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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 particulate, free flowing detergent particle by a slurry making and spray drying technique. It relates to the process for preparing a slurry with higher detersive surfactant content to produce a spray dried laundry detergent particle. The spray-dried 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] Typically, granular laundry detergent composition is prepared by spray-drying. In spray-drying process the detergent components, such as surfactants and builders are mixed with around 20% to 50% by weight water to form an aqueous slurry, the aqueous slurry is maintained at temperatures ranging from 60°C to 85°C and then spray-dried in a spray-drying tower.

[0003] Nowadays, there is an increasing trend towards concentrating solid laundry detergent composition which have higher concentrations of cleaning agents or detersive surfactants, particularly loaded with high concentration of anionic surfactants. To formulate a high concentration laundry composition would require a spray-dried detergent particle with a desired surfactant concentration as high as 30% to 55% by total weight of spray-dried detergent particle. The spray dried-detergent particle is also known as base powder.

[0004] However, there are challenges in both manufacturing such concentrated spray-dried detergent particle and their incorporation into a laundry composition, especially when the surfactants are predominantly anionic surfactants.

[0005] Aqueous detergent slurries with a high level of anionic detersive surfactant content, typically at levels of 21 wt.% or more in slurry, requires a higher slurry moisture content compared to the ones with moderate to low active content. This is due to the gel phase formation during the formation of the detergent salt. Any attempt to reduce the moisture can result in a lumpy and sticky slurry which is difficult to pump or not pumpable.

[0006] The high slurry moisture content and increased level of detersive surfactant salt content in the slurry, necessitates increased energy consumption to reduce the final spray-dried particle moisture content to below 2.5 wt.% in order to obtain optimum physical properties. However, the high drying temperatures needed for drying off the excess water, costs extra energy and may lead to incidences of fire in the tower and/or the dry cyclones.

[0007] Another known route to manage the stickiness of the spray-dried particle with high detersive surfactant content, involves post dosing the spray-dried particle with an inorganic salt. However, the possibility of adding desired amounts of carbonate salts which provides good powder properties is severely limited in such formulation having high detersive surfactant due to the little space available for additional ingredients in the formulation.

[0008] It is also a fact that the presence of high levels of organic detersive active content, increasing the propensity of the formulation to cake on extended storage. The present inventors have found that to improve powder properties over extended storage it is essential that the higher detersive active containing spray dried particle preferably has a weight ratio of inorganic carbonate salt to detersive active content of more than 1. However, as explained above, the limitations to maximise the level of the carbonate salt in the formulations exists as the content of detersive surfactant levels go up.

[0009] Yet another challenge towards increasing or adding higher levels of carbonate in a high detersive surfactant slurry comes from the necessity that a certain amount filler such as alkali metal sulphate is required to prevent the overflow of the slurry. This is especially the case in geographies located at higher altitude. At higher altitudes the lower boiling point of water and the heat liberated from in-situ formation of the detersive active salt together may cause slurry eruptions. Antifoam addition may avoid this to some extent, but the risk remains. A solution to this problem is to make the sodium LAS reaction mix heavier by adding neutral salt or fillers selected from the group consisting of alkali metal sulphate, alkali metal chloride, alkaline earth metal carbonate or mixtures thereof, more preferred examples of fillers include sodium sulphate or sodium chloride (salt). Hence, any amount of sodium carbonate that may be further added to maintain good extended storage properties and physical properties becomes still limited.

[0010] Another reason which limits the addition of the sodium carbonate to a high detersive surfactant formulation is the requirement to keep the alkalinity of the formulation within limits to avoid risk of harshness on hands of the consumers.

[0011] Thus, it is a challenge to provide a spray-dried detergent particle where the amount of the detersive surfactant is more than 30 wt.%. It is further challenging to provide a higher detersive surfactant content formulation where the ratio of carbonate to detergent active is less than 1.

[0012] US3849346 (Lion, 1974) discloses a process for preparing a granular detergent composition containing particles of relatively high mechanical strength and which are essentially non-caking. The process includes the step of spray-drying a slurry comprising anionic surface-active agent, sodium tripolyphosphate, sodium silicate and magnesium sulphate to form in-situ magnesium silicate and magnesium phosphate.

[0013] More recently, WO 2006/029676 A1 (Unilever) discloses a laundry detergent composition with a soluble alkali metal silicate and 0.01 wt.% to 25 wt.% alkaline earth silicate seeds. The alkaline earth silicate seeds are formed in-situ by reaction of sodium silicate and soluble alkaline earth metal salt in presence of anionic surfactant during the laundry detergent base powder processing.

[0014] On the other hand, EP 3301152 A1 (Procter & Gamble, 2018) discloses a spray-dried base detergent particle having a low pH profile and with good solubility profile and cleaning profile. The spray-dried base detergent particle has a low or no level of sodium carbonate and/or sodium silicate and provides the desired powder properties by a careful control and combination of levels of organic acid and magnesium sulphate.

[0015] US 3975280 A (Hachmann Klaus et. al., 1976) discloses a washing composition with high stability on storage and where the embedded active agent does not lose its activity even on prolonged storage.

[0016] US 4139486 A (Bailey John et. al., 1979) discloses a detergent composition having superior whiteness maintenance and lower undesirable residual deposits on the fabrics which composition includes orthophosphate and pyrophosphate together as builder along with the addition of magnesium silicate. This document teaches employing a combination of phosphate builder.

[0017] WO 2005/037712 A1 (Osinga Theo Jan, 2005) discloses a detergent composition having a soluble carbonate salt in combination with a soluble alkali metal silicate. It discloses a method of preparing amorphous particles of silicate-based calcium salt or silicate based magnesium salt having the step of mixing an aqueous solution of a calcium salt or magnesium salt with a soluble alkali metal silicate salt and a soluble carbonate salt.

[0018] Thus, there is a need to provide a spray dried detergent particle which has a high level of detersive surfactant whilst maintaining good powder properties over extended storage life.

[0019] It is thus an object of the present invention to provide a process for preparing a detergent solution for a spray-dried detergent particle which provides for incorporating higher levels of the detersive surfactant whilst providing good powder properties.

[0020] It is yet another object of the present invention to provide a process for preparing a detergent slurry which upon spray drying provides for a spray-dried detergent particle having lower reserve alkalinity and a desired pH for providing good cleaning performance.

[0021] Accordingly, it is also an object of this invention to provide a process for preparing a granular detergent composition where in the spray-dried particles are characterized by a relatively better structure, but which do not have an increased tendency towards caking.

Summary of the invention

[0022] The present inventors have found that a spray dried detergent particle prepared from a slurry with high detersive surfactant content in presence of an in-situ formed silicate and/or a disilicate salt of alkaline earth metal, formed by reacting an alkali metal silicate with an alkaline earth metal salt, and an alkali metal silicate salt provides for excellent powder properties and extended shelf life without getting caked, even at lower amounts of the sodium carbonate in the spray-dried detergent particle. 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.

[0023] The spray dried detergent particle according to the present invention preferably incorporates optimum amounts of alkaline builders, particularly alkali metal silicates and alkali metal carbonates.

