** Optionally, air injection system may be provided along the pipe system. The air injection system may be provided before or after the pump system. The air injection includes air flow and pressure controls, static mixer, pulsation dampener and compressor set which can aerate the slurry to get a lower bulk density for the spray dried particle. The gas injected into the slurry may be nitrogen, carbon dioxide, or simply atmospheric air introduced under a pressure higher than the pressure of the aqueous slurry maintained in the pipe system. A typical spray drying system can optionally include both the de-aeration system and air injection system to optimize the desired bulk density of the spray dried detergent particle.

**[0081]** Typical spray drying tower for detergent applications are counter-current spray drying tower. To obtain the desired moisture content and the particle size distribution the inlet hot air and/or hot steam temperature introduced into the spray drying tower is the range from 250°C to 500°C depending on the evaporation capacity and sizing of the tower. Preferably the tower exhaust air temperature can range from, 60°C to 200°C, more preferably 80°C to 200°C, still more preferably 80°C to 100°C depending on the loading of the tower. The aqueous slurry introduced into the spray nozzle of the spray drying tower is preferably at a temperature ranging from 60°C to 95°C. The spray drying tower may be a co-current spray drying tower but they are less common. The spray-dried detergent particle existing the tower is maintained at a temperature less than 150°C, still preferably less than 100°C. The spray-drying is preferably conducted in the spray drying zone under a negative pressure of at least  $50 \text{ Nm}^{-2}$ , still preferably the negative pressure is from  $50 \text{ Nm}^{-2}$  to  $600 \text{ Nm}^{-2}$ .

Preferably, the vacuum conditions are achieved by controlling the speed and/or dampener setting of the dampener of either or both the inlet and the outlet fans.

**[0082]** The spray-dried detergent particle collected at the bottom of the tower may be subjected to cooling and conditioning by using an air lift or other similar process known to a person skilled in the art for cooling and conditioning spray dried particle. The spray-dried detergent particle collected from the bottom of the spray-drying tower is preferably mixed with a flow aid chosen from zeolite, silica, precipitated calcite, sodium carbonate, salt, or similar fine mineral particles selected from the group consisting of dolomite, clay, calcite or mixtures thereof, just before being air-lifted. Preferably the zeolite is synthetically prepared. Preferably, the spray-dried detergent particle is subject to particle size classification to remove oversize material (> 2 mm typically) to provide a spray dried detergent particle which is free flowing. Preferably the fine material (< 100 microns typically) is elutriated with the exhaust air in the spray drying tower and captured and recycled back into the system via the dry cyclone, wet cyclone or bag filter system.

Spray-dried detergent particle:

**[0083]** According to a second aspect of the present invention disclosed is a spray-dried particle obtainable by the process of the first aspect. Spray-dried particle formed from the process of the first aspect of the present invention preferably has a pH of 4 or more, preferably a pH ranging from 4 to 8.5, more preferably from 5 to 8.5, still preferably 6 to 8.5 when measured using a 1 wt.% solution with distilled water at 25°C. The spray-dried detergent particle is generally referred to as the base powder. This base powder may be used as a fully formulated laundry detergent composition. Alternately a percentage of the base powder may be mixed with other post dosed ingredients to form the fully formulated laundry detergent composition.

**[0084]** Preferably the spray-dried detergent particle includes:

- (i) from 3 wt.% to 50 wt.% deterative surfactant; preferably anionic deterative surfactant;
- (ii) from 0.4 wt.% to 10 wt.% one or more of organic carboxylic acid salts of aluminium, aluminium complex of organic carboxylic acid or mixtures thereof;
- (iii) preferably from 0 wt.% to 12 wt.% organic carboxylic acid salt of alkali metal;
- (iv) preferably from 0 wt.% to 2 wt.% silicate salt of alkali metal;
- (v) preferably from 0 wt.% to 2 wt.% aluminium hydroxide;
- (vi) preferably 25 wt.% to 88 wt.% filler;
- (vii) preferably from 1 wt.% to 3.5 wt.% moisture content.

**[0085]** Preferably the amount of deterative surfactant in the spray dried detergent particle is not less than 3 wt.%, still preferably not less than 5 wt.%, more preferably not less than 8 wt.%, still more preferably not less than 10 wt.%, but typically not more than 45 wt.%, preferably not more than 40 wt.% or still preferably not more than 30 wt.%.

**[0086]** Preferably the amount of one or more of the organic carboxylic acid salts of aluminium, aluminium complex of organic carboxylic acid or mixtures thereof in the spray dried detergent particle is not less than 0.45 wt.%, still preferably not less than 1 wt.%, more preferably not less than 1.5 wt.%, still more preferably not less than 2 wt.%, but typically not more than 9.5 wt.%, preferably not more than 9 wt.% or still preferably not more than 8.5 wt.%.

**[0087]** Preferably the amount of organic carboxylic acid salt of alkali metal in the spray dried detergent particle is not less than 2.5 wt.%, still preferably not less than 3.5 wt.%, more preferably not less than 5, still more preferably not less than 5.5 wt.%, furthermore preferably not less than 6 wt.%, but typically not more than 11.5 wt.%, preferably not more than 11 wt.% or still preferably not more than 10 wt.%, more preferably not more than 8 wt.%.

**[0088]** Preferably the amount of silicate salt of alkali metal in the spray dried detergent particle is not less than 0.1 wt.%, still preferably not less than 0.2 wt.%, more preferably not less than 0.25 wt.%, still more preferably not less than 0.3 wt.%, but typically not more than 1 wt.%, preferably not more than 2.5 wt.% or still preferably not more than 2 wt.%.

**[0089]** Preferably the amount of aluminium hydroxide in the spray dried detergent particle is not less than 0.1 wt.%, still preferably not less than 0.25 wt.%, more preferably not less than 0.8 wt.%, still more preferably not less than 1 wt.%, but typically not more than 1.8 wt.%, preferably not more than 1.75 wt.% or still preferably not more than 1.5 wt.%.

