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(54) **SPRAY-DRIED POWDER DETERGENT COMPRISING LIGHT SILICATE**

(57) A solid laundry detergent composition having a bulk density of 600 g/L or less, comprising relative to the total weight of the composition:
a) 60 to 95 wt.-% of a spray-dried powder, comprising relative to the total weight of the spray-dried powder:
a1) 5 to 60 wt.-% of a detersive surfactant;
a2) 0 to 50 wt.-% carbonate salt;
a3) 0 to 50 wt.-% sulfate salt;
a4) less than 5 wt.-% phosphate builder;
a5) less than 5 wt.-% aluminosilicate builder;
a6) 0 to 15 wt.-% water; and
a7) 1 to 25 wt.-% silicate salt;
wherein the spray-dried powder has a bulk density of up to 800 g/L and
b) 1 to 40 wt.-% of a dried light density silicate salt having a bulk density of less than 400 g/L.

Process for the preparation of the composition by spray drying of powder a) and combining it with b)

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Description

[0001] The present invention relates to a solid laundry detergent composition comprising a spray-dried powder comprising a detersive surfactant and silicate and a dried light density silicate salt. The present invention also relates to a process for preparing such a solid laundry detergent composition comprising dried light density silicate salt.

[0002] Laundry detergents are widely used in powder form. The majority of these detergent powders include or consist of a spray-dried powder component produced in a typically continuous process in a spray drying tower. While it would be desirable to provide stable powder detergents with low density, the spray-dried powders of low bulk density tend to compact over time, since during storage the comparably instable low density spray-dried powder particles are susceptible to crumbling, thus forming dust, with the result that the bulk density of the powder increases over time. The consumer thus ends up with a product having a very high bulk density, which is undesirable for a number of reasons, such as ease of dosing, solubility, dust exposure and the like. Furthermore, the production of low density spray-dried detergent powders is highly energy consuming and therefore causes high carbon dioxide emissions. Finally, the low density spray dried detergent powders tend to have higher segregation, lumping and suboptimal solubility.

[0003] The above problems are particularly encountered when detergent powders free of phosphate and zeolite builders are to be produced and also occur when low density silicate salts are used in the spray-drying processes to produce the solid detergent powders.

[0004] European patent application EP 2 123 743 A1 describes that the incorporation of a silicate salt that has a low bulk density and a very small weight average particle size into chemically compacted zeolite-free formulations enables them to be produced using traditional batch processes, and even a batch single mixer processes and provides a solid laundry detergent composition and a process for its preparation including such a silicate salt. While the compositions described in this document show good mechanical properties, they are limited to those produced in a batch process.

[0005] In view of the above, it would be desirable to provide a process for the continuous production of spray-dried powder detergent compositions that overcomes the above-mentioned drawbacks and provides a low bulk density solid powder detergent composition with good mechanical properties, in particular good particle stability over extended periods of storage, good solubility and similar or increased wash performance. At the same time, said powders should be less susceptible to segregation, lumping and dispensability loss.

[0006] The inventors of the present invention have surprisingly found that sad objects are met by a solid laundry detergent composition that comprises, in addition to a conventionally produced spray-dried powder detergent, a dried light density silicate salt in amounts of at least 1 wt.-%. It could be shown that such compositions that comprise in addition to the spray-dried powder detergent particles, particles of light density silicate salts, in particular spray-dried particles of said silicate salt, are more resistant towards segregation and lumping and show increased mechanical properties and good wash performance.

[0007] In a first aspect, the present invention therefore relates to a solid laundry detergent composition having a bulk density of 600 g/L or less, comprising relative to the total weight of the composition:

a) 60 to 95 wt.-%, preferably 70 to 95 wt.-% of a spray-dried powder, comprising relative to the total weight of the spray-dried powder:

- a1) 5 to 60 wt.-% of a detersive surfactant;
- a2) 0 to 50 wt.-% carbonate salt;
- a3) 0 to 50 wt.-% sulfate salt;
- a4) less than 5 wt.-% phosphate builder;
- a5) less than 5 wt.-% aluminosilicate builder;
- a6) 0 to 15 wt.-% water; and
- a7) 1 to 25 wt.-% silicate salt;

wherein the spray-dried powder has a bulk density of up to 800 g/L, preferably 350 to 600 g/L; and

b) 1 to 40 wt.-% of a dried light density silicate salt having a bulk density of less than 400 g/L.