[0024] According to a first aspect of the present invention, disclosed is a process for preparing a spray-dried detergent particle, said process comprising the steps of:

(i) contacting an alkaline earth metal salt with an alkali metal silicate salt in an aqueous mixture comprising detersive surfactant, wherein the alkaline earth metal salt reacts with the alkali metal silicate salt to form in-situ silicate salt and/or disilicate salt of alkaline earth metal;
(ii) adding sodium carbonate to the aqueous mixture to form an aqueous slurry, wherein the aqueous slurry comprises alkali metal silicate, in-situ formed silicate salt and/or disilicate salt of alkaline earth metal, 21 wt.% to 40 wt.% detersive surfactant and sodium carbonate;
(iii) spray-drying said aqueous slurry to form said spray dried detergent particle wherein the weight ratio of the sodium carbonate to the detersive surfactant in the spray-dried particle is less than 1.

Detailed description of the invention

Process of making a spray-dried detergent particle

[0025] According to a first aspect disclosed is a process of preparing a spray-dried detergent particle comprising the steps as described herein below.

Step (i): Contacting an alkaline earth metal salt with an alkali metal silicate

[0026] According to the first aspect of the present invention disclosed is a process of contacting an alkaline earth metal salt with an alkali metal silicate in an aqueous mixture comprising a detersive surfactant.

Alkaline earth metal salt:

[0027] The alkaline earth metal salt is preferably a magnesium or a calcium salt or mixtures thereof. The alkaline earth metal salt may be preferably selected from calcium sulphate, magnesium sulphate, calcium chloride, magnesium chloride or mixtures thereof. Preferably the alkali earth metal is a magnesium salt and still preferably the alkaline earth metal salt is magnesium sulphate.

Alkali metal silicate:

[0028] The process according to the present invention includes addition of an alkali metal silicate to the aqueous mixture. Preferably the alkali metal silicate is a soluble silicate. Soluble silicates are common ingredients in the laundry detergent compositions. Some commercial grades of silicates may contain a trace level of alkaline earth metal silicate, as contamination. The composition of trace materials in the water used for making an aqueous mixture having detersive surfactant may also contribute to the alkaline earth metal. However, the amount of amorphous material introduced through this impurity route will be low. The spray dried detergent particle prepared according to the process of the first aspect of the present invention preferably includes 0.1 wt.% to 3 wt.% amorphous alkaline earth metal silicate formed in-situ.

[0029] The alkali metal silicate salt preferably has a weight ratio of SiO_2:M_2O where M is an alkali metal, within the range of 1.6 to 3.3 more preferably 1.6 to 2.4, and most preferably 2.0 to 2.85. The alkali metal silicate salt employed is in the form of an aqueous solution, generally having 30 wt.% to 45 wt.% solid content.

[0030] 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. Preferably the alkali metal silicate salt employed is sodium silicate having a weight ratio, SiO_2:Na_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.

[0031] Preferably the amount of alkali metal silicate present in the aqueous mixture is in stoichiometric excess of the amount required for reacting with alkaline earth metal salt. Preferably the amount of alkali metal silicate present in the aqueous mixture is such that the spray dried detergent particle formed preferably comprises from 5 wt.% to 17 wt.% alkali metal silicate salt. The amount of alkali metal silicate added to the aqueous mixture is from 8 wt.% to 30 wt.% by weight of the aqueous mixture. Alternately, the excess amount of alkali metal silicate (after the formation of the in-situ alkaline earth metal silicate) may be added into the slurry at any stage before spray-drying, more preferably after the addition of filler.

[0032] Preferably the weight ratio between the alkaline earth metal salt and the alkali metal silicate added to the aqueous mixture is in the range from 260:1 to 5:1, preferably 24:1 to 12:1.

Aqueous mixture:

[0033] The aqueous mixture comprises a detersive surfactant.

[0034] 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 which 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. Pre-neutralized detersive surfactant is commercially available in solid form or in the form of paste. Preferably the detersive surfactant is added to the aqueous mixture before addition of the alkaline earth metal salt. In some embodiments the detersive surfactant is added to the aqueous mixture after addition of the alkaline earth metal salt or along with the alkaline earth metal salt.

[0035] When the detersive surfactant is added into the aqueous mixture in the form of a partly neutralized surfactant, the partly neutralized anionic surfactant is preferably prepared by a neutralization process which involves the step of (i) mixing a liquid acid form of the anionic surfactant and a neutralizing agent to form a partially neutralized solution; preferably the neutralizing agent is an alkali metal hydroxide, wherein the amount of alkali metal hydroxide neutralizing agent is sufficient to react with a portion of liquid acid anionic surfactant precursor to form in-situ anionic surfactant salt. The neutralized anionic surfactant formed by neutralizing the acid form with the alkali metal hydroxide neutralizing agent contributes from 84 parts to 98 parts of the total anionic surfactant by weight present in the spray-dried particle. The partly neutralized anionic surfactant is then added to the aqueous mixture where the remaining unreacted acid form of the anionic surfactant from 2 parts to 16 parts reacts with the alkali metal silicate salt to form fully neutralized salt form of the anionic surfactant. 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 a neutralizing agent, preferably 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 in the aqueous mixture.

[0036] It is most preferred that the detersive surfactant is present when the alkaline earth metal salt is contacted with the alkali metal silicate salt. The most preferred order of addition is to contact the pre-neutralized detersive surfactant or the acid detersive surfactant precursor with water followed by contacting with the alkali metal silicate salt and then adding the alkaline earth metal salt. Preferably the part or full neutralization may be carried out in the same vessel by contacting the acid form of the anionic surfactant with an aqueous solution of neutralizing agent (alkali metal hydroxide) to form the neutralized anionic surfactant salt. Alternately in the process of the present invention, the order of addition may be reversed wherein the step involves adding alkaline earth metal salt to the aqueous mixture followed by the alkali metal silicate salt.

[0037] Suitable detersive surfactants include anionic detersive surfactants, non-ionic detersive surfactant, cationic detersive surfactants, zwitterionic detersive surfactants and amphoteric detersive surfactants. Suitable detersive surfactants may be linear or branched, substituted or un-substituted, and may be derived from sources well known to the person skilled in the art.

[0038] Preferably the detersive surfactant is an anionic surfactant. Suitable anionic detersive surfactants include sulphonate and sulphate surfactants. Suitable sulphonate surfactants include methyl ester sulphonate, alpha olefin sulphonate, alkyl benzene sulphonate, especially alkyl benzene sulphonate, preferably C_{10} to C_{13} 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_{12} to C_{18} alkyl group. Suitable alkyl benzene sulphonate (LAS) is obtainable, preferably obtained, by sulphonating commercially available linear alkyl benzene (LAB); suitable LAB includes low 2-phenyl LAB, other suitable LAB includes high 2-phenyl LAB, such as those supplied by Sasol under the tradename Hyblene®. Suitable sulphate surfactants include alkyl sulphate, preferably C_8 to C_{18} alkyl sulphate, or predominantly C_{12} to C_{18} alkyl sulphate. One or more anionic surfactant may be present in the spray-dried detergent particle.

[0039] 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 of from 0.5 to 10, preferably from 0.5 to 5, more preferably from 0.5 to 3 and most preferably from 0.5 to 1.5. The alkyl sulphate, alkyl 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 surfactants include, Soaps, alkyl ether carboxylates. Suitable anionic detersive surfactants may be in salt form, suitable counter-ions include sodium, calcium, magnesium, amino alcohols, and any combinations thereof. A preferred counterion is sodium. Preferably the detersive surfactant is anionic surfactant selected from alkyl benzene sulphonate, primary alkyl sulphate, secondary alkyl sulphate, alkyl ether sulphate or mixtures thereof, still preferably selected from linear alkyl benzene sulphonate, alkyl ether sulphate or mixtures thereof, still preferably selected from LAS, SLES or mixtures thereof.