**[0090]** Preferably the amount of water in the spray dried detergent particle is not less than 0.5 wt.%, still preferably not less than 1 wt.%, more preferably not less than 1.5 wt.%, still more preferably not less than 2 wt.%, but typically not more than 3 wt.%, preferably not more than 2.75 wt.% or still preferably not more than 2.5 wt.%.

**[0091]** Preferably the filler is present in the spray dried detergent particle an amount ranging from 25 wt.% to 88 wt.%. Preferably the amount of filler is not less than 26 wt.%, still preferably not less than 30 wt.%, more preferably not less than 32 wt.%, still more preferably not less than 35 wt.%, but typically not more than 87 wt.%, preferably not more than 75 wt.% or still preferably not more than 65 wt.%. The filler acts as a balancing ingredient and can be a neutral inorganic salt or mineral, preferably sodium sulphate or sodium chloride. In one preferred embodiment, the filler is sodium chloride.

**[0092]** Additionally, one or more of optional ingredients may be present in the spray-dried detergent particle. The

optional ingredients may include but it not limited to polymer, optical brighteners which is preferably selected from fluorescers, colourants, shading dye, hydrotropes, pigments, or mixtures thereof and antifoam. Non-limiting examples of hydrotropes are preferably selected from the group consisting of sodium toluene sulphonate, sodium cumene sulphonate, sodium xylene sulphonate or mixtures thereof.

**[0093]** Preferably the spray-dried detergent particle includes silica. Preferably the silica is present in an amount ranging from 0 wt.% to 2 wt.%, still preferably from 0.2 wt.% to 1.5 wt.%, further preferably from 0.2 wt.% to 1.3 wt.%, still more preferably 0.5 wt.% to 1.2 wt.% in the spray-dried detergent particle, still preferably the 0.5 wt.% to 1.5 wt.%, most preferably from 0 wt.% to 1.2 wt.%.

**[0094]** Preferably the spray-dried detergent particle has less than 2 wt.% alkali metal silicate, still preferably less than 1 wt.%, further preferably 0 wt.% alkali metal silicate. Preferably the spray-dried detergent particle has 0 wt.% to 2 wt.% alkali metal silicate

**[0095]** Preferably the spray-dried detergent particle has less than 2 wt.% carbonate builder, still preferably less than 1 wt.%, further preferably 0 wt.% carbonate builder. Preferably the spray-dried detergent particle has 0 wt.% to 2 wt.% carbonate builder. Examples of the carbonate builder salt includes alkaline earth metal and alkali metal carbonates or mixtures thereof. Typically, the alkali metal carbonates are sodium and/or potassium carbonate of which sodium carbonate is mostly preferred. Alkali metal carbonate according to the invention refers to carbonates, bicarbonates, sesquicarbonates or mixtures thereof.

**[0096]** Preferably the spray-dried detergent particle has less than 2 wt.% inorganic phosphate builder, still preferably less than 1 wt.%, further preferably 0 wt.% inorganic phosphate builder. Preferably the spray-dried detergent particle has 0 wt.% to 2 wt.% inorganic phosphate builder. Examples of inorganic phosphate builder includes sodium orthophosphate, pyrophosphate and tripolyphosphate.

**[0097]** Preferably the spray-dried detergent particle has less than 2 wt.% zeolite builder, still preferably less than 1 wt.%, further preferably 0 wt.% zeolite builder. Preferably the spray-dried detergent particle has 0 wt.% to 2 wt.% zeolite builder. Examples of the zeolite builder includes zeolite A, zeolite 4A, aluminium zeolite P (zeolite MAP) described and claimed in EP 384 070A (Unilever). Zeolite MAP is an alkali metal aluminosilicate of the P type having a silicon to aluminium ratio not exceeding 1.33, preferably not exceeding 1.15, and more preferably not exceeding 1.07.

**[0098]** Optionally, the spray-dried detergent particle includes from 0 wt.% to 4 wt.% polymer, still more preferably from 0.5 wt.% to 4 wt.% furthermore preferably from 0 to 0.5 wt.% polymer. Preferably the polymer is a polyacrylate polymer, still preferably a copolymer of acrylic acid or methacrylic acid with maleic acid. The spray dried detergent particle may include further polymer selected from antiredeposition polymer, soil release polymer, structuring polymer or mixtures thereof. Preferably the polymer is a polymeric carboxylate, preferably polyacrylate or a copolymer of acrylic acid and maleic acid. However other polymers may also be suitable such as polyamines (including the ethoxylated variants thereof), polyethylene glycol and polyesters. Polymeric soil suspending aids and polymeric soil release agents are particularly suitable.

**[0099]** The spray dried detergent particle may be optionally contacted with a non-ionic surfactant, a fatty acid, or combinations thereof. The non-ionic surfactant and the fatty acid is in liquid form. In addition to the non-ionic surfactant and the fatty acid in liquid form any other liquid laundry ingredient which is not suitable to be added via slurry or tower, may be added by spraying the liquid onto the spray-dried detergent particle. The sprayed liquid is soaked onto the hot base powder coming out of the tower. The spraying may be carried out while the spray-dried detergent particle passes through an inline low shear rotary drum, or an online densification kit which is typically a plough shear mixer.

**[0100]** After spray-drying, the collected spray-dried detergent particle is preferably layered with a layering agent. Preferably the layering agent is selected from the group consisting of zeolite, silica, precipitated calcite, sodium carbonate, salt, calcite, clay dolomite or mixtures thereof. Preferably the zeolite is synthetically prepared. Preferably the level of the layering agent added to the spray-dried particle is from 0 wt.% to 10 wt.% of the surfactant content present in the spray-dried detergent particle. Preferably the layering agent is added to give additional anticaking benefit. The spray-dried detergent particle mixed with layering agent is preferably referred to as base powder.

**[0101]** Preferably the spray-dried detergent particle has a bulk density of less than 550g/L. Preferably the spray-dried detergent particle has a weight average particle size ranging from 300 micrometres to 600 micrometres.

**[0102]** The spray-dried detergent particle comprises from 3 wt.% to 50 wt.% anionic surfactants, which is preferably a C<sub>10</sub> to C<sub>20</sub> linear alkyl benzene sulphonate and which is substantially neutralized with little or no acid residues.