[0008] Another aspect of the invention is directed to a process for the preparation of a solid laundry detergent composition as described herein, the process comprising the steps of

A) spray drying an aqueous slurry comprising a detersive surfactant, silicate salt, water and optionally a carbonate salt and/or sulfate salt to obtain the spray-dried powder a); and

B) combining the spray-dried powder obtained in step A) with the dried light density silicate salt b) to obtain the solid laundry detergent composition.

[0009] "At least one", as used herein, relates to one or more, i.e. 1, 2, 3, 4, 5, 6, 7, 8, 9, or more. If used in combination with a compound, the term does not relate to the absolute number of molecules but rather to the number of different types of said compound. "At least one surfactant" thus means that at least one type but that also 2 or more different surfactant types can be present.

[0010] If not indicated otherwise, all viscosities referred to herein are viscosities measured at 20°C by a Brookfield LVT, Spindle No. 3 at 12 rpm.

[0011] If not indicated otherwise, all percentages are by weight relative to the total weight of the composition.

[0012] "Free of", as used herein in relation to a specific type of component, means that the referenced composition does not contain the respective component in deliberately added form. In various embodiments, this means that the respective component is present in concentrations of no more than 1 wt.%, preferably no more than 0.5 wt.%, more preferably no more than 0.1 wt.% of said component relative to the total weight of the composition. Most preferably, said component is not contained at all.

[0013] The detergent compositions of the present invention can be used as detergents for textiles, carpets or natural fibers.

[0014] "Wash(ing) performance", as used herein, relates to the removal of stains, in particular stains sensitive to surfactants. The removal can be evaluated by measuring a brightening of the stain either instrumentally or by visual inspection.

[0015] The solid laundry detergent composition of the invention has a bulk density of 600g/l or less, preferably 500g/l or less, or 450g/l or less, or 400g/l or less, or even 350g/l or less. A method for measuring the bulk density of a powder is described in more detail below. The solid laundry detergent composition typically has a cake strength of from 5N to 20N.

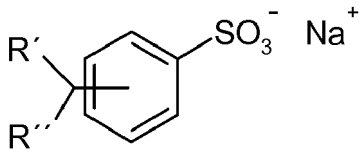
[0016] The solid laundry detergent composition of the invention comprises as a first component, 60 to 95 wt.-%, preferably 70 to 95 wt.-% of a spray-dried powder, comprising relative to the total weight of the spray-dried powder:

a1) 5 to 60 wt.-% of a detersive surfactant;
a2) 0 to 50 wt.-% carbonate salt;
a3) 0 to 50 wt.-% sulfate salt;
a4) less than 5 wt.-% phosphate builder;
a5) less than 5 wt.-% aluminosilicate builder;
a6) 0 to 15 wt.-% water; and
a7) 1 to 25 wt.-% silicate salt

[0017] The spray-dried powder component comprises from 5 wt.-% to 60 wt.-% of a detersive surfactant. The detersive surfactant can be selected from anionic detersive surfactants, cationic detersive surfactants, nonionic detersive surfactants, zwitterionic detersive surfactants, amphoteric detersive surfactants, and mixtures thereof.

[0018] Preferably, the detersive surfactant comprises anionic detersive surfactant. Suitable anionic detersive surfactants are alkoxylated alcohol sulfate anionic detersive surfactants such as linear or branched, substituted or unsubstituted ethoxylated C12-18 alcohol sulfates having an average degree of ethoxylation of from 1 to 10, preferably from 3 to 7. Other suitable anionic detersive surfactant are alkyl benzene sulfonate anionic detersive surfactants such as linear or branched, substituted or unsubstituted C8-18 alkyl benzene sulfonates, preferably linear unsubstituted C10-13 alkyl benzene sulfonates. Other suitable anionic detersive surfactants are alkyl sulfates, alkyl sulfonates, alkyl carboxylates or any mixture thereof.