[0040] Suitable non-ionic detersive surfactants 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. Preferably the detersive surfactant is anionic surfactant selected from alkyl benzene sulphonate, primary alkyl sulphate, secondary alkyl sulphate, alkyl ether sulphate or mixtures thereof, still preferably selected from linear alkyl benzene sulphonate, alkyl ether sulphate or mixtures thereof, still preferably selected from LAS, SLES or mixtures thereof.

[0041] Suitable non-ionic detersive surfactants 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.

[0042] Zwitterionic surfactant: Suitable zwitterionic detersive surfactants include amine oxides and/or betaines.

[0043] One or more detersive surfactant may be present in the spray-dried particle according to the present invention.

The surfactants are preferably those which are thermally stable during processing conditions of a tower with inlet air temperature ranging from 250°C to 500°C and those which are chemically stable at the pH conditions of the spray-drying slurry. Non-limiting examples of the anionic surfactant includes the ones mentioned above.

Step (ii): Forming in-situ silicate salt or disilicate salt of alkaline earth metal

[0044] The alkali metal silicate reacts with the alkaline earth metal salt to form in-situ silicate or disilicate salt of alkaline earth metal or mixtures thereof. Preferably the alkaline earth metal salt present in the reaction is magnesium sulphate or magnesium chloride, more preferably magnesium sulphate. Preferably the magnesium sulphate reacts with alkali metal silicate to form in-situ magnesium silicate or magnesium disilicate or mixtures thereof. Preferably the alkali metal silicate is sodium silicate.

[0045] Preferably the reaction of the alkali metal silicate with the alkaline earth metal salt is carried out by heating the aqueous mixture in a mixer at a temperature of 20°C to 80°C, more preferably from 70°C to 80°C. The reaction is carried out for a duration of 0.5 minutes to 30 minutes by continuously stirring the aqueous mixture in the slurry handling system.

[0046] In addition to the in-situ formed silicate salt or disilicate salt of alkaline earth metal or mixtures thereof, the reaction may also produce some amount of hydroxide of alkaline earth metal and may include some amount of unreacted magnesium sulphate. After the reaction, the aqueous mixture includes the in-situ formed silicate salt or disilicate salt of alkaline earth metal or mixtures thereof, alkali metal silicate salt, detersive surfactant optionally any unreacted alkaline earth metal salt and water.

[0047] Preferably the silicate salt or disilicate salt of the alkaline earth metal is 50% amorphous, more preferably 60% amorphous, still preferably 80%, further preferably 90% amorphous. In a highly preferred embodiment, all of the in-situ formed silicate salt and/or disilicate salt of alkaline earth metal salt is amorphous.

[0048] In addition to this, the aqueous mixture preferably also includes hydroxide of alkaline earth metal and some amount of unreacted alkaline earth metal salt.

[0049] Preferably the aqueous mixture after forming the in-situ silicate salt of alkaline earth metal comprises:

(i) 0.07 wt.% to 2.5 wt.% in-situ formed silicate salt and/or disilicate salt of alkaline earth metal;
(ii) 6 wt.% to 17 wt.% alkali metal silicate salt;
(iii) 30 wt.% to 45 wt.% detersive surfactant; and,
(iv) 35 wt.% to 50 wt.% water.

Step (iii): Adding a carbonate builder salt to the aqueous mixture to form an aqueous slurry

[0050] The next step involves adding a carbonate builder salt to form an aqueous slurry. In the process according to the first aspect, 14 wt.% to 36 wt.% a sodium carbonate builder salt is added to the aqueous mixture obtained in step (ii) followed preferably with addition by minors and fillers, to form an aqueous slurry.

[0051] Examples of the carbonate builder salt includes alkaline earth metal and alkali metal carbonates or mixtures thereof. The carbonate builder salt is preferably an alkali metal carbonate, alkaline earth metal carbonate or mixtures thereof. Preferred alkali carbonates are sodium and/or potassium carbonate of which sodium carbonate is particularly preferred. Preferably the carbonate builder salt is sodium carbonate. It is further preferred that sodium carbonate makes up at least 75 wt.%, more preferably at least 85 wt.% and even more preferably at least 90 wt.% of the total weight of the carbonate builder salt.

[0052] Preferably other non-carbonate builder may be included. Typically, inorganic builders include of alkali metals, crystalline and amorphous aluminosilicates for example, zeolites as disclosed in GB 1 473 201 (Henkel), amorphous aluminosilicates as disclosed in GB 1 473 202 (Henkel) and mixed crystalline/amorphous aluminosilicates as disclosed in GB 1 470 250 (Procter & Gamble); sodium alkaline silicates and layered silicates as disclosed in EP 164 514 B (Hoechst).

[0053] Inorganic phosphate builders for example sodium orthophosphate, pyrophosphate and tripolyphosphate are preferably present at relatively low levels, for example less than 5 wt.%, still preferably less than 3 wt.%, further preferably less than 1 wt.%. Most preferably the spray-dried detergent particle prepared from the process according to the first aspect of the present invention is substantially free of inorganic phosphate builders. By substantially free it is meant that the spray dried particle prepared according to the process of the first aspect does not include any deliberately added inorganic phosphate builder.

[0054] Zeolite builders used in most commercial particulate detergent compositions is zeolite A. Advantageously, aluminium zeolite P (zeolite MAP) described and claimed in EP 384 070A (Unilever) may be used. 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. Zeolite builders are preferably present at relatively low levels, for example less than 5 wt.%, still preferably less than 3 wt.%, further preferably less than 1 wt.% in the aqueous slurry. Most preferably the spray-dried detergent particle prepared from the process according to the first aspect of the present invention is substantially free of zeolite builders. By substantially free it is meant that the spray dried particle prepared according to the process of the first aspect does not include any deliberately added inorganic zeolite builder.

[0055] Optionally the aqueous slurry may include an organic builder. Non-limiting examples of organic builder include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di-and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts. Preferably the organic builder is selected from monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di-and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates, more preferably alkali metal citrate, most preferably it is sodium citrate. Organic builders may be used in minor amounts as supplement to carbonate builder.

[0056] Preferred supplementary organic builders are citrates, suitably used in amounts of from 0.1 wt.% to 30 wt.% more preferably of alkaline metal compounds, preferably from 10 wt.% to 25 wt.%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 wt.% to 15 wt.%, preferably from 1 wt.% to 10 wt.%.

[0057] Powder flow properties may be improved by the incorporation of a small amount of a powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate polymer. One preferred powder structurant is fatty acid soap, suitably present in an amount of from 1 wt.% to 5 wt.%.

[0058] 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, sodium bicarbonate, other inorganic salts, fluorescers, foam controllers, foam boosters, dyes, anti-redeposition agents, colourants, shading dyes and combinations thereof.

