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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

[0001] The present invention relates to a process for preparing a spray-dried detergent particle. Particularly to 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.

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 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.

[0004] These days consumers prefer laundry detergent composition which are milder on the skin. 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 low pH composition necessitates significantly reducing or completely removing the alkaline ingredients such as sodium carbonate and sodium silicate.

[0006] One such document is WO 2018/067494 A1 (The Procter & Gamble Company) which discloses a process for preparing a spray-dried laundry detergent particle substantially free of silicate salt and carbonate salt. The process involves the step of contacting zeolite to monomeric organic carboxylic acid in an aqueous mixture to form silica.

[0007] While reducing or total removal of sodium carbonate and sodium silicate brings down the pH of the spray-dried particle, their reduction also negatively impacts the Ca and Mg ions sequestering ability in hard water, as sodium carbonate is known to be an effective builder.

[0008] 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 detergent particle were not satisfactory because the resultant spray-dried detergent particle was found to have acceptable powder properties as soon as formed, however, on storage the powder showed caking.

[0009] 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 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.

[0010] Hence, preparing a low pH spray-dried detergent particle with good powder flow properties especially on prolonged storage has been a challenge in the past.

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

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

[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 having an in-situ formed reaction product selected from the group consisting of organic carboxylic acid salt of aluminium, 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 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 laundry detergent particle is substantially free of 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) contacting an aluminate salt solution, alkali metal silicate solution and an organic carboxylic acid in an aqueous mixture 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 from 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 detersive 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): Forming an organic carboxylic acid salt of aluminium or a complex in an aqueous mixture

[0022] According to the first aspect of the present invention disclosed is a process of contacting an aluminate salt solution, an alkali metal silicate solution, and an organic carboxylic acid to form a carboxylic acid salt of aluminium in an aqueous mixture.

Aluminate salt solution:

[0023] The aluminate salt solution added to the aqueous mixture is preferably an alkali metal salt of aluminate, alkaline earth metal salt of aluminate or mixtures thereof. More preferably an alkali metal salt of aluminate. Preferably the aluminate salt is a sodium aluminate, a magnesium aluminate, or mixtures thereof. Most preferably the aluminate salt is sodium aluminate. Aluminate salt may be in a liquid form, solid form or prepared in-situ. Sodium aluminate is commercially available in anhydrous form. Commercially available sodium aluminate preferably includes from 50 wt.% to 60 wt.% aluminium oxide (Al_2O_3) and 35 wt.% to 45 wt.% sodium oxide (Na_2O). The sodium aluminate is used in the present invention in the form of an aqueous solution. Available sodium aluminate are alkaline and have an Al_2O_3 to Na_2O 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 Al_2O_3 to Na_2O 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.

[0024] In an embodiment of the present process the sodium aluminate is formed by digesting an aluminium compound with an alkaline source to form the sodium aluminate. Preferably the aluminium compound is aluminium sulphate which is digested in presence of sodium hydroxide to form sodium aluminate.

Alkali metal silicate:

[0025] The process according to the present invention includes an alkali metal silicate. Preferably the alkali metal silicate is a soluble silicate. Soluble silicates are common ingredients in the laundry detergent compositions. Preferably the alkali metal silicate salt may be selected from the group consisting of sodium silicate, potassium silicate, sodium-potassium double silicate or mixtures thereof. Preferably the alkali metal silicate salt is water-soluble. The alkali metal silicate salt preferably has a weight ratio of SiO_2:M_2O, within the range of 1.6 to 3.3 more preferably 1.6 to 2.4, and most preferably 2.0 to 2.85, wherein M is an alkali metal. The alkali metal silicate salt employed is in the form of an aqueous solution, generally having 30 wt.% to 45 wt.% solid content. The alkali metal silicate added to the aqueous mixture is preferably sodium silicate.

[0026] Preferably the step (i) involves a two-step process. In a first step, aluminate salt solution and the alkali metal silicate solution are contacted. This is followed by a second step of adding an organic carboxylic acid into the aqueous mixture. The aluminate salt solution and the alkali metal silicate solution react to form a precipitate. This precipitate preferably includes alkali metal aluminosilicate. Preferably the alkali metal aluminosilicate is sodium aluminosilicate. Preferably the alkali metal silicate solution is an aqueous sodium silicate solution having 30 wt.% to 45 wt.% solid content. The aqueous mixture including aluminate salt solution and the alkali metal silicate solution is preferably heated at a temperature from 70 to 80°C. Preferably the aluminate salt solution is contacted with an alkali metal silicate solution for 1 minute to 5 minutes, still preferably from 1 minute to 3 minutes, still preferably from 1 minute to 2 minutes before the addition of an organic carboxylic acid.

Organic carboxylic acid salt of aluminium:

[0027] The aluminate salt solution, alkali metal silicate solution and the organic carboxylic acid react in an aqueous mixture to form an organic carboxylic acid salts of aluminium. 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 preferably selected from the group consisting of alkali metal aluminium silicate, aluminium hydroxide, alkali metal salt of organic carboxylic acid or mixtures thereof. Preferably the organic carboxylic acid is citric acid which reacts with aluminate salt solution and alkali metal silicate salt solution to form aluminium citrates.

[0028] Preferably when the organic carboxylic acid is reacted with alkaline sodium aluminate salt solution, alkali metal silicate 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 preferably selected from the group consisting of alkali metal aluminium silicate, aluminium hydroxide, alkali metal salt of organic carboxylic acid or mixtures thereof. Preferably the aqueous mixture also includes 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 carboxylic acid:

[0029] The sodium aluminate solution, alkali metal silicate solution, organic carboxylic acid reacts in an aqueous mixture to preferably form an aluminium complex of carboxylic acid. Preferably the organic carboxylic acid is citric acid.