[0019] In various embodiments, the detersive surfactant comprises at least one alkyl benzene sulfonate. Exemplary alkyl benzene sulfonates include, but are not limited to linear and branched alkyl benzene sulfonates, preferably linear alkyl benzene sulfonates. Exemplary compounds are those of formula (I)



(I),

wherein R' and R" are independently H or alkyl and combined comprise 9 to 19, preferably 9 to 15 and more preferably 9 to 13 carbon atoms. Particularly preferred are dodecyl and tridecyl benzene sulfonates, in particular the sodium salts thereof. Preferred contents of the alkyl benzene sulfonates range from 10 to 25 wt.-%, preferably 12.0 to 20.0 wt.%, relative to the total weight of the spray dried powder. While reference is made herein to the sulfonates and particularly the sodium salts thereof, it is understood that the invention also encompasses salts with other metals, ammonium or organic bases, such as alkanolamines. Further, it is understood that also the free acid forms, also referred to as "acidic

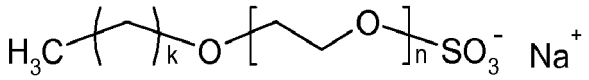
precursors" may be used.

[0020] The detersive surfactant may further or alternatively comprises at least one alkyl ether sulfate. Preferred alkyl ether sulfates are those of formula (II)

$$R^1\text{-}O\text{-}(AO)_n\text{-}SO_3^-\ X^+ \qquad \text{(II)}.$$

In formula (II) R^1 represents a linear or branched, substituted or unsubstituted alkyl group, preferably a linear, unsubstituted alkyl group, more preferably a fatty alcohol moiety. Preferred R^1 moieties are selected from the group consisting of decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl, eicosyl moieties and mixtures thereof, wherein those groups with an even number of carbon atoms are preferred. Particularly preferred R^1 moieties are derived from C_{10}-C_{18} fatty alcohols, such as those derived from coconut oil alcohols, tallow fatty alcohols, lauryl, myristyl, cetyl or stearyl alcohol or from C_{10}-C_{20} oxoalcohols. AO represents an ethyleneoxide (EO) or propyleneoxide (PO) group, preferably an ethyleneoxide group. The index n represents an integer from 1 to 50, preferably from 1 to 20 and more preferably from 1 to 10. Particularly preferably, n is 1, 2, 3, 4, 5, 6, 7 or 8. X represents a monovalent cation or the n-th part of an n-valent cation, preferred are alkali metal cations, specifically Na^+ and K^+, most preferably Na^+. Further cations X^+ may be selected from NH_4^+, ½ Zn^{2+},½ Mg^{2+},½ Ca^{2+},½ Mn^{2+}, and combinations thereof.

[0021] In various embodiments, the detersive surfactants comprise an alkyl ether sulfate selected from fatty alcohol ether sulfates of formula (III)



(III)

wherein k = 9 to 19, and n = 1, 2, 3, 4, 5, 6, 7 or 8. Preferred are C_{10-16} fatty alcohol ether sulfates with 1-7, more preferably 1-3 EO (k = 9-15, n = 1-7, 1-3), even more preferred the C_{12-14} fatty alcohol ether sulfates with 1-3, particularly 2 EO (k = 11-13, n = 1-3 or 2). The level of ethoxylation is an average value and can, for a specific compound, be an integer or fractional number. The alkyl ether sulfate may be contained in the spray dried powder in an amount of 2.0 to 8.0 wt.% relative to the total weight of the powder, preferably 3.0 to 7.0 wt.%.

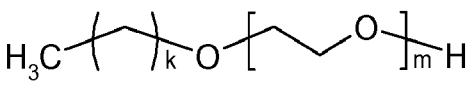
[0022] The detersive surfactant may also comprise non-ionic detersive surfactants. Suitable non-ionic detersive surfactants are selected from: C8-18 alkyl alkoxylated alcohols having an average degree of alkoxylation of from 1 to 20, preferably from 3 to 10, most preferred are C12-18 alkyl ethoxylated alcohols having an average degree of alkoxylation of from 3 to 10; and mixtures thereof.

[0023] Preferred nonionic surfactants are those of formula (IV)

$$R^2\text{-}O\text{-}(AO)_m\text{-}H \qquad \text{(IV)},$$

wherein R^2 represents a linear or branched substituted or unsubstituted alkyl moiety, AO represents an ethylene oxide (EO) or propylene oxide (PO) group and m is an integer from 1 to 50. In formula (IV) R^2 preferably represents a linear or branched, substituted or unsubstited alkyl group, preferably a linear, unsubstituted alkyl group, particularly preferred a fatty alcohol group. Preferred groups are R^2 are selected from decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl, eicosyl groups and combinations thereof, wherein those groups with an even number of carbon atoms are preferred. Particularly preferred are R^2 groups derived from C_{12}-C_{18} fatty alcohols, such as coconut oil alcohol, tallow oil alcohol, lauryl, myristyl, cetyl or stearyl alcohol or from C_{10}-C_{20} oxoalcohols. AO represents an ethyleneoxide (EO) or propyleneoxide (PO) group, preferably an ethyleneoxide group. The index m represents an integer from 1 to 50, preferably from 1 to 20 and more preferably from 3 to 10. Particularly preferably, m is 3, 4, 5, 6 or 7.