[0059] Preferably a filler may be added to the aqueous slurry before spray-drying. The filler may be added either before the addition of the carbonate salt or after the addition of the carbonate salt to the aqueous mixture to form the aqueous slurry. Preferably the filler is added after the addition of the carbonate salt. Preferably the filler is selected from the group consisting of sodium sulphate, sodium chloride, calcium carbonate, magnesium carbonate, calcite, dolomite or mixtures thereof. The filler acts as a balancing ingredient and can be a neutral inorganic salt or mineral, preferably sodium sulphate or sodium chloride.

[0060] The aqueous slurry prepared/obtainable according to the process of the first aspect comprises 21 wt.% to 40 wt.% detersive surfactant and preferably comprises:

(i) 21 wt.% to 36 wt.% detersive surfactant;
(ii) 14 wt.% to 36 wt.% sodium carbonate;
(iii) 3 wt.% to 12 wt.% alkali metal silicate;
(iv) 0.06 wt.% to 2 wt.% silicate salt and/or disilicate salt of alkaline earth metal;
(v) preferably from 0.7 wt.% to 25 wt.% filler selected from sodium sulphate, sodium chloride, calcite, dolomite, or mixtures thereof;
(vi) 30 wt.% to 40 wt.% water;
(vii) optionally, from 0 wt.% to 2 wt.% hydroxide salt of alkaline earth metal.
(viii) optionally, from 0 wt.% to 2 wt.% unreacted alkaline earth metal salt.
(ix) optionally, from 0 wt.% to 3 wt.% polymer;
(x) optionally organic builder, preferably citrate salt;
(xi) optionally optical brighteners which is preferably selected from fluorescers, colourants, shading dye, pigments;
(xii) optionally antifoams, preferably selected from silicone oil.

[0061] Preferably the amount of silicate and/or disilicate salt of alkaline earth metal is from 0.06 to 1.7 wt.%. Preferably the amount of filler is from 0.7 wt.% 23 wt.% of the slurry. Preferably the polymer is selected from a cleaning polymer, soil releasing polymer, care polymer, antiredeposition polymer or mixtures thereof. Preferably the detersive surfactant present in the aqueous slurry is an anionic surfactant. It may also be a mixture of anionic surfactant and nonionic surfactant where the mixture has a higher content of anionic surfactant. Preferably the amount of citrate salt present is from 0 wt.% to 10 wt.%.

Step (iv): Spray drying the aqueous slurry to form the spray-dried particle

[0062] In the next step, the aqueous slurry is spray dried to form a spray-dried particle.

[0063] 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.

[0064] A typical spray-drying process involves the step of transferring the aqueous slurry through a pipe system leading to a first pump and then through a second pump and from a second pump to a plurality of spray nozzles. The first pump is typically a low-pressure pump, such as a pump that can generate a pressure of from 1×10^5 Nm^{-2} to 1×10^6 Nm^{-2}, which ensures proper flooding of the second pump. Typically, the second pump is a highpressure pump, such as a pump that is capable of generating a pressure ranging from $2x10^6Nm^{-2}$ to $2\times10^7Nm^{-2}$. Optionally, the aqueous detergent 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 50,000 Kg/hour.

[0065] 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.

[0066] 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 particle.

[0067] 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/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 detergent 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 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 where 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}. Preferably, the vacuum conditions is achieved by controlling the speed and/or dampener setting of the inlet and the outlet air fans.

[0068] The spray-dried particle collected at the bottom of the tower may be subjected to cooling and conditioning by using an air lift or any similar process. Preferably the collected spray dried particle may be mixed with flow aids which includes zeolite or similar fine particles of minerals such as dolomite, calcite or mixtures thereof. The spray-dried particle is mixed with flow aid just before the airlift operation. Preferably, the spray-dried detergent 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:

[0069] According to an aspect of the present invention disclosed is a spray-dried particle obtainable by the process of the first aspect. Spray-dried particle formed from the process of the first aspect of the present invention preferably has a pH of 11.5 or less, preferably a pH ranging from 10.5 to 11.5 when measured using a 1% solution with distilled water at 25°C. The spray-dried particle is generally referred to as the base powder. This base powder may be used as a fully formulated laundry detergent composition.

[0070] The spray-dried detergent particle includes:

(i) 31 wt.% to 55 wt.% detersive surfactant;
(ii) 22 wt.% to 54 wt.% sodium carbonate;
(iii) 5 wt.% to 17 wt.% alkali metal silicate;
(iv) 0.1 wt.% to 2.5 wt.% in-situ formed silicate salt and/or disilicate salt of alkaline earth metal;
(v) preferably from 1 wt.% to 25 wt.% filler selected from sodium sulphate, magnesium carbonate, sodium chloride, calcium carbonate, calcite, dolomite or mixtures thereof.
(vi) preferably from 0.1 wt.% to 2.5 wt.% water;
(vii) optionally from 0 wt.% to 2.5 wt.% hydroxide salt of alkaline earth metal;
(viii) optionally, from 0 wt.% to 2.5 wt.% unreacted alkaline earth metal salt;
(ix) optionally organic builder, preferably from 0 wt.% to 10 wt.% citrate salt.

[0071] The weight ratio of the sodium carbonate to detersive surfactant in the spray-dried detergent particle is less than 1, still preferably less than 0.95, still preferably less than 0.9, still more preferably less than 0.85, furthermore preferably less than 0.83, still further preferably less than 0.8, most preferably less than 0.75, or even preferably less than 0.7.

[0072] It is preferred to keep the moisture content of the spray-dried detergent particle not more than 2.5 wt.% to ensure that the spray-dried particle is free-flowing and shows improved powder properties and extended shelf life. Preferably the spray-dried detergent particle includes from 22 wt.% to 54 wt.% sodium carbonate, still preferably 22 wt.% to 50 wt.% sodium carbonate. Preferably the carbonate salt is sodium carbonate. Preferably the detersive surfactant in the spray dried detergent particle is LAS. Preferably the ratio of the carbonate builder salt to the LAS active in the spray-dried detergent particle is less than 1.

[0073] The spray dried detergent particle may preferably include from 0 wt.% to 4 wt.% polymer, preferably the polymer is 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.

[0074] 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 of from 300 micrometres to 600 micrometres.

[0075] The spray-dried detergent particle comprises from 31 wt.% to 55 wt.% anionic surfactants, which is preferably a C_{10} to C_{20} linear alkyl benzene sulphonate and which is substantially neutralized with little or no acid residues.

[0076] The spray-dried 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

[0077] 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 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 the known tableting process. Further, the spray dried detergent particle may be used for preparing an unit dose product where 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, polyvinyl pyrollidone and other known film forming polymer.

[0078] The base powder/spray-dried particle is preferably formulated into a finished 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 detergent composition.

[0079] 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. These optional ingredients are well known to be used in a laundry detergent composition and added preferably by post-dosing.

[0080] 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

[0081] 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-l,3,5-triazin-2-yl)]amino} stilbene-2-2'disulfonate, and disodium 4,4'- bis(2-sulfostyryl)biphenyl. A suitable fluorescent brightener is S C.I. Fluorescent Brightener 260, which may be used in its beta or alpha crystalline forms, or a mixture of these forms.

Enzymes:

[0082] 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.