**[0103]** The pH of the spray-dried detergent particle is preferably from 4 to 8.5, still preferably from 6 to 8.5, further preferably 7 or above, still preferably from 7 to 8.

**[0104]** The spray-dried laundry detergent particle is typically post dosed with ingredients that are incompatible with the spray-drying process conditions to form a fully formulated laundry detergent composition. These components may be incompatible for many reasons including heat sensitivity, pH sensitivity or degradation in aqueous systems.

Laundry detergent composition

**[0105]** According to a further aspect of the present invention, disclosed is a laundry detergent composition having the

spray-dried laundry detergent particle of the first aspect of the invention. Detergent compositions of low to moderate bulk density may be prepared by spray-drying the aqueous slurry to form a spray-dried particle and optionally postdosing (dry-mixing) further ingredients. Alternately "compact" detergent compositions may be prepared by further mixing the spray dried laundry detergent particle prepared according to the present invention in a highspeed mixer/granulator, or other non-tower processes. The spray dried detergent particle may also be used for preparing a tablet composition by compacting powders, especially "concentrated" powders using a known tableting process. Further, the spray dried detergent particle may be used for preparing an unit dose product wherein the spray-dried detergent particle is enclosed in a pouch, preferably a water-soluble pouch, more preferably a water-soluble pouch comprising a film forming polymer selected from polyvinyl alcohol, polyvinylpyrrolidone and other known film forming polymer.

**[0106]** The spray-dried detergent particle (generally also referred to as a base powder) is preferably formulated into a finished laundry detergent composition by dry mixing heat sensitive ingredients into the base powder. In addition to heat sensitive ingredients some amount of alkalinity may be added back into the base powder by addition of alkaline ingredients, additionally other acidic or neutral may also be added to formulate the finished laundry detergent composition.

**[0107]** The spray-dried detergent particle may be used as a fully formulated laundry detergent composition or may be additionally combined with other optional ingredients to form a fully formulated laundry detergent composition. Non-limiting examples of the optional post-dosed benefit ingredients includes but is not limited to enzymes, anti-redeposition polymers, perfumes, additional surfactant selected from amphoteric surfactant, zwitterionic surfactant, cationic surfactant and non-ionic surfactant, optical brighteners, antifoaming agent, foam boosters, fabric softeners such as smectite clays, amine softeners and cationic softeners; bleach and bleach activators; dyes or pigments, fillers, fluorescers, salts, soil release polymers, dye transfer inhibitors. Preferably the antiredeposition agent is sodium carboxymethyl cellulose.

**[0108]** These optional ingredients are well known to be used in a laundry detergent composition and added preferably by post-dosing.

**[0109]** The laundry detergent composition includes from 5 wt.% to 100 wt.% spray-dried detergent particle obtainable according to the first aspect of the present invention. More preferably from 20 wt.% to 95 wt.%, still preferably 30 wt.% to 95 wt.% of the spray-dried detergent particle obtainable according to the first aspect of the present invention.

**[0110]** Non-limiting examples of the post-dosed polymers include cleaning polymers, antiredeposition polymers, soil release polymers structuring polymers. Some examples include PET-PEOT polymer (Repel-o-Tex® SF2 ex. Solvay), copolymer of acrylic acid and maleic acid (Sokalan CP5 ex. BASF).

#### Fluorescers

**[0111]** Suitable fluorescent brighteners include dis-styryl biphenyl compounds example Tinopal® CBS-X, di-amino stilbene di-sulfonic acid compounds, e.g. Tinopal® DMS pure Xtra and Blankophor® HRH, and Pyrazoline compounds, e.g. Blankophor® SN, and coumarin compounds, e.g. Tinopal® SWN. Preferred brighteners 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. A suitable fluorescent brightener is S C.I. Fluorescent Brightener 260, which may be used in its beta or alpha crystalline forms, or a mixture of these forms.

#### Enzymes:

**[0112]** The composition of the present invention preferably includes an enzyme. It may preferably include one or more enzymes. Preferred examples of the enzymes include those which provide cleaning performance and/or fabric care benefits.

**[0113]** Examples of suitable enzymes include, but are not limited to, hemicellulases, peroxidases, proteases, cellulases, xylanases, lipases, xyloglucanase, phospholipases, esterases, cutinases, pectinases, mannanases, pectate lyases, keratinases, reductases, oxidases, phenoloxidases, lipoxigenases, ligninases, pullulanases, tannases, pentosanases, malanases, G-glucanases, arabinosidases, hyaluronidase, chondroitinase, laccase, and amylases, or mixtures thereof. A typical combination is an enzyme cocktail that may comprise, for example, a protease and lipase in conjunction with one or more of amylase, mannanase and cellulase. When present in a detergent composition, the enzymes may be present at levels from about 0.00001 % to about 2%, from about 0.0001 % to about 1% or from 0.001 % to about 0.5% enzyme protein by weight of the detergent composition.

#### Packaging and dosing:

**[0114]** According to another aspect of the present invention, disclosed is a packaged article comprising a flexible container enclosing a laundry detergent composition having a spray dried detergent particle prepared according to the first aspect of the present invention. The flexible container is preferably made from a packaging material suitable for packaging

laundry detergent composition and the packaging material includes but not limited to polyolefin film, laminates, paper based films or laminates, multilayered structures include two or more flexible structures and other materials known to a person skilled in the art. Typically the flexible container includes polyethylene films having polymer selected from HDPE, LLDPE, mLLDPE, LDPE or combination thereof. The flexible films may be made of monomaterial or combination of different materials. Preferably the flexible packaging container is a flexible pouch or a bag. It is preferably selected from a material which is biodegradable, compostable, recyclable or combinations of those. The flexible container may preferably include a measurement means which may be supplied with the package either as a part of the closure of the container or as an integrated system or a separate dosing measure may be provided along with the package.

**[0115]** In yet another embodiment the laundry detergent composition comprising the spray dried detergent particle of the present invention may be packaged as unit dose product enclosed within a polymeric film, wherein the polymeric film is water soluble or disintegrates upon addition to the wash water. Alternatively, the spray-dried detergent particle or a laundry detergent composition including the spray-dried particle of the invention may be supplied in multidose plastics packs with a top or bottom closure.