Aqueous mixture:

[0030] The aqueous mixture preferably includes a detersive surfactant. Suitable detersive surfactant includes anionic, nonionic, cationic, amphoteric, zwitterionic detersive surfactant or mixtures thereof. Suitable detersive surfactant may be linear or branched, substituted or un-substituted. The detersive surfactant may be derived from petrochemical material or is bioderived.

[0031] Preferably the detersive surfactant is anionic, nonionic or mixtures thereof. More preferably the aqueous mixture includes an anionic surfactant. Suitable anionic detersive surfactant is an alkyl sulphonate surfactant, alkyl sulphate surfactant or mixtures thereof. Preferably the anionic surfactant is preferably selected from the group consisting of alkyl benzene sulphonate, alkyl ether sulphate, alkyl sulphate or mixtures thereof. The anionic surfactant and/or nonionic surfactant may be linear or branched, substituted or unsubstituted. The detersive surfactant may be derived from petrochemical material or is bioderived.

[0032] Detersive surfactant: The aqueous mixture preferably includes a detersive surfactant. The detersive surfactant is preferably an anionic surfactant. The detersive 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 un-neutralized part of the liquid acid form of the anionic surfactant is neutralized in-situ in the aqueous mixture. Fully pre-neutralized anionic surfactant commercially available in solid form or in the form of paste may also be suitably used. Preferably the detersive surfactant is added to the aqueous mixture before addition of the aluminate salt solution. In some embodiments the detersive surfactant, especially in the fully pre-neutralized salt form is added to the aqueous mixture after formation of the organic carboxylic acid of aluminium. In further embodiments the detersive surfactant in the fully neutralized salt form may be added to the base mixture after the formation of the organic carboxylic acid salt of alkaline earth metal. Pre-neutralized surfactant is commercially available in solid form or in the form of paste.

[0033] Preferably the detersive surfactant is added to the aqueous mixture before addition of the aluminate salt. In some embodiments the detersive 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 the alkali metal silicate solution to the aqueous mixture.

[0034] In some other embodiments of the present invention the detersive surfactant in the neutralized salt form or acid form may be added to the aqueous mixture after the addition of the aluminate salt solution and the alkali metal silicate solution. In still other embodiments of the present invention the detersive surfactant in the neutralized salt form or acid form may be added to the aqueous mixture after the formation of the reaction product including one or more ingredients selected from the group consisting of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof.

[0035] When the detersive surfactant is added into the aqueous mixture in the form of a partly anionic 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 preferably contributes from 28 parts to 98 parts of the total anionic surfactant by weight present in the spray-dried detergent particle. On addition of aluminate salt and/or the alkali metal silicate salt to the partly neutralized anionic surfactant in the aqueous mixture, the remaining unreacted acid form of the anionic surfactant reacts with aluminate salt, and/or alkali metal silicate to form fully neutralized salt form of the anionic surfactant.

[0036] 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. One or more anionic surfactant may be present in the spray-dried detergent particle.

[0037] Typically, the detersive surfactant is present in the aqueous mixture when the aluminate salt and the alkali metal silicate is added to the aqueous mixture. Further typically, the aqueous mixture includes a detersive surfactant before the addition of the organic carboxylic acid. The preferred order of addition is to contact the pre-neutralized detersive surfactant or the acid precursor form of the anionic detersive surfactant with water followed by adding the sodium aluminate salt solution, alkali metal silicate salt and organic carboxylic acid to the aqueous mixture. Preferably in a first step sodium aluminate salt solution and alkali metal silicate salt are contacted followed by the addition of an organic carboxylic acid to the aqueous mixture. Preferably the part or full neutralization may be carried out in the same vessel by contacting the acid precursor form of the anionic surfactant with an aqueous solution of neutralizing agent (alkali metal hydroxide) to form the neutralized anionic surfactant.

[0038] When the detersive surfactant is pH sensitive, that is those detersive surfactants which undergo hydrolyses at low pH conditions, then it is preferred that the detersive surfactant is added to the aqueous slurry after the pH is raised to 7 or above. Non-limiting example of the detersive surfactant is primary alkyl sulphate surfactant. Preferably PAS has an alkyl chain length of C_8 to C_{18}, more preferably with an alkyl chain length of C_{12} to C_{14}. Preferably the primary alkyl sulphate surfactant is linear or branched, preferably linear. Preferably the primary alkyl sulphate surfactant is substituted or unsubstituted.

[0039] Preferably the detersive surfactant is an anionic surfactant. Suitable anionic detersive surfactant include sulphonate and sulphate surfactant. Suitable sulphonate surfactant include methyl ester sulphonate, alpha olefin sulphonate, alkyl benzene sulphonate, especially alkyl benzene sulphonate, preferably C_{10} to C_{14} alkyl benzene sulphonate. A preferred detersive 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_{10} to C_{18} alkyl group, still preferably C_{10} to C_{14} 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®. One or more anionic surfactant may be present in the spray-dried detergent particle.

[0040] Suitable sulphate surfactant include alkyl sulphate, preferably C_8 to C_{18} alkyl sulphate, or predominantly C_{12} to C_{18} alkyl sulphate. A preferred sulphate detersive surfactant is alkyl alkoxylated sulphate, preferably alkyl ethoxylated sulphate, preferably a C_8 to C_{18} alkyl alkoxylated sulphate, preferably a C_8 to C_{18} alkyl ethoxylated sulphate, preferably the alkyl alkoxylated sulphate has an average degree of alkoxylation of from 0.5 to 20, preferably from 0.5 to 10, preferably the alkyl alkoxylated sulphate is a C_8 to C_{18} alkyl ethoxylated sulphate having an average degree of ethoxylation 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 alkoxylated 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 detersive surfactant include, soaps, alkyl ether carboxylates. Suitable anionic detersive surfactant may be in salt form, suitable counter-ions include sodium, calcium, magnesium, amino alcohol, and any combinations thereof. A preferred counterion is sodium.