[0024] In various preferred embodiments, the detergent compositions comprise an alkyl ether selected from fatty alcohol ethers of formula (V)



(V)

wherein k = 11 to 19, m = 3, 4, 5, 6 or 7. Preferred are C_{12-18} fatty alcohols with 3-7 EO (k = 11-17, m = 3-7 in formula (V)). Such nonionic alkyl ethers may be contained in the spray dried powder in amounts of 0.0 to 5.0 wt.%, preferably 2.0 to 3.0 wt.%.

[0025] The detergents may further include other nonionic surfactants, such as alkyl glucosides of the general formula $RO(G)_x$, where R is a primary linear or 2-methyl-branched aliphatic radical containing 8 to 22 and preferably 12 to 18 carbon atoms and G stands for a glucose unit. The degree of oligomerization x, which indicates the distribution of monoglucosides and oligoglucosides, is a number of 1 to 10 and preferably a number of 1.2 to 1.4.

[0026] In various embodiments, the detersive surfactant comprises at least two anionic surfactants, namely at least one alkyl ether sulfate and preferably at last one alkyl benzene sulfonate, and optionally an alkyl ether.

[0027] The detersive surfactant may also comprise cationic detersive surfactants. Preferred cationic detersive surfactants are mono-C6-18 alkyl mono-hydroxyethyl di-methyl quaternary ammonium chlorides, more preferred are mono-C8-10 alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride, mono-C10-12 alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride and mono-C10 alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride.

[0028] Suitable amphoteric and zwitterionic surfactants include amine oxides and betaines.

[0029] The spray-dried powder comprises from 0 wt.-% to 50 wt.-%, preferably 1 to 45, more preferably 10 to 30 wt.-%, carbonate salt. A preferred carbonate salt is sodium carbonate, sodium bicarbonate and a mixture thereof. A most preferred carbonate salt is sodium carbonate.

[0030] The spray-dried powder comprises from 0 wt.-% to 50 wt.-%, preferably 1 to 45, more preferably 10 to 30 wt.-% sulfate salt. A preferred sulfate salt is sodium sulfate.

[0031] The spray-dried powder comprises less than 5 wt.-%, preferably from 0 wt.-% to < 2wt.-%, more preferably less than 1 wt.-% phosphate builder. It may even be preferred for the composition to be free from phosphate builder. Phosphate builders include sodium tripolyphosphate. Preferably, the solid laundry detergent composition is essentially free of or free of phosphate.

[0032] The spray-dried powder comprises less than 5 wt.-%, preferably from 0 wt.-% to < 2wt.-%, more preferably less than 1 wt.-% aluminosilicate builder. It may even be preferred for the composition to be free from aluminosilicate builder. Aluminosilicate builders include zeolite, such as zeolite A, zeolite X, zeolite P and zeolite MAP. Preferably, the solid laundry detergent composition is essentially free of or free of aluminosilicate, in particular zeolite builder.

[0033] The spray-dried powder typically comprises 0 to 15 wt.-%, preferably 0 to 12, more preferably from 0 to 3 wt.-% water. The method for determining the moisture level of the solids described herein is described in more detail below.

[0034] The spray-dried powder further comprises a silicate salt, in particular sodium silicate. Suitable silicate salts include crystalline sodium silicates of the general formula $NaMSi_xO_{2x+1}\cdot yH_2O$, wherein M represents sodium or hydrogen, x represents an number from 1,9 to 4 and y represents a number from 0 to 20, with x preferably being 2, 3 or 4. Such crystalline silicate salts are, for example, described in the European patent application EP-A-0 164 514. Particularly preferred are crystalline silicates of the above formula, wherein M represents sodium and x is 2 or 3. Particularly preferred are beta- as well as delta-sodium disilicates of the formula $Na_2Si_2O_5\cdot yH_2O$.

[0035] Also preferred are amorphous sodium silicates with a module $Na_2O:SiO_2$ of 1:2 to 1:3,3, preferably 1:2 to 1:2,8 and more preferably 1:2 to 1:2,6. "Amorphous", as used herein, includes "X-ray amorphous", meaning that the silicates in X-ray diffraction measurements do not yield sharp peaks, typical for crystalline substances, but rather one or more broad maxima. Such X-ray amorphous silicates are, for example, described in DE-A-44 00 024.