**[0116]** According to another aspect of the present invention, provided is a method of laundering fabric using a spray dried detergent particle or a laundry composition comprising a spray dried detergent particle according to the present invention which method involves the step of diluting an amount of the laundry detergent composition with water to obtain a wash liquor followed by the step of washing fabrics with the wash liquor so formed. In automatic washing machines a measured amount of detergent composition (dose) is typically put into a dispenser and from there it is flushed into the machine by the water flowing into the machine, thereby forming the wash liquor. From 5 up to about 65 litres of water may be used to form the wash liquor depending on the machine configuration. The dose of detergent composition may be adjusted accordingly to give appropriate wash liquor concentration. The dilution step preferably provides a wash liquor having from 3 to 20 g/wash of deterative surfactants (as are further defined above).

**[0117]** The product obtainable by a process according to the present invention has different product characteristics due to its process of manufacture. The product has good physical properties, such as good cake strength and good flowability. The product also has good dispensing properties, dissolution properties, and minimal fabric residue deposition. This is due to the in-situ formation of organic carboxylic acid of alkaline earth metal.

## Examples

### Example 1: Process for preparing the spray-dried detergent particle according to the present invention

**[0118]** A spray-dried detergent particle according to the present invention was prepared by adding water and sodium aluminate was added to the aqueous mixture. The sodium aluminate used had an  $Al_2O_3$  content of around 54% and Na<sub>2</sub>O content of around 40%.

**[0119]** The acidification of the aqueous mixture was done with citric acid to bring the pH down to 3. The sodium aluminate reacted with the citric acid in the aqueous mixture to form aluminium citrates and some amount of unreacted aluminium hydroxide.

**[0120]** Thereafter the alkaline source (sodium hydroxide, 50% active content) and acid form of the anionic surfactant (LAS acid, 97% active content) were added to the mixing unit to form neutralized NaLAS.

**[0121]** Thereafter the pH of the mixture was increased by adding an alkaline source (sodium hydroxide) where the sodium hydroxide reacted with the citric acid to form sodium citrate. The pH of the end slurry was later raised by using caustic. The final pH of the aqueous slurry was maintained at 7.0.

Table 1

Preparing an aqueous slurry according to the invention					
Ingredients		Order of addition	Quantity in kg	Temp (deg, C)	pH
Water	Heating off	1	322.73	40	
Sodium Aluminate (95%)		2	18.93	45	
Carboxylic acid (Citric Acid)		3	60.23	54	
Sodium hydroxide (50%)		4	67.34	62	
LAS Acid (97%)		5	260.16	86	
Sodium Sulphate (filler)	Heating On	7	538.33	85	3.5
Sodium hydroxide (50%)	Heating On	8	18.00	83	7

(continued)

Preparing an aqueous slurry according to the invention					
Ingredients		Order of addition	Quantity in kg	Temp (deg, C)	pH
Total slurry batch size (Kg)			<b>1286</b>		

**[0122]** The composition of the prepared aqueous slurry was as given below in Table 1A

Table 1A

Ingredients	Ex 1 wt. %
Na LAS	20.95
Al Citrates and/or aluminium complex of citric acid	2.21
Aluminium Hydroxide	0.35
Sodium citrates (citrates of disodium, monosodium or mixtures thereof)	3.46
Na Sulphate	40.54
Water	32.04
NDOM	0.44
pH	7
<b>TOTAL</b>	<b>100</b>

**[0123]** Next the above aqueous detergent slurry was spray dried in a counter-current spray drying tower. The aqueous detergent slurry was heated to 80°C and pumped under pressure ( $7.5 \times 10^6 \text{ Nm}^{-2}$ ), into a counter current spray-drying tower with an air inlet temperature ranging from between 250°C to 330°C. The inlet fan was set to provide the tower inlet airflow of  $187,500 \text{ kg h}^{-1}$ . The exhaust fan was controlled to create a negative pressure in the tower of  $-200 \text{ Nm}^{-2}$  (typically the outlet air flow rate through the exhaust fan is between  $220,000 \text{ kg h}^{-1}$  to  $240,000 \text{ kg h}^{-1}$ , this includes the evaporated water from the slurry). The aqueous slurry was atomised into the tower where the atomised slurry was dried to produce a spray-dried detergent particle, which was then cooled and sieved to remove oversize material ( $> 1.8 \text{ mm}$ ) to form a spray-dried detergent particle, which was free-flowing. Fine material ( $< 0.175 \text{ mm}$ ) was elutriated along with the exhaust air in the spray-drying tower and collected in a post tower containment system. The spray-dried detergent particle has a moisture content of 2.0 wt.%, a bulk density of 350g/L and a particle size distribution such that greater than 90 wt. % of the spray-dried detergent particle has a particle size of from 175 to 710 micrometres. The temperature of the spray-dried detergent particle exiting the tower has a temperature of below 150°C. The composition of the spray-dried detergent particle obtained by spray-drying the aqueous detergent slurry is given below. The spray-dried detergent particle may preferably be mixed with 3 wt. % zeolite as the layering agent while exiting the tower before the air lift to form the base powder.

Table 2

Spray dried detergent particle according to the present invention	
Ingredients	Ex 1 (wt. %)
Na LAS	30
Aluminium citrates and/or aluminium complex of citric acid	3.18
Aluminium hydroxide	0.5
Sodium citrates (disodium citrate, trisodium citrate or mixtures thereof)	5.0
Sodium Sulphate	58.47
Sodium Silicate	0
Sodium carbonate	0
NDOM	0.64
Water	2

(continued)

Spray dried detergent particle according to the present invention	
Ingredients	Ex 1 (wt.%)
pH	7
<b>TOTAL</b>	<b>100</b>

**[0124]** The spray-dried particle was collected and characterized for the storage behaviour.

#### Example 2: Evaluation of the storage properties of the spray-dried detergent particle

**[0125]** Spray-dried detergent particle according to the present invention (Ex 1) was evaluated for the powder properties using the compression test method. The caking tendency of the spray dried particle was measured and compared with the comparative examples.