[0041] Suitable non-ionic detersive surfactant are selected from the group consisting of: C_8 to C_{18} alkyl ethoxylates, such as, NEODOL® non-ionic surfactants from Shell; C_6 to C_{12} alkyl phenol alkoxylates wherein preferably the alkoxylate units are ethyleneoxy units, propyleneoxy units or a mixture thereof; C_{12} to C_{18} alcohol and C_6 to C_{12} 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.

[0042] Suitable non-ionic detersive surfactant are alkyl polyglucoside and/or an alkyl alkoxylated alcohol. Suitable non-ionic detersive surfactants include alkyl alkoxylated alcohols, preferably C_8 to C_{18} alkyl alkoxylated alcohol, preferably a C_8 to C_{18} alkyl ethoxylated alcohol, preferably the alkyl alkoxylated 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 alkoxylated alcohol is a C_8 to C_{18} alkyl ethoxylated 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 alkoxylated alcohol can be linear or branched and substituted or un-substituted. Suitable nonionic detersive surfactants include secondary alcohol-based detersive surfactants.

[0043] Zwitterionic surfactant: Suitable zwitterionic detersive surfactants include amine oxides and/or betaines.

[0044] One or more detersive surfactant may be present in the spray-dried laundry detergent particle according to the present invention. The surfactant is preferably those which are thermally stable during processing conditions of a spray-drying tower wherein typically the inlet air temperature ranges 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.

Organic carboxylic acid:

[0045] Preferably the organic carboxylic acid is a monomeric 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 carboxylic acid is selected from the group citric acid, malic acid, succinic acid, lactic acid, glycolic acid, fumaric acid, tartaric acid, formic acid, and mixtures thereof. More preferably, the organic carboxylic 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.

[0046] Adding the organic carboxylic acid to the aqueous mixture forms one or more reaction products selected from the group consisting of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof. Preferably in addition to the reaction product mentioned, the reaction may also preferably result in the formation of one or more additional reaction products selected from the group consisting of silica, alkali metal aluminosilicate and aluminium hydroxide, alkali metal salt of organic carboxylic acid or mixtures thereof. Preferably the aqueous mixture also includes an organic carboxylic acid salt of sodium.

[0047] Preferably the organic carboxylic acid is citric acid which reacts with the components present in the aqueous mixture to form reaction products selected from the group consisting of sodium aluminosilicate, aluminium citrate, silica, aluminium hydroxide. Preferably the citric acid also reacts to form sodium citrate.

[0048] At the end of step (i), the reaction results in the formation of an aqueous mixture comprising organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof preferably along with one or more of additional reaction products selected from the group consisting of silica, aluminium hydroxide, organic carboxylic acid salt of alkali metal or mixtures thereof. The aqueous mixture may preferably include a detersive surfactant.

[0049] The pH of the aqueous mixture is less than 4, more preferably the pH of the aqueous mixture is from 2 to 3.5. 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 particle prone to caking upon extended storage periods.

Step (iii): Forming an aqueous slurry

[0050] 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 from 4 to 8.5. The alkaline source may include any salt which enables the pH to be adjusted in a range from 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 the group consisting of sodium silicate, 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_2:M_2O within the range of 1.6 to 3.3 more preferably 1.6 to 2.4, and most preferably 2.0 to 2.85, wherein M is an alkali metal. The alkali metal silicate salt employed is in the form of an aqueous solution, generally having 30 wt.% to 45 wt.% solid content.

[0051] In a preferred process, sodium hydroxide is added to the aqueous mixture to bring the pH of the aqueous mixture to range from 4 to 6.5, followed by the addition of sodium silicate to raise the pH from 7 to 8.5.

[0052] 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 in the aqueous slurry. The organic carboxylic acid salt is preferably an alkali metal salt, more preferably selected from the group consisting of an organic carboxylic acid salt of a mono alkali metal, organic carboxylic acid salt of a dialkali metal or organic carboxylic acid salt of a trialkali metal salt. Preferably the aqueous slurry has from 2 wt.% to 8 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, more preferably the organic carboxylic acid salt of a trialkali metal salt. The aqueous slurry preferably includes organic carboxylic acid salt of a dialkali metal, organic carboxylic acid salt of a trialkali metal or mixtures thereof. Preferably the organic carboxylic acid salt of an alkali metal includes disodium citrate, trisodium citrate, mono sodium citrate and mixtures thereof. Preferably disodium citrate, trisodium citrate and mixtures thereof.

Filler

[0053] 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. More preferably the filler is sodium sulphate. 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. 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.

[0054] The aqueous slurry prepared according to the process of the first aspect of the present invention includes:

(i) from 2 wt.% to 35 wt.% detersive surfactant;
(ii) from 0.4 wt.% to 5 wt.% of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof;
(iii) preferably 0.5 wt.% to 2 wt.% silica;
(iv) preferably from 0 wt.% to 2 wt.% silicate salt of alkali metal;
(v) preferably 0.04 wt.% to 0.5 wt.% aluminium hydroxide;
(vi) from 20 wt.% to 40 wt.% water;
(vii) preferably 15 wt.% to 70 wt.% filler; and,
(viii) preferably 2 wt.% to 8 wt.% organic carboxylic acid salt of alkali metal.

[0055] Preferably the amount of detersive 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.%.

[0056] Preferably the amount of one or more of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof in the aqueous slurry is not less than 0.45 wt.%, still preferably not less than 0.5 wt.%, more preferably not less than 0.6 wt.%, still more preferably not less than 1 wt.%, but typically not more than 5 wt.%, preferably not more than 4.8 wt.% or still preferably not more than 4.5 most preferably not more than 4.4 wt.%.