[0036] The silicate salt used in the spray-dried powder may be a light density silicate salt, similar to the silicate salt component b). Such light density silicate salt generally has a bulk density of less than 400g/l, preferably less than 350g/l, or less than 300g/l, or less than 250g/l, or less than 200g/l, or less than 150g/l, or less than 100g/l. Preferably the bulk density is in the range of 75 to 150 g/l. Typically, the light density silicate salt is a sodium silicate salt, such as those described above.

[0037] In one aspect, the light density silicate has a weight average particle size of less than 300 micrometers, or less than 200 micrometers, or even less than 100 micrometers. Preferred are weight average particle sizes of 100 micrometers or less. Typically, the light density silicate salt is obtainable, or obtained, by a flash-drying process or by spray-drying.

[0038] The silicate salt content in the spray-dried powder may range from 7 to 20 wt.-%, relative to the total weight of the spray-dried powder. This silicate salt a7) is preferably a light density silicate salt, as defined above and/or comprises, consists essentially of or consists of sodium silicate.

[0039] In various embodiments, the composition comprises, relative to the total weight of the composition, 1 to 25, preferably 1 to 20, more preferably 2 to 15 wt.-% dried light density silicate salt b). It has been found that each 1 wt.-% of the light density silicate salt of a bulk density of about 100 g/l decreases the bulk density of the solid composition by about 20 g/l. "About", as used herein, relates to the numerical value $\pm$10%, preferably $\pm$5%.

[0040] The dried light density silicate salt may be the light density silicate salt described above. In various embodiments, the dried light density silicate salt b) is a flash-dried or spray-dried light density silicate salt and/or has a bulk density in the range of about 75 to about 150 g/L and/or has a water content of less than 5 wt.-%, preferably less than 3 wt.-% and/or has a weight average particle size of less than 300 micrometers, or less than 200 micrometers, or even less than 100 micrometers, and/or comprises, consists essentially of or consists of sodium silicate.

[0041] In addition to the afore-mentioned components, the composition may comprise adjunct detergent ingredients

c). Suitable adjunct detergent ingredients are selected from: bleaches, in particular a source of peroxygen such as percarbonate salts and/or perborate salts, preferred is sodium percarbonate, the source of peroxygen is preferably at least partially coated, preferably completely coated, by a coating material such as a carbonate salt, a sulphate salt, a silicate salt, borosilicate, or mixtures, including mixed salts, thereof; bleach activator such as tetraacetyl ethylene diamine, oxybenzene sulphonate bleach activators such as nonanoyl oxybenzene sulphonate, caprolactam bleach activators, imide bleach activators such as N-nonanoyl-N-methyl acetamide, preformed peracids such as N,N-pthaloylamino peroxycaproic acid, nonylamido peroxyadipic acid or dibenzoyl peroxide; polymeric carboxylates, preferably copolymers of maleic acid and acrylic acid and salts thereof; enzymes such as amylases, carbohydrases, cellulases, laccases, lipases, oxidases, peroxidases, proteases, pectate lyases and mannanases; suds suppressing systems such as silicone based suds suppressors; fluorescent whitening agents; photobleach; fabric-softening agents such as clay, silicone and/or quaternary ammonium compounds; flocculants such as polyethylene oxide; dye transfer inhibitors such as polyvinylpyrrolidone, poly 4-vinylpyridine N-oxide and/or copolymer of vinylpyrrolidone and vinylimidazole; fabric integrity components such as hydrophobically modified cellulose and oligomers produced by the condensation of imidazole and epichlorhydrin; soil dispersants and soil anti-redeposition aids such as alkoxylated polyamines and ethoxylated ethyleneimine polymers; anti-redeposition components such as carboxymethyl cellulose and polyesters; perfumes; sulphamic acid or salts thereof; citric acid or salts thereof as well as other organic builders; dyes such as orange dye, blue dye, green dye, purple dye, pink dye, or any mixture thereof; and further complexing agents, such as phosphonates, including those based on hydroxyl alkanes, amino alkanes or amino acids, such as 1-hydroxyethane-1,1-diphosphonate (HEDP), ethylenediamine tetramethylene phosphonate (EDTMP), diethylenetriamine pentamethylene phosphonate (DTPMP), and lysine tetramethylene phosphonate (LTMP). The term "phosphate-free", as used herein, does not relate to phosphonates.