[0083] 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

[0084] The spray dried detergent particle or a laundry composition having the spray dried detergent particle prepared according to the invention may be packaged as unit doses in polymeric film soluble in the wash water. Alternatively, the spray-dried detergent particle or a composition including the particle of the invention may be supplied in multidose plastics packs with a top or bottom closure. A dosing measure may be supplied with the pack either as a part of the cap or as an integrated system. The packaging material suitable for packaging may include but not limited to multilayer polyethylene film, laminate, paper based and other materials known to a person skilled in the art. Preferably the packaging material is selected from material which are biodegradable or recyclable.

[0085] According to another aspect of the present invention, provided is a method of laundering fabric using a spray dried detergent particle or a laundry composition comprising a spray dried detergent particle according to the present invention which involves the step of diluting the dose of detergent composition with water to obtain a wash liquor and washing fabrics with the wash liquor so formed. In automatic washing machines the dose of detergent composition 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 concentrations. The dilution step preferably provides a wash liquor which comprises *inter alia* from about 3 to about 20 g/wash of detersive surfactants (as are further defined above).

Examples

Example 1: Preparing an aqueous slurry and a spray dried detergent particle according to the present invention

[0086] A spray-dried laundry detergent particle according to the present invention was prepared by first mixing water, neutralizing agent (NaOH, 45% aqueous solution) and linear alkyl benzene sulphonic acid in a crutcher where they were agitated to neutralize the linear alkyl benzene sulphonic acid to its salt form. Thereafter an excess of sodium silicate (47% aqueous solution, alkali metal silicate) was added to the aqueous mixture. The sodium silicate has a Na_2O to SiO_2 ratio of 1:2.4. The addition of sodium silicate was followed by addition of magnesium sulphate (alkaline earth metal salt). The aqueous mixture was continuously agitated, and the mixture was heated to a temperature of around 78°C to 80°C upon addition of sodium silicate and the temperature was maintained till the end of the batch preparation. The sodium silicate and magnesium sulphate react to form in-situ magnesium silicate and/or in-situ magnesium disilicate. Further sodium carbonate, sodium sulphate as filler, were added in this order to form an aqueous slurry. The composition of the ingredients added to form a batch of 10000 Kg aqueous slurry is provided in Table 1. The aqueous detergent slurry composition is shown in Table-2. The slurry composition of two examples according to the present invention (Ex 1 and Ex 2) and one comparative example (Comp A) were prepared having the composition as shown in Table 1 and with a slurry composition as shown in Table 2.

Table -1

Aqueous Slurry prepared according to the present invention				
Raw material used for slurry preparation	Temp (°C)	Comp A (KG)	Ex 1 (Kg)	Ex 2 (Kg)
Water	40	1975.20	1942.90	1951.30
Caustic (50%)	47	732.00	732.00	732.00

(continued)

Aqueous Slurry prepared according to the present invention				
Raw material used for slurry preparation	Temp (°C)	Comp A (KG)	Ex 1 (Kg)	Ex 2 (Kg)
LAS Acid (97%)	80	2828.30	2828.30	2828.30
Sodium Silicate (45%)	79	1737.50	1737.50	1737.50
Magnesium Sulphate (50%)	78	0.00	65.20	65.20
Sodium Carbonate	78	2513.80	2480.60	1645.40
Sodium Sulphate (filler balancing ingredient)	78	213.20	213.50	1040.30
Total slurry batch size (kg)		10000	10000	10000

Table -2

Aqueous Slurry composition			
Ingredients	Comp A (wt.%)	Ex 1 (wt.%)	Ex 2 (wt.%)
Na LAS	29.3	29.3	29.3
Sodium carbonate	25.39	25.39	16.25
Sodium silicate	7.81	7.24	7.24
Magnesium silicate/ magnesium disilicate	0	0.5	0.5
Water	36	36	36
Sodium sulphate	0.7	0.77	9.91
NDOM	0.8	0.8	0.8
Total	100.0	100.0	100.0

[0087] The aqueous slurry made was spray-dried in a conventional counter current spray drying tower to form a spray dried detergent particle with a moisture content of around 2 wt.% to 3.5 wt.%. The composition of the spray dried particle is provided in Table 3 below.

Table-3

Spray-dried detergent particle			
Ingredients	Comp A(wt.%)	Ex 1 (wt.%)	Ex 2 (wt.%)
Na LAS	45	45	45
Sodium carbonate	38.19	37.47	25
Sodium silicate	12	11.12	11.12
Magnesium silicate	0	0.8	0.8
Water	2.5	2.5	2.5
Sodium sulphate	1.305	2.105	14.57
NDOM	1.01	1.01	1.01
Carbonate to LAS ratio	0.85	0.85	0.55
Total	100.0	100.0	100.0

[0088] The spray-dried detergent particle prepared according to the present invention (Ex 1, Ex 2) and the comparative example (Comp A) was evaluated for powder properties as described below.

[0089] *Compression test*: This test evaluates the tendency of the powder towards caking. A split cylinder with a polished internal surface is positioned on a firm base to form a hollow cylindrical mould with a diameter of 9 centimetres. Spray dried detergent particle prepared according to the present invention (Ex 1) was filled inside the hollow cylindrical mould and levelled. A plastic disc is placed on levelled spray-dried detergent particle mass. A weight of 12 kilogram is slowly placed on the plastic disc in such a way that the weight is uniformly applied on the spray-dried detergent particle mass in the mould and the disc was allowed to compact the spray-dried detergent particle mass to form a compacted cake. After 2 minutes the weight was removed, and the cylindrical mould is opened slowly without disturbing the compacted cake mass. Next, incremental weights of 200 grams is added at an interval of 10 seconds till the compacted cake mass collapses. Total vertical load required to collapse the compacted cake mass is noted and expressed in grams and this amount in grams is indirectly defined as the caking tendency. Higher the value of the vertical load required to collapse the compacted cake mass the greater is the caking tendency of the powder under evaluation. For the present evaluation, values lower than 1 Kilogram is considered good and values beyond 2 Kilogram is classified as cohesive and classified as powder with high caking tendency.

[0090] Each spray dried particle was evaluated for their tendency to cake using the compression test as described above immediately as formed. Also, each of the comparative spray-dried detergent particle and the spray dried detergent particle prepared according to the present invention were packed in laminates with water vapour transmission rate of less than 5 gram/m^2/day and stored at storage condition of 45°C and 80 RH for a period of 8 weeks and 12 weeks., following which the compression test (UCT) was performed again. The measured values of various spray dried detergent particles both according to the present invention and comparative examples were evaluated.

[0091] It was found that the spray-dried detergent particle prepared according to the present invention having in-situ magnesium silicate/disilicate performed better and showed lower caking tendency upon storage after 8 weeks period. The comparative example having no in-situ formed magnesium silicate/disilicate having a carbonate to LAS ratio of less than 1 performed poorly.

Example 2: Evaluation of the effect of the ratio of carbonate to LAS ratio on the powder properties.

[0092] Two spray dried detergent particle with different ratio for carbonate to LAS were prepared in a conventional counter current spray drying tower to form a spray dried detergent particle with a moisture content of around 2 wt.% to 3.5 wt.%. The composition of the spray dried particle is provided in Table 4 below.