[0057] Preferably the amount of organic carboxylic acid salt of alkali metal in the aqueous slurry is not less than 2.1 wt.%, still preferably not less than 2.5 wt.%, more preferably not less than 2.8, still more preferably not less than 3 wt.%, furthermore preferably not less than 7.8 wt.%, but typically not more than 7.6 wt.%, preferably not more than 7.5 wt.% or still preferably not more than 7 wt.%, more preferably not more than 6.5 wt.%.

[0058] 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.%. Still more preferably the slurry has 0 wt.% silicate salt of alkali metal. 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.

[0059] Preferably the amount of aluminium hydroxide is not less than 0.045 wt.%, still preferably not less than 0.05 wt.%, more preferably not less than 0.08 wt.%, still more preferably not less than 0.1 wt.%, but typically not more than 0.48 wt.%, preferably not more than 0.45 wt.% or still preferably not more than 0.44 wt.%.

[0060] 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.%.

[0061] 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.%.

[0062] The aqueous slurry preferably includes silica. Preferably the silica is present in an amount ranging from 0.5 wt.% to 2 wt.%, still preferably from 0.5 to 1 wt.%. The silica is preferably formed during the step (i).

[0063] Preferably the aqueous slurry has less than 2 wt.% carbonate builder, still preferably less than 1 wt.%, further preferably 0 wt.% carbonate builder. Preferably the aqueous slurry includes from 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. More preferably alkali metal carbonate is sodium carbonate.

[0064] 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 includes from 0 wt.% to 2 wt.% inorganic phosphate builder. Examples of inorganic phosphate builder includes but is not limited to sodium orthophosphate, pyrophosphate and tripolyphosphate.

[0065] Preferably the aqueous slurry has less than 2 wt.% zeolite builder, still preferably less than 1 wt.%, further preferably no added zeolite builder. Preferably the aqueous slurry includes from 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.

[0066] Preferably the aqueous slurry includes an alkali metal aluminosilicate formed in step (i) by the reaction between sodium aluminate salt solution and the alkali metal silicate salt solution which remains unreacted in the aqueous slurry. Preferably the amount of the alkali metal aluminosilicate in the aqueous slurry ranges from 0.2 wt.% to 2.2 wt.%.

[0067] 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 which is preerably 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 1000 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.

[0068] 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 preferably present in an amount of 1 wt.% to 5 wt.% in the aqueous slurry.

[0069] 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.

[0070] 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, hydrotropes, 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.

[0071] 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, or mixtures thereof and antifoams.

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

[0072] In the next step, the aqueous slurry is spray dried to form a spray-dried laundry detergent particle.

[0073] 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.

[0074] 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 $1x10^5$ Nm^{-2} to $1x10^6$ Nm^{-2} , which ensures proper flooding of the second pump. Typically, the second pump is a high-pressure pump, such as a pump that is capable of generating a pressure ranging from $2x10^6Nm^{-2}$ to $2x10^7Nm^{-2}$. 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/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.

[0075] 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.

[0076] 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.

[0077] 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 wherein the spray drying zone is under a negative pressure of at least 50 Nm^{-2}, still preferably the negative pressure is from 50 Nm^{-2} to 600 Nm^{-2}.

[0078] Preferably, the vacuum conditions are achieved by controlling the speed setting of the dampener of either or both the inlet and the outlet air fans.

[0079] 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 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, calcite, clay, 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:

[0080] According to a second aspect of the present invention disclosed is a spray-dried detergent according to claim 14, obtainable by the process of the first aspect. Spray-dried detergent 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 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.

[0081] The spray-dried detergent particle includes:

(i) from 3 wt.% to 50 wt.% detersive surfactant; preferably anionic detersive surfactant;
(ii) from 0.5 wt.% to 10 wt.% of one or more of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof;
(iii) preferably from 2 wt.% to 12 wt.% organic carboxylic acid salts of alkali metal;
(iv) preferably from 0.75 wt.% to 3.5 wt.% silica;
(v) preferably from 0 wt.% to 2 wt.% silicate salt of alkali metal;
(vi) preferably from 0 wt.% to 1 wt.% aluminium hydroxide;
(vii) preferably 25 wt.% to 88 wt.% filler; and,
(viii) preferably from 1 wt.% to 3.5 wt.% moisture content.

[0082] Preferably the amount of detersive 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.%.

[0083] Preferably the amount of one or more of reaction products selected from the group consisting of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof in the spray dried detergent particle is not less than 0.5 wt.%, still preferably not less than 0.6 wt.%, more preferably not less than 0.8 wt.%, still more preferably not less than 1 wt.%, but typically not more than 9.5 wt.%, preferably not more than 8 wt.% or still preferably not more than 7.5 wt.%, further preferably not more than 7 wt.%, most preferably not more than 6.5 wt.%.

[0084] 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 wt.%, more preferably not less than 3.2, still more preferably not less than 3.5 wt.%, but typically not more than 11.5 wt.%, preferably not more than 11 wt.% or still preferably not more than 10.5 wt.%, more preferably not more than 10.3 wt.%, still more preferably not more than 10 wt.%.

[0085] 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 2 wt.%, preferably not more than 1.5 wt.% or still preferably not more than 1 wt.%. Preferably the spray dried detergent particle does not include alkali metal salt of silicate.

[0086] Preferably the amount of aluminium hydroxide in the spray dried detergent particle is not less than 0.01 wt.%, still preferably not less than 0.04 wt.%, more preferably not less than 0.06 wt.%, still more preferably not less than 1 wt.%, but typically not more than 0.8 wt.%, preferably not more than 0.7 wt.% or still preferably not more than 0.6 wt.%.

[0087] Preferably the amount of water in the spray dried detergent particle is not less than 1 wt.%, still preferably not less than 1.25 wt.%, more preferably not less than 1.5 wt.%, still more preferably not less than 1.75 wt.%, but typically not more than 3 wt.%, preferably not more than 2.75 wt.% or still preferably not more than 2.5 wt.%, more preferably not more than 2 wt.%.