[0042] In various embodiments, the one or more additional components c) are selected from enzymes, perfumes, bleaches, bleach catalysts, complexing agents, and polymers.

[0043] The processes to prepare the solid laundry detergent compositions described herein include spray drying an aqueous slurry comprising a detersive surfactant, silicate salt, water and optionally a carbonate salt and/or sulfate salt to obtain the spray-dried powder a). All other components present in spray-dried powder a) should also be included in the slurry or may be added afterwards to the spray-dried particles. However, it is preferred that all components a1) to a7), if present, are also included in the slurry, which is subjected to spray-drying. The slurry is preferably an aqueous slurry, with water as the main or only solvent. The spray-drying is preferably carried out by conventional means in a spray-drying tower in a continuous process, all of which are well-known to those skilled in the art.

[0044] The thus obtained spray-dried powder is in a second step combined with the dried light density silicate salt b) to obtain the solid laundry detergent composition. To achieve this, the spray-dried powder may be introduced in a mixer and the light density silicate salt may be added to said mixer. The mixer may be any suitable mixer known in the art for that purpose. In preferred embodiments, the spray-dried powder and the light density silicate salt are simply combined without excessive mixing to keep mechanical stress low.

[0045] In the processes of the invention, the light density silicate salt may itself be produced by spray-drying, preferably in a continuous process in a spray-drying tower. In such embodiments, the silicate salt is introduced in form of an aqueous slurry into the spray-drying tower. Alternatively, the light density silicate salt may be produced by flash-drying.

[0046] The above-described adjunct components may be added to the spray-dried powder and/or the silicate salt at any stage of the process after spray-drying is completed.

[0047] To determine the bulk density of a solid component or powder, as described herein, the following method is used: A 500 ml graduated cylinder is filled with a powder, the weight of the sample is measured and the bulk density of the powder is calculated in g/l. Typically, the following equipment is used:

Balance. The balance has a sensitivity of 0.5g.

Graduated cylinder. The graduated cylinder has a capacity 500ml. The cylinder should be calibrated at the 500ml mark, by using 500g of water at 20°C. The cylinder is cut off at the 500ml mark and ground smooth.

Funnel. The funnel is cylindrical cone, and has a top opening of 110mm diameter, a bottom opening of 40mm diameter, and sides having a slope of 76.4° to the horizontal.

Spatula. The spatula is a flat metal piece having of a length of at least 1.5 times the diameter of the graduated cylinder.

Beaker. The beaker has a capacity of 600ml.

Tray. The tray is either a metal or plastic square, is smooth and level, and has a side length of at least 2 times the diameter of the graduated cylinder.

Ring stand.

Ring clamp.

Metal gate. The metal gate is a smooth circular disk having a diameter of at least greater than the diameter of the bottom opening of the funnel.

[0048] The measurement is carried out indoors at conditions of 20°C temperature, 1013 mbar pressure and a relative

humidity of 25%.

[0049] The procedure is as follows:

1. Weigh the graduated cylinder to the nearest 0.5g using the balance. Place the graduated cylinder in the tray so that it is horizontal with the opening facing upwards.
2. Support the funnel on a ring clamp, which is then fixed to a ring stand such that the top of the funnel is horizontal and rigidly in position. Adjust the height of the funnel so that its bottom position is 38mm above the top centre of the graduated cylinder.
3. Support the metal gate so as to form an air-tight closure of the bottom opening of the funnel.
4. Completely fill the beaker with a 24 hour old powder sample and pour the powder sample into the top opening of the funnel from a height of 2cm above the top of the funnel.
5. Allow the powder sample to remain in the funnel for 10 seconds, and then quickly and completely remove the metal gate so as to open the bottom opening of the funnel and allow the powder sample to fall into the graduated cylinder such that it completely fills the graduated cylinder and forms an overtop. Other than the flow of the powder sample, no other external force, such as tapping, moving, touching, shaking, etc, is applied to the graduated cylinder. This is to minimize any further compaction of the powder sample.
6. Allow the powder sample to remain in the graduated cylinder for 10 seconds, and then carefully remove the overtop using the flat edge of the spatula so that the graduated cylinder is exactly full. Other than carefully removing the overtop, no other external force, such as tapping, moving, touching, shaking, etc, is applied to the graduated cylinder. This is to minimize any further compaction of the powder sample.
7. Immediately and carefully transfer the graduated cylinder to the balance without spilling any powder sample. Determine the weight of the graduated cylinder and its powder sample content to the nearest 0.5g.
8. Calculate the weight of the powder sample in the graduated cylinder by subtracting the weight of the graduated cylinder measured in step 1 from the weight of the graduated cylinder and its powder sample content measured in step 7.
9. Immediately repeat steps 1 to 8 with two other replica powder samples.
10. Determine the mean weight of all three powder samples.
11. Determine the bulk density of the powder sample in g/l by multiplying the mean weight calculated in step 10 by 2.0.