Table 4

Spray dried particle	C-1 (wt.%)	C-2 (wt.%)
LAS AD	40	40
Carbonate	26	44.87
In-situ Silicate	0	0
Carbonate / LAS AD	0.65	1.12
Compression (in gm) @ T=0	200	200
Compression (in gm) @ T=8 weeks	2000	1200
Caking @ T=8 weeks	14.8	2.71

[0093] The data in table 4 clearly demonstrates that when a spray-dried detergent particle having a high AD (40 wt.%) is prepared along with carbonate salt but in absence of the in-situ silicate formation, then in C-1 where the ratio of the carbonate to active is less than 1 (0.65) the spray-dried particle showed a greater tendency to cake upon storage. In comparison to C-1, when the spray-dried particle was preparing keeping the ratio of carbonate to LAS higher than 1 (1.12) then the powder performed relatively better and showed lesser tendency to cake.

Example 3: Evaluation of the powder properties of a spray-dried detergent particle in accordance to the present invention.

[0094] A spray-dried laundry detergent particle according to the present invention was prepared by first mixing water, neutralizing agent (NaOH, 50% aqueous solution) and linear alkyl benzene sulphonic acid in a crutcher where they were agitated to neutralize the linear alkyl benzene sulphonic acid to its salt form. Thereafter an excess of sodium silicate (42% aqueous solution, alkali metal silicate) was added to the aqueous mixture. Magnesium Sulphate (alkaline earth metal salt) was added followed by sodium silicate which has a Na2O to SiO2 ratio of 1:2.4. The aqueous mixture was continuously agitated, and the mixture was heated to a temperature of around 78°C to 80°C upon addition of sodium silicate and the temperature was maintained till the end of the batch preparation. The sodium silicate and magnesium sulphate react to form in-situ magnesium silicate, in-situ magnesium disilicate or mixtures thereof. Further sodium carbonate, sodium chloride as filler was added in this order to form an aqueous slurry. The composition of the ingredients added to the form a batch of 10000 Kg aqueous slurry is provided in Table 5.

Table -5a

Aqueous Slurry prepared according to the present invention		
Raw material used for slurry preparation	Temp (°C)	Ex 3 (Kg)
Water	40	2388.05
Caustic (49.5%)	47	677
LAS Acid (97%)	80	2661.93
Magnesium Sulphate (50%)	79	61.6
Sodium Silicate (42%)	78	1465.53
Sodium Carbonate	78	1884.3
Sodium Sulphate (filler)	78	861.67
Total slurry batch size (kg)		10000

Table -5b

Aqueous Slurry composition	
Ingredients	Ex 3 (wt.%)
Na LAS	27.68
Sodium carbonate	18.71
Sodium silicate	5.64
Magnesium silicate	0.5
Water	38.3
Sodium sulphate	9.17
Total	100

[0095] The aqueous slurry made was spray-dried in a conventional counter current spray drying tower to form a spray dried detergent particle with a moisture content of around 2 wt.% to 3.5 wt.%. The composition of the spray dried detergent particle is provided in Table 5c below.

[0096] *Evaluation of powder properties of the spray-dried detergent particle*: To evaluate the properties of the spray-dried detergent particle, after spray-drying and cooling the spray-dried particle were stored in paper bags (filling approximately 40 to 50% of the volume of the bag) and placed in a hot and humid condition for 4 hours.

[0097] The caking tendency of the spray-dried particle was evaluated at the end of the 4 hours by carefully transferring the spray-dried particle from the paper bag onto a 2mm sieve and gently shaking the sieve to allow the free-flowing particles to pass through the sieve. The bigger and lumped spray-dried particle were collected on the surface of the sieve, the weight of the particle collected on the sieve is measured and the weight % caking is determined.

Table-5c

Spray-dried detergent particle	
Ingredients	Ex 3 (wt.%)
Na LAS	45
Sodium carbonate	30
Sodium silicate	8.96
Magnesium silicate	0.8

(continued)

Spray-dried detergent particle	
Ingredients	Ex 3 (wt.%)
Water	3.5
Sodium sulphate	11.5
Carbonate to LAS ratio	0.66
caking after 4 hours storage at 45°C and 85 RH (wt. %)	27.5

[0098] The data shows that the spray-dried detergent particle prepared according to the present invention having in-situ formed magnesium silicate has relatively less caking tendency compared in spite of having lower level of carbonate and a weight ratio of Carbonate to detersive surfactant which less than 1. The spray-dried particle according to the present invention (Ex 1) also provides for incorporating higher level of sodium sulphate/sodium chloride and these optimum levels of filler in the slurry enables easy processing the detergent slurry at higher altitude countries. Further the spray-dried detergent particle according to the present invention employs lower levels of the sodium carbonate which provides the spray-dried particle with lower alkalinity and thereby is milder to the skin of handwash consumers.

Claims

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

(i) contacting an alkaline earth metal salt with an alkali metal silicate salt in an aqueous mixture comprising a detersive surfactant, wherein the alkaline earth metal salt reacts with the alkali metal silicate salt to form in-situ silicate salt and/or disilicate salt of alkaline earth metal;
(ii) adding sodium carbonate to form an aqueous slurry; wherein the aqueous slurry comprises alkali metal silicate, in-situ formed silicate salt and/or disilicate salt of alkaline earth metal, 21 wt.% to 40 wt.% detersive surfactant and sodium carbonate;
(iii) spray-drying said aqueous slurry to form said spray dried detergent particle wherein the weight ratio of the sodium carbonate to the detersive surfactant in the spray-dried particle is less than 1.

2. A process according to claim 1 wherein the aqueous slurry comprises 14 wt.% to 36 wt.% sodium carbonate.

3. A process according to claim 1 or 2 wherein the amount of alkali metal silicate remaining in the aqueous slurry after forming in-situ silicate salt or disilicate salt of alkaline earth metal is from 3 wt.% to 12 wt.% of the aqueous slurry.

4. A process according to any one of the preceding claims wherein the alkaline earth metal salt is selected calcium sulphate, calcium chloride, magnesium sulphate, magnesium chloride or combinations thereof, preferably magnesium sulphate.

5. A process according to any one of the preceding claims wherein the alkali metal silicate is calcium silicate or sodium silicate, preferably sodium silicate.

6. A process according to any one of the preceding claims wherein the in-situ formed silicate or disilicate salt of alkaline earth metal is at least 50% amorphous.

7. A process according to any one of the preceding claims wherein the weight ratio of the alkali metal silicate to the alkaline earth metal salt added to the aqueous mixture is in the range from 260:1 to 5:1, preferably 24:1 to 12:1.

8. A process according to any one of the preceding claims wherein the process involves the step of adding a filler to the aqueous slurry, preferably the filler is a neutral salt or a mineral, more preferably selected from sodium sulphate, sodium chloride, calcium carbonate, magnesium carbonate or mixtures thereof.

9. A process according to any one of the preceding claims wherein the detersive surfactant is an anionic surfactant, preferably selected from the group consisting of alkyl benzene sulfonate, alkoxylated alkyl sulphate, alkyl sulphate, alkoxylated alcohol; and mixtures thereof.

10. A process according to claim 9 wherein the anionic surfactant is selected from a fully neutralized anionic surfactant salt, a partially neutralized anionic surfactant or an acid form of the anionic surfactant which is fully neutralized to the salt form in-situ.