**[0126]** Compression Test Method: This test evaluates the tendency of the powder towards caking. A split cylinder with a polished internal surface was positioned on a firm base to form a hollow cylindrical mould with a diameter of 9 centimetres. Spray dried detergent particle to be tested was filled and levelled. A plastic disc was placed on the powder mass. A weight of 12 kilograms was slowly placed on the plastic disc in such a way that the weight was uniformly applied on the powder mass in the mould allowing it to compact. After 2 minutes the weight was removed, and the cylindrical mould was opened slowly without disturbing the compacted cake. Incremental weights of 200 grams were added at an interval of 10 seconds till the compacted cake collapses. Total vertical load required to collapse the formed cake was noted and expressed in kilograms and provides an indication of the caking tendency of the particle. Higher the value, greater is the caking tendency of the spray-dried detergent particle under evaluation. For the present evaluation, value lower than 1 Kilogram is considered good and any value above 2 Kilograms is classified as cohesive and the spray dried detergent particle is considered to have high caking tendency.

#### Example 2A: Evaluation of the storage behaviour of a spray dried detergent particle according to the present invention and comparative examples with higher pH

**[0127]** The storage behaviour of the spray dried detergent particle according to the present invention was evaluated along with comparative spray dried detergent particle having higher pH. In a control spray dried detergent particle (Control), both silicate and carbonate were added at conventional levels for providing good powder structuring properties. In a first comparative example (Ex A), high levels of carbonate were used for structuring, but the spray dried particle had no silicate. In a second comparative example (Ex B), carbonate levels of Ex A were maintained, and a low amount of silicate was also added. In a third comparative example (Ex C), silicate was used for providing the structuring and no carbonate was used.

**[0128]** The prepared slurries were processed and spray-dried under similar conditions as described in Example 1.

**[0129]** Next the collected spray-dried particle was packed and sealed in packs with a water vapour transmission <5 gram/m<sup>2</sup>/day. The packs were then stored for 8 weeks and 12 weeks at a temperature of 45°C and relative humidity of 80RH. The caking tendency of the spray-dried particle was determined by performing the compression test on the spray-dried particle, at different time intervals which were first immediately after collecting the (t=0), then after 8 weeks and then after 12 weeks.

**[0130]** The results were recorded and is provided in table 3 below.

Table 3

Comparison of inventive spray dried particle with high pH/alkaline particle						
	Spray dried detergent particle (wt.%)			Compression Test Values (in Kg)		
	Na LAS	Na silicate	Na carbonate	Freshly prepared	Post storage (in weeks)	
				t=0	After 8	After 12
Control	25	12	15	0.2 (200 g)	0.8 (800 g)	1 (1000 g)
Ex A	25	0	35	Slurry not processable		
Ex B	25	5	35	0.7 (700 g)	2.6 + (2600+ g)	Fully Caked
Ex C	25	12	0	2.45 (2450 g)	Fully Caked	Fully Caked

(continued)

Comparison of inventive spray dried particle with high pH/alkaline particle						
	Spray dried detergent particle (wt.%)			Compression Test Values (in Kg)		
	Na LAS	Na silicate	Na carbonate	Freshly prepared	Post storage (in weeks)	
				t=0	After 8	After 12
Ex 1	30	0	0	0.05 (50 g)	0.6 (600 g)	0.8 (800 g)

**[0131]** The data in table 3 shows that the spray-dried detergent particle according to the present invention having no carbonate and no silicate and structured using aluminium citrates and/or aluminium complex of citric acid provided good storage properties and the spray-dried particle was free flowing even after 12 weeks and performed slightly better than the control having higher pH and where both carbonate and silicate are incorporated.

**[0132]** In contrast, the comparative Ex A with high levels of carbonate but no silicate was not processable due to slurry thickening, while Ex B and Ex C did not show good storage properties.

**[0133]** From the above results it was found that even when having higher levels of the deterative surfactant (30 wt.% in the Ex 1 as compared to 25 wt.% in control and comparative examples), the spray-dried particle prepared according to the present invention had good powder properties and fared better in terms of caking propensity at T=zero as well as on prolonged storage.

Example 3: Process for preparing the spray-dried detergent particle according to the present invention wherein organic carboxylic acid is reacted with in-situ formed aluminium hydroxide (using aluminium salt)

**[0134]** A spray-dried detergent particle according to the present invention was prepared by adding water, alkaline source (sodium hydroxide, 50% active content) and acid form of the anionic surfactant (LAS acid, 97% active content) to a mixing unit to form an aqueous mixture. The ingredients were mixed well and while the agitation was continued the remaining ingredients were added in the order of addition as mentioned in the Table 4 below. During the mixing the temperature in the mixing unit was maintained with steam at 80°C +/-2°C. Thereafter aluminium sulphate was added to the aqueous mixture. The aluminium sulphate was an aqueous solution with 50% concentration. The aluminium sulphate was added into the alkaline aqueous mixture and in this alkaline condition aluminium sulphate reacted with sodium hydroxide and formed aluminium hydroxide and sodium sulphate. Next the acidification of the aqueous mixture was done with citric acid to bring the pH down to 3. The aluminium hydroxide formed from aluminium sulphate reacted with the citric acid in the aqueous mixture to form aluminium citrate and some amount of unreacted aluminium hydroxide.

**[0135]** Thereafter the pH of the mixture was increased by adding an alkaline source (sodium hydroxide) where the sodium hydroxide is added to raise the pH to 6, wherein the sodium hydroxide reacted with the citric acid to form sodium citrate. The pH of the end slurry was later raised by using sodium silicate. The final pH of the aqueous slurry was maintained at 8.5.