[0088] 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 30wt.%, 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.

[0089] 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, hydrotropes, shading dye, pigments, or mixtures thereof and antifoam. Non-limiting examples of hydrotropes includes sodium cumene sulphonate, sodium toluene sulphonate, sodium xylene sulphonate or mixtures thereof.

[0090] Preferably the spray-dried detergent particle includes silica. Preferably the silica is present in an amount ranging from 0 to 3.5 wt.%, still preferably from 0.2 to 3.5 wt.%, further preferably from 0.25 to 3, still more preferably 0.5 to 2.5 wt.% in the spray-dried detergent particle, still preferably the 0.75 to 1.5. The silica may be generated in-situ.

[0091] 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.

[0092] 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.

[0093] 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.

[0094] Optionally, the spray-dried detergent particle includes from 0 wt.% to 5 wt.% polymer, preferably from 0.5 to 5 wt.%, still preferably from 0.5 to 4 wt.% carboxylate polymer. Preferably the polymer is a carboxylate polymer, still preferably 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. Preferably the anti-redeposition agents are sodium carboxyl methyl cellulose.

[0095] 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. 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. Preferably the spray-dried detergent particle includes 0.3 wt.% to 3.5 wt.% alkali metal aluminosilicate formed during the step (i) and which may remain in unreacted form.

[0096] 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.

[0097] After spray-drying, the collected spray-dried 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.

[0098] 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.

[0099] The pH of the spray dried detergent particle is preferably from 4 to 8.5, more preferably above 7, still preferably from 7 to 8.

[0100] The spray-dried detergent particle comprises from 3 wt.% to 50 wt.% anionic surfactant, which is preferably a C_{10} to C_{20} linear alkyl benzene sulphonate and which is substantially neutralized with little or no acid residues.

[0101] 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

[0102] According to a further aspect of the present invention, disclosed is a laundry detergent composition having the spray-dried laundry detergent particle of the second 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 high-speed 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.

[0103] 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.

[0104] 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 anti-redeposition agents are sodium carboxyl methyl cellulose. These optional ingredients are well known to be used in a laundry detergent composition and added preferably by post-dosing.

[0105] The laundry detergent composition includes from 5 wt.% to 100 wt.% spray-dried detergent particle obtainable according to the second 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 second aspect of the present invention.

[0106] 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

[0107] 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-styry)-3-sulfophenyl)-2H-napthol(1,2-d]triazole, disodium 4,4'bis{[4-anilino-6-(N methyl-N-2 hydroxyethyl)amino 1,3,5- triazin-2-yl)]amino]stilbene-2-2' disulfonate, disodium 4,4'bis([(4-anilino-6-morpholino-1,3,5-triazin-2-yl)]amino} stilbene-2-2'di-sulfonate, 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:

[0108] 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.

[0109] 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, lipoxygenases, 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:

[0110] 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 is not limited to polyolefin film, laminates, paper based films or laminates, multilayered structures including two or more flexible structures and other materials known to a person skilled in the art.

[0111] 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.

[0112] 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.

[0113] Disclosed 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 detersive surfactants (as are further defined above).

[0114] 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, and minimal fabric residue deposition. This is due to the in-situ formation of organic carboxylic acid of aluminium.

Examples

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

[0115] 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 to form sodium LAS. 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 1 below. During the mixing the temperature in the mixing unit was maintained with steam at 80°C +/-2°C. While the heating was still on, sodium aluminate solution (95% conc.) and sodium silicate solution (47% conc., 2.4 ratio) was added to the aqueous mixture. Next citric acid (organic carboxylic acid) was added to the aqueous mixture to form aluminium citrate. After the formation of aluminium citrate, the pH of the aqueous mixture was raised by first adding sodium hydroxide (50% concentration) and then pH was further raised from 6.5 to 7.5 by addition of sodium silicate (47% conc., 2.4 ratio).

Table 1

Preparing an aqueous slurry according to the invention					
Ingredients		Order of Addition	Quantity in kg	Temp (deg, C)	pH
Water		1	337.01	40	
sodium hydroxide (50%)		1	59.59	47	
LAS Acid (97%)		2	230.21	80	
Sodium aluminate (95%)	Heating on	3	16.75	79	
Sodium silicate (47%)	Heating on	4	33.85	79	
Sodium citrate	Heating on	5	53.30	78	
Sodium sulphate	Heating on	6	461.05	77	3.5
Sodium hydroxide (50%)	Heating on	7	10.00	80	6.5
Sodium silicate (47%)	Heating on	8	24.40	80	7.5
Total slurry batch size			1226		

[0116] The composition of the prepared aqueous slurry was as given below in Table 1A

Table 1A

Ingredients	Ex 1 (wt.%)
Na LAS	19.5
Aluminium citrate	1.86
Aluminium hydroxide	0.19
Sodium citrate (disodium citrate, trisodium citrate)	3.30
Sodium Sulphate	36.51
Sodium Silicate	0.37
Silica	1.05

(continued)

Ingredients	Ex 1 (wt.%)
Sodium aluminosilicate	0.93
Sodium aluminate	0
Sodium carbonate	0
NDOM	0.46
Water	35.87
TOTAL	**100**

[0117] 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 (7.5×10^6 Nm^{-2}), into a counter current spray-drying tower with an air inlet temperature ranging between 250°C to 330°C. The inlet fan was set to provide a tower inlet airflow of 187,500 kgh^{-1}. The exhaust fan was controlled to create a negative pressure in the tower of - 200 Nm^{-2} (typically the outlet air flow rate through the exhaust fan is between 220,000 kgh^{-1} to 240,000 kgh^{-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 (> 1.8mm) to form a spray- dried powder, which was found to be free-flowing. Fine material (<0.175mm) was elutriated along with the exhaust air coming out of the spray-drying tower and was later collected in a post tower containment system. The spray-dried detergent particle had 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 ranging 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.