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

(i) 21 wt.% to 40 wt.%, preferably 21 wt.% to 36 wt.% detersive surfactant;
(ii) 0.06 wt.% to 2 wt.% silicate salt and/or disilicate salt of alkaline earth metal;
(iii) 14 wt.% to 36 wt.% sodium carbonate;
(iv) 3 wt.% to 12 wt.% alkali metal silicate;
(v) 30 wt.% to 40 wt.% water.
(vi) preferably from 0.7 wt.% to 25 wt.% filler selected from sodium sulphate, magnesium carbonate, sodium chloride, calcium carbonate, calcite, dolomite or mixtures thereof;
(vii) optionally, from 0 wt.% to 2 wt.% hydroxide salt of alkaline earth metal.
(viii) optionally, from 0 wt.% to 2 wt.% unreacted alkaline earth metal salt.

12. A spray-dried detergent particle obtainable from the process according to any one of the preceding claims, wherein the spray-dried detergent particle comprises:

(i) 31 wt.% to 55 wt.% detersive surfactant;
(ii) 0.1 wt.% to 2.5 wt.% in-situ formed silicate salt and/or disilicate salt of alkaline earth metal salt;
(iii) 22 wt.% to 54 wt.%, preferably 25 wt.% to 50 wt.% sodium carbonate;
(iv) 5 wt.% to 17 wt.% alkali metal silicate salt;
(v) preferably from 1 wt.% to 25 wt.% filler selected from sodium sulphate, magnesium carbonate, sodium chloride, calcium carbonate, calcite, dolomite or mixtures thereof;
(vi) optionally from 0 wt.% to 2.5 wt.% hydroxide of the alkaline earth metal;
(vii) optionally from 0 wt.% to 2.5 wt% unreacted alkaline earth metal salt;
(viii) optionally from 0 to 5 wt.% water.

13. A spray dried detergent particle according to claim 12 wherein the ratio of the carbonate builder salt to the detersive surfactant wherein the detersive surfactant is LAS active is less than 1.

14. A laundry detergent composition comprising from 5 wt.% to 95 wt.% of the spray dried detergent particle as claimed in any one of the preceding claims.

15. A laundry detergent composition as claimed in claim 14 wherein the composition comprises or more laundry ingredients selected from the group consisting of enzyme, sequestrants, foam boosters, antifoam, perfume, dyes, shading dye, visual cues or mixtures thereof.

Patentansprüche

1. Verfahren zur Herstellung eines sprühgetrockneten Waschmittelpartikels, wobei das Verfahren die Schritte umfasst:

(i) Kontaktieren eines Erdalkalimetallsalzes mit einem Alkalimetallsilikatsalz in einer wässrigen Mischung, umfassend ein Reinigungstensid, wobei das Erdalkalimetallsalz mit dem Alkalimetallsilikatsalz reagiert, um in situ Silikatsalz und/oder Disilikatsalz eines Erdalkalimetalls zu bilden;
(ii) Zugabe von Natriumcarbonat, um eine wässrige Aufschlämmung zu bilden, wobei die wässrige Aufschlämmung Alkalimetallsilikat, in situ gebildetes Silikatsalz und/oder Disilikatsalz des Erdalkalimetalls, 21 bis 40 Gew.-% Reinigungstensid und Natriumcarbonat umfasst;
(iii) Sprühtrocknen der wässrigen Aufschlämmung, um das sprühgetrocknete Waschmittelpartikel zu bilden, wobei das Gewichtsverhältnis des Natriumcarbonats zu dem Reinigungstensid in dem sprühgetrockneten Partikel weniger als 1 beträgt.

2. Verfahren nach Anspruch 1, wobei die wässrige Aufschlämmung 14 bis 36 Gew.-% Natriumcarbonat umfasst.

3. Verfahren nach Anspruch 1 oder 2, wobei die Menge des Alkalimetallsilikats, die in der wässrigen Aufschlämmung nach der in situ-Bildung von Silikatsalz oder Disilikatsalz des Erdalkalimetalls verbleibt, 3 bis 12 Gew.-% der wässrigen Aufschlämmung beträgt.

4. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Erdalkalimetallsalz unter Calciumsulfat, Calciumchlorid, Magnesiumsulfat, Magnesiumchlorid oder Kombinationen davon, bevorzugt Magnesiumsulfat, ausgewählt wird.

5. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Alkalimetallsilikat Calciumsilikat oder Natriumsilikat, bevorzugt Natriumsilikat, ist.

6. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das in situ gebildete Silikat- oder Disilikatsalz des Erdalkalimetalls zu mindestens 50% amorph ist.

7. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Gewichtsverhältnis des Alkalimetallsilikats zu dem Erdalkalimetallsalz, das der wässrigen Mischung zugesetzt wird, in dem Bereich von 260:1 bis 5:1, bevorzugt von 24:1 bis 12:1, liegt.

8. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Verfahren den Schritt des Hinzufügens eines Füllstoffs zu der wässrigen Aufschlämmung einbezieht, wobei der Füllstoff bevorzugt ein Neutralsalz oder ein Mineral ist, bevorzugter ausgewählt unter Natriumsulfat, Natriumchlorid, Calciumcarbonat, Magnesiumcarbonat oder Mischungen davon.

9. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Reinigungstensid ein anionisches Tensid ist, bevorzugt ausgewählt aus der Gruppe, bestehend aus Alkylbenzolsulfonat, alkoxyliertem Alkylsulfat, Alkylsulfat, alkoxyliertem Alkohol und Mischungen davon.

10. Verfahren nach Anspruch 9, wobei das anionische Tensid unter vollständig neutralisiertem anionischen Tensidsalz, einem partiell neutralisierten anionischen Tensid oder einer Säureform des anionischen Tensids, die in situ vollständig zur Salzform neutralisiert wird, ausgewählt ist.

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

(i) 21 bis 40 Gew.-%, bevorzugt 21 bis 36 Gew.-% Reinigungstensid;
(ii) 0,06 bis 2 Gew.-% Silikatsalz und/oder Disilikatsalz des Erdalkalimetalls;
(iii) 14 bis 36 Gew.-% Natriumcarbonat;
(iv) 3 bis 12 Gew.-% Alkalimetallsilikat;
(v) 30 bis 40 Gew.-% Wasser;
(vi) bevorzugt 0,7 bis 25 Gew.-% Füllstoff, ausgewählt unter Natriumsulfat, Magnesiumcarbonat, Natriumchlorid, Calciumcarbonat, Calcit, Dolomit oder Mischungen davon;
(vii) optional 0 bis 2 Gew.-% Hydroxidsalz des Erdalkalimetalls;
(viii) optional 0 bis 2 Gew.-% nicht umgesetztes Erdalkalimetallsalz.

12. Sprühgetrocknetes Waschmittelpartikel, erhältlich durch das Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das sprühgetrocknete Waschmittelpartikel umfasst:

(i) 31 bis 55 Gew.-% Reinigungstensid;
(ii) 0,1 bis 2,5 Gew.-% in situ-gebildetes Silikatsalz und/oder Disilikatsalz eines Erdalkalimetallsalzes;
(iii) 22 bis 54 Gew.-%, bevorzugt 25 bis 50 Gew.-% Natriumcarbonat;
(iv) 5 bis 17 Gew.-% Alkalimetallsilikatsalz;
(v) bevorzugt 1 bis 25 Gew.-% Füllstoff, ausgewählt unter Natriumsulfat, Magnesiumcarbonat, Natriumchlorid, Calciumcarbonat, Calcit, Dolomit oder Mischungen davon;
(vi) optional 0 bis 2,5 Gew.-% Hydroxid des Erdalkalimetalls;
(vii) optional 0 bis 2,5 Gew.-% nicht umgesetztes Erdalkalimetallsalz;
(viii) optional 0 bis 5 Gew.-% Wasser.