Table 4

Preparing an aqueous slurry according to the invention					
Ingredients		Order of addition	Quantity in kg	Temp (deg, C)	pH
Water	Heating off	1	363.16	40	
Sodium hydroxide (50%)		2	100.56	62	
LAS Acid (97%)		3	228.97	65	
Aluminium sulphate (50%)	Heating On	4	58.55	70	
Citric Acid		5	53.01	74	
Sodium Sulphate (filler)		6	29.67	77	3.5
Sodium hydroxide (50%)		7	45.51	80	6
Sodium Silicate (47%)		9	35.00	80	8.5
Total slurry batch size (Kg)			<b>1316</b>		

**[0136]** The composition of the prepared aqueous slurry was as given below in Table 1A



Table 4A

Ingredients	Ex 2 wt. %
Na LAS	18.03
Aluminium citrates and/or aluminium complex of citric acid	1.41
Aluminium Hydroxide	0.51
Sodium citrates (disodium citrate, trisodium citrate or mixtures thereof)	3.65
Na Sulphate	36.01
Sodium silicate	0.41
Sodium carbonate	0
NDOM	0.35
Water	39.62
pH	8.5
<b>TOTAL</b>	<b>100</b>

[0137] Next the above aqueous detergent slurry was spray dried in a counter-current spray drying tower (as described above) to provide a spray dried detergent particle as shown in table 5.

Table 5

Spray dried particle according to the present invention	
Ingredients	Ex 2 (wt. %)
Na LAS	30
Aluminium citrates and/or aluminium complex of citric acid	2.29
Aluminium hydroxide	0.83
Sodium citrates (disodium citrate, trisodium citrate or mixtures thereof)	5.93
Sodium Sulphate	57.72
Sodium Silicate	0.67
Sodium carbonate	0
NDOM	0.57
Water	2
<b>TOTAL</b>	<b>100</b>

[0138] The spray-dried particle was collected and characterized for the storage behaviour using compression test.

Table 6

Comparison of inventive spray dried particle with high pH/alkaline particle						
	Spray dried detergent particle (wt. %)			Compression Test Values (in Kg)		
	Na LAS	Na silicate	Na carbonate	Freshly prepared	Post storage (in weeks)	
				t=0	After 8	After 12
Control	25	12	15	0.2 (200 g)	0.8 (800 g)	1 (1000 g)
Ex A	25	0	35	Slurry not processable		
Ex B	25	5	35	0.7 (700 g)	2.6 + (2600+ g)	Fully Caked
Ex C	25	12	0	2.45 (2450 g)	Fully Caked	Fully Caked
Ex 2	30	0	0	0.05 (50 g)	0.8 (800 g)	1.4 (1400 g)

**[0139]** The data in table 6 shows that the spray-dried detergent particle according to the present invention (Ex 2) having no carbonate and no silicate and structured using aluminium citrates and/or aluminium complex of citric acid provided good storage properties and the powder was free flowing even after 12 weeks.

## Claims

1. A process for preparing a spray-dried laundry detergent particle comprising the steps of:

- (i) reacting an organic carboxylic acid with any one of (a) aluminate salt solution, (b) aluminium hydroxide solution, or mixtures thereof wherein the aluminium hydroxide solution is formed in-situ by any one of the reaction selected from the group consisting of (i) reacting sodium aluminate solution with an acid; or, (ii) reacting an aluminium salt with an alkaline source; to form one or more reaction product selected from the group consisting of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof;
- (ii) adding an amount of alkaline source to the aqueous mixture to provide an aqueous slurry having a pH of 4 to 8.5, wherein the aqueous slurry comprises one or more reaction product selected from the group consisting of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof and a deterative surfactant; and,
- (iii) spray-drying the aqueous slurry to form a spray-dried detergent particle.

2. A process according to claim 1 wherein the aqueous slurry further comprises an organic carboxylic acid salt of alkali metal.

3. A process according to claim 1 wherein the aluminium salt is selected from aluminium sulphate, aluminium chloride, sodium aluminate, or mixtures thereof.

4. A process according to claim 3 wherein the aluminium sulphate reacts with an alkaline source preferably sodium hydroxide to form aluminium hydroxide and preferably sodium aluminate.

5. A process according to claim any one of the preceding claims wherein the alkaline source in step (ii) added to provide an aqueous slurry is selected from the group consisting of alkali metal hydroxide, alkali metal silicate or mixtures thereof.

6. A process according to claim 5 wherein the alkali metal silicate has a  $\text{SiO}_2:\text{M}_2\text{O}$  weight ratio ranging from 1.6 to 3.3, more preferably 1.6 to 2.4 and most preferably 2.0 to 2.85.

7. A process according to any one of the preceding claims wherein the deterative surfactant is added to the aqueous mixture before or after the formation of the one or reaction products selected from the group consisting of carboxylic acid salt of aluminium, an aluminium complex of carboxylic acid or mixtures thereof.

8. A process according to any one of the preceding claims wherein the organic carboxylic acid is citric acid.

9. A process according to any one of the preceding claims wherein the aqueous slurry comprises not more than 2 wt.% alkali metal silicate.

10. A process according to any one of the preceding claims wherein the aqueous slurry comprises less than 3 wt.% phosphate builder.

11. A process according to any one of the preceding claims wherein the deterative surfactant is selected from the group consisting of anionic surfactant, nonionic surfactant, cationic surfactant, amphoteric surfactant, zwitterionic surfactant or mixtures thereof.

12. A process according to claim 11 wherein the anionic surfactant is preferably selected from the group consisting of alkyl benzene sulphonate, alkyl ether sulphate, alkyl sulfate or mixtures thereof.

13. A process according to any one of the preceding claims wherein aqueous slurry comprises:

- (i) from 2 wt.% to 35 wt.% deterative surfactant;

- (ii) from 0.2 wt.% to 8 wt.% of one or more of organic carboxylic acid salts of aluminium, aluminium complex of organic carboxylic acid or mixtures thereof;
- (iii) preferably from 0 to 10 wt.% organic carboxylic acid salt of alkali metal;
- (iv) preferably 0 wt.% to 2 wt.% silicate salt of alkali metal;
- (v) preferably from 0 wt.% to 1 wt.% aluminium hydroxide;
- (vi) preferably from 15 to 70 wt.% filler; and,
- (vii) from 20 to 40 wt.% water.