Table 2

Spray dried particle according to the present invention	
Ingredients	Ex 1 (wt.%)
Na LAS	30
Aluminium citrate	2.9
Aluminium hydroxide	0.3
Sodium citrate (disodium citrate, trisodium citrate)	5.1
Sodium Sulphate	55.69
Sodium Silicate	0.6
Silica	1.6
Sodium aluminosilicate	1.4
Sodium aluminate	0
Sodium carbonate	0
NDOM	0.7
Water	2
TOTAL	**100**

[0118] 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

[0119] 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 detergent particle was measured and compared with the comparative examples.

[0120] 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 which provided an indication of the caking tendency of the spray-dried detergent 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

[0121] 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.

[0122] The prepared slurries were processed, and spray-dried detergent particle were prepared under similar conditions as described in Example 1.

[0123] Next the collected spray-dried particle was packed and sealed in packs with a water vapour transmission <5 gram/m^2/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:- once immediately after collecting the spray-dried particle (t=0, freshly prepared powder), then after 8 weeks and another at 12 weeks. The results were recorded and is provided in table 3 below.

Table 3

Comparison of inventive spray dried particle with high pH/alkaline base particle						
	Spray dried detergent particle (wt.%)			Compression Test Values (in Kg)		
	Na LAS	Na silicate	Na carbonate	Freshly prepared powder t=0	Post Storage (in week)	
					after 8 weeks	after 12 weeks
Control	25	12	15	0.2 (200g)	0.8 (800 g)	1(1000g)
Ex A	25	0	35	Slurry not processable		
Ex B	25	5	35	0.7 (700g)	2.6+ (2600g) +	Fully Caked
Ex C	25	12	0	2.45 (2450g)	Fully Caked	Fully Caked
Ex 1	30	0	0	0.2 (200g)	0.6 (600g)	0.8 (800g)

[0124] The data in table 3 shows that the spray-dried detergent particle according to the present invention (Ex 1) having no carbonate and no silicate and having an in-situ formed aluminium citrate, aluminium complex of citric acid or mixtures thereof to provided good storage properties and the spray-dried particle was free flowing even after 12 weeks and had slightly better powder properties than the control wherein the control powder has higher pH and high levels of carbonate and silicate. 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.

[0125] From the above results it was found that even when having higher levels of the detersive 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, the spray dried particle according to the invention was free flowing when freshly prepared (t = 0) as well as on prolonged storage.

Example 2B: Evaluation of the storage behaviour of a spray dried detergent particle according to the present invention and comparative examples with alternate ingredients for providing good powder properties

[0126] The storage behaviour of the spray dried detergent particle according to the present invention was evaluated along with comparative spray dried detergent particle having alternate ingredients for providing good powder properties. In a first comparative example (Ex D), pre-formed silica was added for structuring. In a second comparative example (Ex E), in-situ formed sodium citrate was used for structuring along with pre-formed added silica. In-situ sodium citrate was formed by reacting citric acid with sodium hydroxide in the slurry which was thereafter spray-dried. In a third comparative example (Ex F), silica and sodium citrate which were formed in-situ was used for improving the powder properties. The silica was formed by reacting citric acid with sodium silicate during slurry making. In all the comparative example and the invention, the active was Na LAS made in-situ during the slurry making by reacting LAS acid with sodium hydroxide as per stoichiometry.

[0127] The slurries prepared were processed and spray-dried under similar conditions as described in Example 1.

[0128] Next the collected spray-dried particles were packed and sealed in packs having a water vapour transmission <5 gram/m^2/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 that is, immediately after collecting the (t=0), after 8 weeks and 12 weeks.

[0129] The results were recorded and is provided in table 4 below.

Table 4

Comparison of spray dried particle according to the present invention with the comparative examples.				
Spray dried detergent particle (wt.%)	Ex D	Ex E	Ex F	Ex 1
Na LAS	30	30	30	30
Silica	1*	1*	1	0
Na Citrates	0	9	9	5.1
Al citrate	0	0	0	2.9
Na silicate	0	0	0	0.6
Na carbonate	0	0	0	0
Compression Test Values (in Kg)				
Fresh powder (t=0)	2.2 (2200 g)	0.05 (50 g)	0.05 (50 g)	0.2 (200 g)
Storage for 8 weeks	Fully caked	1.2 (1200 g)	3 (3000 g)	0.6 (600 g)
Storage for 12 weeks	Fully caked	2 (2000 g)	3 (3000 g)	0.8 (800 g)
** silica in Ex D and Ex E is preformed and added to the slurry before spray drying. All other ingredients mentioned namely Na citrate, Mg citrate and silica in the different examples are in-situ formed.*				

[0130] The data in table 4 demonstrates that the spray-dried detergent particle according to the present invention (Ex 1) having no carbonate and no silicate and structured using aluminium citrate, aluminium complex of citric acid or mixtures thereof provided good storage properties and the powder was free flowing even after 12 weeks. In contrast, other comparative powders (Ex E and Ex F) having in-situ formed ingredients for providing good powder properties (silica and/or sodium citrate) showed higher propensity to cake over extended storage time (12 weeks for Ex E and 8 weeks for Ex F). It was also found that Ex D having added silica had a higher propensity to cake even immediately after spray-drying.

[0131] From these results it is clear that the powder properties of the spray dried particle made according to the present invention (Ex 1) is better in terms of storage behaviour and the spray-dried particle has good powder properties and is free flowing both measured after freshly prepared as well as post prolonged storage where the spray-dried particle had a detersive active content of 30 wt.% and sulphate as a preferred filler.