13. Sprühgetrocknetes Waschmittelpartikel nach Anspruch 12, wobei das Verhältnis des Carbonatbuildersalzes zu dem Reinigungstensid weniger als 1 beträgt, wobei das Reinigungstensid LAS-aktiv ist.

14. Wäschewaschmittelzusammensetzung, umfassend 5 bis 95 Gew.-% des sprühgetrockneten Waschmittelpartikels, wie in irgendeinem der vorhergehenden Ansprüche beansprucht.

15. Wäschewaschmittelzusammensetzung, wie in Anspruch 14 beansprucht, wobei die Zusammensetzung Waschmittelbestandteile umfasst, ausgewählt aus der Gruppe, bestehend aus Enzym, Komplexbildnern, Schaumverstärkern, Antischaummittel,
Parfüm, Farbstoffen, Schattierungsfarbstoff, optischen Hinweisen oder Mischungen davon.

Revendications

1. Procédé de préparation d'une particule de détergent séchée par pulvérisation, ledit procédé comprenant les étapes de :

    (i) mise en contact d'un sel de métal alcalino-terreux avec un sel de silicate de métal alcalin dans un mélange aqueux comprenant un tensioactif détersif, le sel de métal alcalino-terreux réagissant avec le sel de silicate de métal alcalin pour former in situ un sel de silicate et/ou un sel de disilicate de métal alcalino-terreux ;
    (ii) ajout de carbonate de sodium pour former une suspension aqueuse, la suspension aqueuse comprenant un silicate de métal alcalin, un sel de silicate et/ou un sel de disilicate de métal alcalino-terreux formé in situ, de 21 % en poids à 40 % en poids de tensioactif détersif et de carbonate de sodium ;
    (iii) séchage par pulvérisation de ladite suspension aqueuse pour former ladite particule de détergent séchée par pulvérisation, le rapport en poids du carbonate de sodium au tensioactif détersif dans la particule séchée par pulvérisation étant inférieur à 1.

2. Procédé selon la revendication 1, dans lequel la suspension aqueuse comprend de 14 % en poids à 36 % en poids de carbonate de sodium.

3. Procédé selon la revendication 1 ou 2, dans lequel la quantité de silicate de métal alcalin restant dans la suspension aqueuse après formation in situ de sel de silicate ou de sel de disilicate de métal alcalino-terreux est de 3 % en poids à 12 % en poids de la suspension aqueuse.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel le sel de métal alcalino-terreux est choisi parmi le sulfate de calcium, le chlorure de calcium, le sulfate de magnésium, le chlorure de magnésium ou des combinaisons de ceux-ci, de préférence le sulfate de magnésium.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel le silicate de métal alcalin est le silicate de calcium ou le silicate de sodium, de préférence le silicate de sodium.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel le sel de silicate ou disilicate de métal alcalino-terreux formé in situ est amorphe à au moins 50 %.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel le rapport en poids du silicate de métal alcalin au sel de métal alcalino-terreux ajouté au mélange aqueux est dans la plage de 260:1 à 5:1, de préférence de 24:1 à 12:1.

8. Procédé selon l'une quelconque des revendications précédentes, le procédé comprenant l'étape d'ajout d'une charge à la suspension aqueuse, la charge étant de préférence un sel neutre ou un minéral, plus préférablement choisi parmi le sulfate de sodium, le chlorure de sodium, le carbonate de calcium, le carbonate de magnésium ou des mélanges de ceux-ci.

9. Procédé selon l'une quelconque des revendications précédentes, dans lequel le tensioactif détersif est un tensioactif anionique, de préférence choisi dans le groupe constitué d'un alkylbenzènesulfonate, d'un alkylsulfate alcoxylé, d'un alkylsulfate, d'un alcool alcoxylé ; et des mélanges de ceux-ci.

10. Procédé selon la revendication 9, dans lequel le tensioactif anionique est choisi parmi un sel de tensioactif anionique complètement neutralisé, un tensioactif anionique partiellement neutralisé ou une forme acide du tensioactif anionique qui est complètement neutralisée en forme de sel in situ.

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

(i) 21 % en poids à 40 % en poids, de préférence 21 % en poids à 36 % en poids de tensioactif détersif ;
(ii) 0,06 % en poids à 2 % en poids de sel de silicate et/ou sel de disilicate de métal alcalino-terreux ;
(iii) 14 % en poids à 36 % en poids de carbonate de sodium ;
(iv) 3 % en poids à 12 % en poids de silicate de métal alcalin ;
(v) 30 % en poids à 40 % en poids d'eau ;
(vi) de préférence de 0,7 % en poids à 25 % en poids d'une charge choisie parmi le sulfate de sodium, le carbonate de magnésium, le chlorure de sodium, le carbonate de calcium, la calcite, la dolomite ou des mélanges de ceux-ci ;
(vii) éventuellement, de 0 % en poids à 2 % en poids de sel d'hydroxyde de métal alcalino-terreux ;
(viii) éventuellement, de 0 % en poids à 2 % en poids de sel de métal alcalino-terreux n'ayant pas réagi.

12. Particule de détergent séchée par pulvérisation pouvant être obtenue par le procédé selon l'une quelconque des revendications précédentes, la particule de détergent séchée par pulvérisation comprenant :

(i) 31 % en poids à 55 % en poids de tensioactif détersif ;
(ii) 0,1 % en poids à 2,5 % en poids de sel de silicate et/ou sel de disilicate de métal alcalino-terreux formé in situ ;
(iii) 22 % en poids à 54 % en poids, de préférence 25 % en poids à 50 % en poids de carbonate de sodium ;
(iv) 5 % en poids à 17 % en poids de sel de silicate de métal alcalin ;
(v) de préférence de 1 % en poids à 25 % en poids d'une charge choisie parmi le sulfate de sodium, le carbonate de magnésium, le chlorure de sodium, le carbonate de calcium, la calcite, la dolomite ou des mélanges de ceux-ci ;
(vi) éventuellement de 0 % en poids à 2,5 % en poids d'hydroxyde de métal alcalino-terreux ;
(vii) éventuellement de 0 % en poids à 2,5 % en poids de sel de métal alcalino-terreux n'ayant pas réagi ;
(viii) éventuellement de 0 à 5 % en poids d'eau.

13. Particule de détergent séchée par pulvérisation selon la revendication 12, dans laquelle le rapport de l'adjuvant de carbonate au tensioactif détersif, le tensioactif détersif étant un agent actif LAS, est inférieur à 1.

14. Composition de détergent de lessive comprenant de 5 % en poids à 95 % en poids de particules de détergent séchées par pulvérisation selon l'une quelconque des revendications précédentes.

15. Composition de détergent de lessive selon la revendication 14, dans laquelle la composition comprend un ou plusieurs composants de lessive choisis dans le groupe constitué d'une enzyme, de séquestrants, d'agents moussants, d'un antimousse, d'un parfum, de colorants, d'un colorant d'ombrage, de repères visuels ou des mélanges de ceux-ci.

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