14. A spray-dried detergent particle obtainable according to any one of the preceding claims comprising:

- (i) from 3 wt.% to 50 wt.% deterative surfactant; preferably anionic deterative surfactant;
- (ii) from 0.4 wt.% to 10 wt.% one or more of organic carboxylic acid salts of aluminium, aluminium complex of organic carboxylic acid or mixtures thereof;
- (iii) preferably from 0 wt.% to 12 wt.% organic carboxylic acid salt of alkali metal;
- (iv) preferably from 0 wt.% to 2 wt.% silicate salt of alkali metal;
- (v) preferably from 0 wt.% to 2 wt.% aluminum hydroxide;
- (vi) preferably 25 wt.% to 88 wt.% filler; and,
- (vii) preferably from 1 wt.% to 3.5 wt.% moisture content.

15. A laundry detergent composition comprising 5 wt.% to 100 wt.% spray dried detergent particle according to claim 14.

### Patentansprüche

1. Verfahren zur Herstellung eines sprühgetrockneten Waschmittelpartikels, umfassend die Schritte:

(i) Umsetzen einer organischen Carbonsäure mit irgendeiner der Lösungen:

- (a) Aluminatsalzlösung,
  - (b) Aluminiumhydroxidlösung
- oder Mischungen davon,

wobei die Aluminiumhydroxidlösung in situ durch irgendeine der Reaktionen gebildet wird, ausgewählt aus der Gruppe, bestehend aus (i) Umsetzen einer Natriumaluminatlösung mit einer Säure oder (ii) Umsetzen eines Aluminiumsalzes mit einer alkalischen Quelle, um ein oder mehrere Reaktionsprodukte zu bilden, ausgewählt aus der Gruppe, bestehend aus organischem Carbonsäuresalz von Aluminium, einem Aluminiumkomplex organischer Carbonsäure oder Mischungen davon;

(ii) Hinzufügen einer Menge einer alkalischen Quelle zu der wässrigen Mischung, um eine wässrige Aufschlämmung mit einem pH-Wert von 4 bis 8,5 bereitzustellen, wobei die wässrige Aufschlämmung ein oder mehrere Reaktionsprodukte umfasst, ausgewählt aus der Gruppe, bestehend aus organischem Carbonsäuresalz von Aluminium, einem Aluminiumkomplex organischer Carbonsäure oder Mischungen davon und einem Reinigungstensid; und

(iii) Sprühtrocknen der wässrigen Aufschlämmung, um ein sprühgetrocknetes Waschmittelpartikel zu bilden.

2. Verfahren nach Anspruch 1, wobei die wässrige Aufschlämmung ferner ein organisches Carbonsäuresalz eines Alkalimetalls umfasst.

3. Verfahren nach Anspruch 1, wobei das Aluminiumsalz unter Aluminiumsulfat, Aluminiumchlorid, Natriumaluminat oder Mischungen davon ausgewählt ist.

4. Verfahren nach Anspruch 3, wobei das Aluminiumsulfat mit einer alkalischen Quelle reagiert, bevorzugt Natriumhydroxid, um ein Aluminiumhydroxid und bevorzugt Natriumaluminat zu bilden.

5. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die im Schritt (ii) zur Bereitstellung einer wässrigen Aufschlämmung zugegebene alkalische Quelle aus der Gruppe ausgewählt ist, bestehend aus Alkalimetallhydroxid, Alkalimetallsilikat oder Mischungen davon.

6. Verfahren nach Anspruch 5, wobei das Alkalimetallsilikat ein  $\text{SiO}_2:\text{M}_2\text{O}$ -Gewichtsverhältnis in dem Bereich von 1,6

bis 3,3, bevorzugt von 1,6 bis 2,4 und höchst bevorzugt von 2,0 bis 2,85 aufweist.

7. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Reinigungstensid der wässrigen Mischung vor oder nach der Bildung eines oder mehrerer Reaktionsprodukte, ausgewählt aus der Gruppe, bestehend aus einem Carbonsäuresalz von Aluminium, einem Aluminiumkomplex einer Carbonsäure oder Mischungen davon, zugegeben wird.

8. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die organische Carbonsäure Zitronensäure ist.

9. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die wässrige Aufschlämmung nicht mehr als 2 Gew.-% Alkalimetallsilikat umfasst.

10. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die wässrige Aufschlämmung weniger als 3 Gew.-% Phosphatbuilder umfasst.

11. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Reinigungstensid aus der Gruppe ausgewählt ist, bestehend aus anionischem Tensid, nichtionischem Tensid, kationischem Tensid, amphoterem Tensid, zwitter-ionischem Tensid oder Mischungen davon.

12. Verfahren nach Anspruch 11, wobei das anionische Tensid bevorzugt aus der Gruppe ausgewählt ist, bestehend aus Alkylbenzolsulfonat, Alkylethersulfat, Alkylsulfat oder Mischungen davon.

13. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die wässrige Aufschlämmung umfasst:

- (i) 2 bis 35 Gew.-% Reinigungstensid;
- (ii) 0,2 bis 8 Gew.-% eines oder mehrerer organischer Carbonsäuresalze von Aluminium, eines Aluminiumkomplexes organischer Carbonsäure oder Mischungen davon;
- (iii) bevorzugt 0 bis 10 Gew.-% organisches Carbonsäuresalz eines Alkalimetalls;
- (iv) bevorzugt 0 bis 2 Gew.-% Silikatsalz eines Alkalimetalls;
- (v) bevorzugt 0 bis 1 Gew.-% Aluminiumhydroxid;
- (vi) bevorzugt 15 bis 70 Gew.-% Füllstoff; und
- (vii) 20 bis 40 Gew.-% Wasser.

14. Sprühgetrocknetes Waschmittelpartikel, erhältlich nach irgendeinem der vorhergehenden Ansprüche, umfassend:

- (i) 3 bis 50 Gew.-% Reinigungstensid, bevorzugt anionisches Reinigungstensid;
- (ii) 0,4 bis 10 Gew.-% eines oder mehrerer organischer Carbonsäuresalze von Aluminium, eines Aluminiumkomplexes organischer Carbonsäure oder Mischungen davon;
- (iii) bevorzugt 0 bis 12 Gew.-% organisches Carbonsäuresalz eines Alkalimetalls;
- (iv) bevorzugt 0 bis 2 Gew.-% Silikatsalz eines Alkalimetalls;
- (v) bevorzugt 0 bis 2 Gew.-% Aluminiumhydroxid;
- (vi) bevorzugt 25 bis 88 Gew.-% Füllstoff; und
- (vii) bevorzugt 1 bis 3,5 Gew.-% Feuchtigkeitsgehalt.