Claims

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

(i) contacting an aluminate salt solution, an alkali metal silicate solution and an organic carboxylic acid in an

aqueous mixture to form one or more reaction product selected from the group consisting of an 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 from 4 to 8.5, wherein the aqueous slurry comprises one or more reaction product selected from the group consisting of an organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof and a detersive surfactant; and,
(iii) spray-drying the aqueous slurry to form a spray-dried detergent particle.

2. A process according to claim 1 wherein the step (i) involves a two-step process,
wherein a first step involves contacting aluminate salt solution and the alkali metal silicate solution followed by a second step of adding organic carboxylic acid into the aqueous mixture.

3. A process according to claim 1 or 2 wherein the step (i) further forms one or more of the reaction product selected from the group consisting of aluminium hydroxide, silica, alkali metal salt of organic carboxylic acid or mixtures thereof.

4. A process according to any one of the preceding claims wherein the aluminate salt is selected from an alkali metal aluminate, an alkaline earth metal aluminate or mixtures thereof, preferably the aluminate salt is sodium aluminate.

5. A process according to claim any one of the preceding claims wherein the alkaline source is selected from the group consisting of alkali metal hydroxide, alkali metal silicate or mixtures thereof, preferably the alkali metal silicate is sodium silicate.

6. A process according to claim 5 wherein the sodium silicate has a SiO_2:Na_2O 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 detersive surfactant is added to the aqueous mixture before or after the formation of the organic carboxylic acid salt of aluminium, an aluminium complex of organic 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 further comprises an in-situ formed organic carboxylic acid salt of alkali metal formed by reacting the organic carboxylic acid with the alkaline source.

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

11. A process according to any one of the preceding claims wherein the detersive 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 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) 2 wt.% to 35 wt.% detersive surfactant;
(ii) 0.4 wt.% to 5 wt.% of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof;
(iii) preferably from 0.5 wt.% to 2 wt.% silica;
(iv) preferably from 0 wt.% to 2 wt.% silicate salt of alkali metal;
(v) preferably from 0.04 wt.% to 0.5 wt.% aluminium hydroxide;
(vi) preferably from 15 wt.% to 70 wt.% filler;
(vii) preferably from 2 to 8 wt.% organic carboxylic acid salt of alkali metal; and,
(viii) 20 wt.% to 40 wt.% water.

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

(i) 3 wt.% to 50 wt.% detersive surfactant; preferably anionic detersive surfactant;
(ii) 0.5 wt.% to 10 wt.% of organic carboxylic acid salt of aluminium, an aluminium complex of organic carboxylic acid or mixtures thereof;
(iii) preferably from 2 wt.% to 12 wt.% organic carboxylic acid salts of alkali metal;
(iv) preferably from 0.75 wt.% to 3.5 wt.% silica;
(v) preferably from 0 wt.% to 2 wt.% silicate salt of alkali metal;
(vi) preferably from 0 wt.% to 1 wt.% aluminium hydroxide;
(vii) preferably from 25 wt.% to 88 wt.% filler; and,
(viii) preferably from 1 wt.% to 3.5 wt.% moisture content.

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

Patentansprüche

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

(i) Inkontaktbringen einer Aluminatsalzlösung, einer Alkalimetallsilikatlösung und einer organischen Carbonsäure in einer wässrigen Mischung, um ein oder mehrere Reaktionsprodukte zu bilden, ausgewählt aus der Gruppe, bestehend aus einem organischen Carbonsäuresalz von Aluminium, einem Aluminiumkomplex einer organischen 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, ausgewählt aus der Gruppe, bestehend aus einem organischen Carbonsäuresalz von Aluminium, einem Aluminiumkomplex einer organischen Carbonsäure oder Mischungen davon, und ein Reinigungstensid umfasst; und
(iii) Sprühtrocknen der wässrigen Aufschlämmung zur Bildung eines sprühgetrockneten Waschmittelpartikels.

2. Verfahren nach Anspruch 1, wobei der Schritt (i) ein zweistufiges Verfahren einbezieht, wobei in einem ersten Schritt die Aluminatsalzlösung und die Alkalimetallsilikatlösung in Kontakt gebracht werden, gefolgt von einem zweiten Schritt der Zugabe von organischer Carbonsäure zu der wässrigen Mischung.

3. Verfahren nach Anspruch 1 oder 2, wobei der Schritt (i) außerdem eines oder mehrere der Reaktionsprodukte bildet, ausgewählt aus der Gruppe, bestehend aus Aluminiumhydroxid, Siliziumdioxid, Alkalimetallsalz einer organischen Carbonsäure oder Mischungen davon.

4. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Aluminatsalz aus Alkalimetallaluminat, Erdalkalimetallaluminat oder Mischungen davon ausgewählt ist, wobei das Aluminatsalz vorzugsweise Natriumaluminat ist.

5. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die alkalische Quelle aus der Gruppe ausgewählt ist, bestehend aus Alkalimetallhydroxid, Alkalimetallsilikat oder Mischungen davon, wobei das Alkalimetallsilikat vorzugsweise Natriumsilikat ist.

6. Verfahren nach Anspruch 5, wobei das Natriumsilikat ein SiO_2:Na_2O-Gewichtsverhältnis in dem Bereich von 1,6 bis 3,3, bevorzugter 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 zu der wässrigen Mischung vor oder nach der Bildung des organischen Carbonsäuresalzes von Aluminium, eines Aluminiumkomplexes einer organischen Carbonsäure oder Mischungen davon zugesetzt 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 außerdem ein in situ gebildetes organisches Carbonsäuresalz eines Alkalimetalls, gebildet durch die Umsetzung der organischen Carbonsäure mit der alkalischen Quelle, umfasst.

10. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die wässrige Aufschlämmung nicht mehr als 2

Gew.-% Alkalimetallsilikat 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, zwitterionischem Tensid oder Mischungen davon.