Examples

Example 1: Solid detergent composition

[0050] An aqueous slurry comprising sodium dodecyl benzene sulfonate, dodecyl benzene sulfonic acid, sodium carbonate, sodium silicate, sodium sulfate and carboxymethyl cellulose (CMC) as subjected to a conventional spray drying process to obtain a spray-dried powder with the composition set forth in Table 1 below.

Table 1: Spray-dried powder (all amounts in wt.-% relative to total weight of the powder)

sodium dodecyl benzene sulfonate	18.3
dodecyl benzene sulfonic acid	19.5
sodium carbonate	13.1
sodium silicate	19.7
sodium sulfate	39.9
carboxymethyl cellulose	0.8
dye	0.02
fluorescent whitening agent	0.13
water	Ad 100

[0051] To obtain the solid detergent powder composition, 76.25 wt.-% of the spray-dried powder of Table 1 were combined with 2.5 wt.-% dried light density silicate salt (Britesil C201), 15 wt.-% sodium carbonate, 5 wt.-% sodium sulfate, 0,25 wt.-% perfume and 1 wt.-% VFM premix which is a colored version of the spray-dried tower powder according to Table 1.

Example 2: Evaluation of solid detergent composition

[0052] A solid detergent powder composed of the spray-dried powder of Table 1 above with (E1) and without (R1) 5 wt.-% light density silicate salt was tested with regard to lumping, flowability, and bulk density. The results depicted in Tale 2 show that the inventive composition including the light density silicate salt performed better with respect to lumping, flowability and bulk density than the reference composition.

Table 2: Physical properties

	R1	E1
Bulk density (g/l)	570	500
Lump test 500 g (g)	65	0
Flowability (%)	93	100
pH	10,7	10,7
Particle size distribution (%)		
>2.0 mm	0	0
>1.6 mm	1	1
>0.8 mm	7	7
>0.4 mm	34	34
>0.2 mm	44	44
>0.1 mm	10	10
<0.1 mm	4	4

[0053] For the lump test a powder pellet is pressed. This pellet is exposed an increasing stress (pressure) under certain conditions. The stress at which it breaks is reported as the lump test in grams. More specifically, the test product is filled into a volumetric cup cut to 15 ml. Excess powder is scraped off. The testing product is put into a steel cylinder. A piston is carefully inserted into the stainless steel cylinder while avoiding any compression of the product and charged with a weight of 500 g. After 20 min, the weight is removed, the cylinder is carefully lifted and the product is pushed out of the cylinder. If the pellet falls apart when taking it out of the steel cylinder, the lump test is defined as "0". If the pellet does not fall apart, it is subjected to the lump test. For this a beam balance is used. In the one balance pan is put a 800ml beaker and in the other pan of the balance are little pellets made out of lead within a cup to balance out the weight of the beaker and the pan. The balance has to 1,0g on the scale. The powder pellet is placed beneath the middle of the balance pan with the beaker is pushed up by a lifting platform below the balance pan. The powder pellet has to push the balance pan upwards until the scale shows 0,0g. Then distilled water is poured slowly and steady into the tared beaker. As soon as the powder pellet starts falling apart the water is stopped. The amount of water used to break the powder pellet is weight back on the balance with the accuracy of +/-1g. The amount of water determined by the procedure above without decimal places is reported as "gram lump test" with reference to this method.

[0054] Flowability is determined by use of a flowability apparatus that measures the flow of powder over time compared to standard flowing sand material.