15. Waschmittelzusammensetzung, umfassend 5 bis 100 Gew.-% sprühgetrocknetes Waschmittelpartikel nach Anspruch 14.

## Revendications

1. Procédé pour préparer une particule détergente pour le linge séchée par pulvérisation, comprenant les étapes de :

- (i) réaction d'un acide carboxylique organique avec l'un quelconque parmi (a) une solution de sel aluminate, (b) une solution d'hydroxyde d'aluminium, et leurs mélanges, dans laquelle la solution d'hydroxyde d'aluminium est formée in situ par l'une quelconque des réactions choisies dans le groupe constitué par (i) la réaction d'une solution d'aluminate de sodium avec un acide ; et (ii) la réaction d'un sel d'aluminium avec une source alcaline ; pour former un ou plusieurs produits réactionnels choisis dans le groupe constitué par un sel d'aluminium et d'acide carboxylique organique, un complexe d'aluminium et d'acide carboxylique organique, et leurs mélanges ;

(ii) addition d'une certaine quantité de source alcaline au mélange aqueux pour former une bouillie aqueuse ayant un pH de 4 à 8,5, dans laquelle la bouillie aqueuse comprend un ou plusieurs produits réactionnels choisis dans le groupe constitué par un sel d'aluminium et d'acide carboxylique organique, un complexe d'aluminium et d'acide carboxylique organique, et leurs mélanges, et un tensioactif détersif ; et

(iii) séchage par pulvérisation de la bouillie aqueuse pour former une particule détergente séchée par pulvérisation.

2. Procédé selon la revendication 1, dans lequel la bouillie aqueuse comprend en outre un sel de métal alcalin et d'acide carboxylique organique.

3. Procédé selon la revendication 1, dans lequel le sel d'aluminium est choisi parmi le sulfate d'aluminium, le chlorure d'aluminium, l'aluminate de sodium, et leurs mélanges.

4. Procédé selon la revendication 3, dans lequel le sulfate d'aluminium réagit avec une source alcaline, de préférence l'hydroxyde de sodium, pour former de l'hydroxyde d'aluminium et de préférence de l'aluminate de sodium.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel la source alcaline dans l'étape (ii), ajoutée pour former une bouillie aqueuse, est choisie dans le groupe constitué par un hydroxyde de métal alcalin, un silicate de métal alcalin, et leurs mélanges.

6. Procédé selon la revendication 5, dans lequel le silicate de métal alcalin a un rapport en poids  $\text{SiO}_2/\text{M}_2\text{O}$  situé dans la plage allant de 1,6 à 3,3, mieux encore de 1,6 à 2,4 et tout spécialement de 2,0 à 2,85.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel le tensioactif détersif est ajouté au mélange aqueux avant ou après la formation du seul ou des produits réactionnels choisis dans le groupe constitué par un sel d'aluminium et d'acide carboxylique, un complexe d'aluminium et d'acide carboxylique, et leurs mélanges.

8. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'acide carboxylique organique est l'acide citrique.

9. Procédé selon l'une quelconque des revendications précédentes, dans lequel la bouillie aqueuse comprend au plus 2 % en poids de silicate de métal alcalin.

10. Procédé selon l'une quelconque des revendications précédentes, dans lequel la bouillie aqueuse comprend moins de 3 % en poids d'adjuvant de type phosphate.

11. Procédé selon l'une quelconque des revendications précédentes, dans lequel le tensioactif détersif est choisi dans le groupe constitué par un tensioactif anionique, un tensioactif non-ionique, un tensioactif cationique, un tensioactif amphotère, un tensioactif zwitterionique, et leurs mélanges.

12. Procédé selon la revendication 11, dans lequel le tensioactif anionique est de préférence choisi dans le groupe constitué par un alkylbenzènesulfonate, un alkyléthersulfate, un alkylsulfate, et leurs mélanges.

13. Procédé selon l'une quelconque des revendications précédentes, dans lequel la bouillie aqueuse comprend :

(i) 2 % en poids à 35 % en poids de tensioactif détersif ;

(ii) 0,2 % en poids à 8 % en poids d'un ou plusieurs parmi les sels d'aluminium et d'acide carboxylique organique, un complexe d'aluminium et d'acide carboxylique organique, et leurs mélanges ;

(iii) de préférence 0 % en poids à 10 % en poids de sel de métal alcalin et d'acide carboxylique organique ;

(iv) de préférence 0 % en poids à 2 % en poids de sel silicate de métal alcalin ;

(v) de préférence 0 % en poids à 1 % en poids d'hydroxyde d'aluminium ;

(vi) de préférence 15 à 70 % en poids d'agent de remplissage ; et

(vii) 20 à 40 % en poids d'eau.

14. Particule détergente séchée par pulvérisation pouvant être obtenue conformément à l'une quelconque des revendications précédentes, comprenant :

(i) 3 % en poids à 50 % en poids de tensioactif détersif ; de préférence de tensioactif détersif anionique ;

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- (ii) 0,4 % en poids à 10 % en poids d'un ou plusieurs parmi les sels d'aluminium et d'acide carboxylique organique, un complexe d'aluminium et d'acide carboxylique organique, et leurs mélanges ;
- (iii) de préférence 0 % en poids à 12 % en poids de sel de métal alcalin et d'acide carboxylique organique ;
- (iv) de préférence 0 % en poids à 2 % en poids de sel silicate de métal alcalin ;
- (v) de préférence 0 % en poids à 2 % en poids d'hydroxyde d'aluminium ;
- (vi) de préférence 25 % en poids à 88 % en poids d'agent de remplissage ; et
- (vii) de préférence 1 % en poids à 3,5 % en poids de teneur en humidité.

**15.** Composition détergente pour le linge comprenant 5 % en poids à 100 % en poids de particule détergente séchée par pulvérisation selon la revendication 14.

**REFERENCES CITED IN THE DESCRIPTION**

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