12. Verfahren nach Anspruch 11, wobei das anionische Tensid 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) 0,4 bis 5 Gew.-% organisches Carbonsäuresalz von Aluminium, eines Aluminiumkomplexes einer organischen Carbonsäure oder Mischungen davon;
(iii) vorzugsweise 0,5 bis 2 Gew.-% Siliziumdioxid;
(iv) vorzugsweise 0 bis 2 Gew.-% Silikatsalz eines Alkalimetalls;
(v) vorzugsweise 0,04 bis 0,5 Gew.-% Aluminiumhydroxid;
(vi) vorzugsweise 15 bis 70 Gew.-% Füllstoff;
(vii) vorzugsweise 2 bis 8 Gew.-% organisches Carbonsäuresalz eines Alkalimetalls; und
(vii) 20 bis 40 Gew.-% Wasser.

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

(i) 3 bis 50 Gew.-% Reinigungstensid, vorzugsweise anionisches Reinigungstensid;
(ii) 0,5 bis 10 Gew.-% organisches Carbonsäuresalz von Aluminium, eines Aluminiumkomplexes einer organischen Carbonsäure oder Mischungen davon;
(iii) vorzugsweise 2 bis 12 Gew.-% organische Carbonsäuresalze eines Alkalimetalls;
(iv) vorzugsweise 0,75 bis 3,5 Gew.-% Siliziumdioxid;
(v) vorzugsweise 0 bis 2 Gew.-% Silikatsalz eines Alkalimetalls;
(vi) vorzugsweise 0 bis 1 Gew.-% Aluminiumhydroxid;
(vii) vorzugsweise 25 bis 88 Gew.-% Füllstoff und
(viii) vorzugsweise 1 bis 3,5 Gew.-% Feuchtigkeitsgehalt.

15. Waschmittelzusammensetzung, umfassend 5 bis 100 Gew.-% sprühgetrocknete Partikel nach Anspruch 14.

Revendications

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

(i) mise en contact d'une solution de sel aluminate, d'une solution de silicate de métal alcalin et d'un acide carboxylique organique dans un mélange aqueux pour former un ou plusieurs produits réactionnels choisis dans le groupe constitué par un sel d'acide carboxylique organique et d'aluminium, un complexe d'aluminium et d'acide carboxylique organique, et leurs mélanges ;
(ii) addition d'une quantité de source alcaline au mélange aqueux pour former une bouillie aqueuse ayant un pH de 4 à 8,5, laquelle bouillie aqueuse comprend un ou plusieurs produits réactionnels choisis dans le groupe constitué par un sel d'acide carboxylique organique et d'aluminium, 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 de détergent séchée par pulvérisation.

2. Procédé selon la revendication 1, dans lequel l'étape (i) implique un procédé en deux étapes, dans lequel une première étape implique la mise en contact d'une solution de sel aluminate et de la solution de silicate de métal alcalin et est suivie d'une deuxième étape d'addition d'acide carboxylique organique dans le mélange aqueux.

3. Procédé selon la revendication 1 ou 2, dans lequel l'étape (i) forme en outre un ou plusieurs des produits réactionnels choisis dans le groupe constitué par l'hydroxyde d'aluminium, la silice, un sel de métal alcalin et d'acide carboxylique organique, et leurs mélanges.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel le sel aluminate est choisi parmi un aluminate de métal alcalin, un aluminate de métal alcalino-terreux et leurs mélanges, de préférence le sel aluminate est l'aluminate de sodium.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel la source alcaline est choisi dans le groupe constitué par un hydroxyde de métal alcalin, un silicate de métal alcalin et leurs mélanges, de préférence le silicate de métal alcalin est le silicate de sodium.

6. Procédé selon la revendication 5, dans lequel le silicate de sodium a un rapport en poids SiO_2/Na_2O 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 sel d'acide carboxylique organique et d'aluminium, d'un complexe d'aluminium et d'acide carboxylique organique, ou d'un mélange de ceux-ci.

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 en outre un sel d'acide carboxylique organique et de métal alcalin formé in situ, formé par réaction de l'acide carboxylique organique avec la source alcaline.

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

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 zwittérionique et leurs mélanges.

12. Procédé selon la revendication 11, dans lequel le tensioactif anionique est 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) 0,4 % en poids à 5 % en poids de sel d'acide carboxylique organique et d'aluminium, d'un complexe d'aluminium et d'acide carboxylique organique, ou d'un mélange de ceux-ci ;
(iii) de préférence 0,5 % en poids à 2 % en poids de silice ;
(iv) de préférence 0 % en poids à 2 % en poids de sel silicate de métal alcalin ;
(v) de préférence 0,04 % en poids à 0,5 % en poids d'hydroxyde d'aluminium ;
(vi) de préférence 15 % en poids à 70 % en poids de charge ;
(vii) de préférence 2 à 8 % en poids de sel d'acide carboxylique organique et de métal alcalin ; et
(viii) 20 % en poids à 40 % en poids d'eau.

14. Particule de détergent 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 ;
(ii) 0,5 % en poids à 10 % en poids de sel d'acide carboxylique organique et d'aluminium, d'un complexe d'aluminium et d'acide carboxylique organique, ou d'un mélange de ceux-ci ;
(iii) de préférence 2 % en poids à 12 % en poids de sels d'acide carboxylique organique et de métal alcalin ;
(iv) de préférence 0,75 % en poids à 3,5 % en poids de silice ;
(v) de préférence 0 % en poids à 2 % en poids de sel silicate de métal alcalin ;
(vi) de préférence 0 % en poids à 1 % en poids d'hydroxyde d'aluminium ;
(vii) de préférence 25 % en poids à 88 % en poids de charge ; 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 séchée par pulvérisation selon la revendication 14.

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- WO 2018067494 A1 **[0006]**
- EP 384070 A **[0065] [0095]**