Claims

1. A solid laundry detergent composition having a bulk density of 600 g/L or less, comprising relative to the total weight of the composition:

   a) 60 to 95 wt.-%, preferably 70 to 95 wt.-% of a spray-dried powder, comprising relative to the total weight of the spray-dried powder:

      a1) 5 to 60 wt.-% of a detersive surfactant;
      a2) 0 to 50 wt.-% carbonate salt;
      a3) 0 to 50 wt.-% sulfate salt;

a4) less than 5 wt.-% phosphate builder;
a5) less than 5 wt.-% aluminosilicate builder;
a6) 0 to 15 wt.-% water; and
a7) 1 to 25 wt.-% silicate salt;

wherein the spray-dried powder has a bulk density of up to 800 g/L, preferably 350 to 600 g/L; and
b) 1 to 40 wt.-% of a dried light density silicate salt having a bulk density of less than 400 g/L.

2. The solid laundry detergent composition according to claim 1, wherein the detersive surfactant comprises 10 to 25 wt.-%, relative to the total weight of the spray-dried powder, of a linear alkyl benzene sulfonic acid or salt thereof.

3. The solid laundry detergent composition according to claim 1 or 2, wherein the spray-dried powder comprises relative to the total weight of the spray-dried powder 10 to 30 wt.-% carbonate salt, preferably sodium carbonate.

4. The solid laundry detergent composition according to any one of the preceding claims, wherein the spray-dried powder comprises relative to the total weight of the spray-dried powder 10 to 30 wt.-% sulfate salt, preferably sodium sulfate.

5. The solid laundry detergent composition according to any one of the preceding claims, wherein the spray-dried powder comprises relative to the total weight of the spray-dried powder 0 to 3 wt.-% water.

6. The solid laundry detergent composition according to any one of the preceding claims, wherein the spray-dried powder comprises relative to the total weight of the spray-dried powder

(i) less than 2, preferably less than 1 wt.-% phosphate builder; and/or
(ii) less than 2, preferably less than 1 wt.-% aluminosilicate builder.

7. The solid laundry detergent composition according to any one of the preceding claims, wherein the spray-dried powder

(i) comprises relative to the total weight of the spray-dried powder 7 to 20 wt.-% of a silicate salt; and/or
(ii) wherein the silicate salt a7) is a light density silicate salt; and/or
(iii) wherein the silicate salt a7) comprises, consists essentially of or consists of sodium silicate.

8. The solid laundry detergent composition according to any one of the preceding claims, wherein the composition comprises, relative to the total weight of the composition, 1 to 25, preferably 1 to 20, more preferably 2 to 15 wt.-% dried light density silicate salt b).

9. The solid laundry detergent composition according to any one of the preceding claims, wherein the dried light density silicate salt b)

(i) is a spray-dried light density silicate salt; and/or
(ii) has a bulk density in the range of about 75 to about 150 g/L; and/or
(iii) has a water content of less than 5 wt.-%, preferably less than 3 wt.-%; and/or
(iv) has a weight average particle size of less than 300 micrometers; and/or
(v) comprises, consists essentially of or consists of sodium silicate.

10. The solid laundry composition according to any one of the preceding claims, wherein the composition

(i) has a bulk density of 500 g/L or less, preferably 450 g/L or less or 400 g/L or less, or even 350 g/L or less; and/or
(ii) has a cake strength of 5N to 20 N.

11. The solid laundry composition according to any one of the preceding claims, wherein the composition further comprises one or more additional components c), preferably selected from enzymes, perfumes, bleaches, bleach catalysts, complexing agents, and polymers.

12. Process for the preparation of a solid laundry detergent composition according to any one of claims 1-11, the process comprising the steps of

A) spray drying an aqueous slurry comprising a detersive surfactant, silicate salt, water and optionally a carbonate salt and/or sulfate salt to obtain the spray-dried powder a); and
B) combining the spray-dried powder obtained in step A) with the dried light density silicate salt b) to obtain the solid laundry detergent composition.

13. The process according to claim 12, wherein the process further comprises the step A2) of spray drying an aqueous slurry comprising the light silicate salt to obtain the dried light silicate salt b) prior to step B).

14. The process according to claim 12 or 13, wherein step B) is carried out by introducing the spray-dried powder obtained in step A) in a mixer and adding the dried light density silicate salt and mixing so as to form the solid laundry detergent composition.

15. The process according to any one of claims 12 to 14, wherein the process further comprises step C) of combining the solid laundry detergent composition of step B) with one or more components c).

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EUROPEAN SEARCH REPORT

Application Number
EP 17 17 3958

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Place of search	Date of completion of the search	Examiner
The Hague	18 August 2017	Cetinkaya, Murat

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