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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
PULVÉRISATION

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Description**Field of the invention**

5 **[0001]** The present invention relates to a process for preparing of a particulate, free flowing detergent particle by a slurry making and spray drying technique. 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

10 **[0002]** Typically, granular or particulate 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.

15 **[0003]** In the manufacture of detergent compositions containing the anionic surfactants, the anionic surfactants are often added in their acid form. One reason for this is that linear alkylbenzene sulphonate which is the most commonly used anionic surfactant, is much easier to handle, store and transport in their acid form as compared to the neutralised form. The acid precursor of anionic surfactant is then converted in-situ into their corresponding surfactant salt by neutralisation with either aqueous or dry neutralising agents.

20 **[0004]** The neutralizing agent employed during the process for preparing a detergent slurry generally include strong alkaline ingredients such as sodium or potassium hydroxide. These alkaline neutralizing agents are used as stoichiometrically required to ensure complete neutralization of the acid form of the anionic surfactant. After the in-situ neutralization of acid precursor of surfactant to form anionic surfactant salt, other alkaline salts, such as alkali metal silicates and alkali metal carbonates are also added to the detergent slurry. These alkali metal silicates and alkali metal carbonates act as builders and also add alkalinity and add to the buffering capacity in the laundry composition. Alkaline ingredients contribute towards good detergency. However, excess alkali in the laundry detergent composition has certain disadvantages. Highly alkaline laundry detergent compositions are harsh on the skin especially in composition designed for hand laundering.

30 **[0005]** Furthermore, when consumers launder fabrics by hand, they desire for a laundry composition which in addition to good cleaning performance also provides mildness to the skin and a desirable feel while washing. In the past, such milder compositions have been formulated by significantly reducing or completely removing the alkaline ingredients such as sodium carbonate and sodium silicate. It was found that the reduction or complete removal of these alkaline ingredients significantly impact cleaning performance and powder properties. Carbonate salts and silicate salts not only provide a wash liquor pH of about 10.5, but they also function as effective builders to sequester calcium and magnesium ions present in water. Besides formulation benefits, the carbonate and silicate salt also contribute to good physical properties to the powder such as free-flowability and good storage stability. The presence of carbonate salt provides good physical characteristics to the spray-dried detergent particle. Such physical characteristics include good shelf life with respect to caking of powders, and good flowability of the powder during extended shelf storage period. Silicate salt which is typically a sodium salt of silicate is generally considered a critical ingredient in spray-dried particulate detergent compositions and serves to provide stability and integrity to the detergent particles or agglomerated particles formed during the spray-drying operation. In the past, attempts at reduction or elimination of sodium silicate has resulted in spray-dried particle with deterioration of flow properties and an increase in caking tendency.

45 **[0006]** It is thus a challenge to incorporate lowered amounts of alkaline builders, particularly alkali metal silicates and alkali metal carbonates, while providing compositions which are milder on hand without adversely affecting the cleaning performance and the powder properties such as free-flowability and extended storage life of spray-dried detergent particle or granular laundry detergent composition including these spray-dried detergent particle.

50 **[0007]** Another concern while preparing a spray-dried detergent particle by a slurry making process is that the raw materials such as linear alkyl benzene sulphonic acid (also referred as LAS acid or HLAS) and sodium hydroxide have varying degree of purity.

55 **[0008]** Depending on the level of purity, a certain amount of unreacted LAS acid or unreacted NaOH may still remain in the fully neutralized solution. Although the unreacted LAS acid will eventually get neutralized with other alkali sources added to the final laundry detergent composition, the unreacted sodium hydroxide continues to remain in the spray-dried particle and the laundry detergent composition to which it is added. The presence of such unreacted sodium hydroxide is found to be a major reason for harshness on skin experienced by the consumer. This problem gets even pronounced when the LAS acid added to the aqueous solution has a lower purity while the sodium hydroxide has a higher purity. It is thus required to address the problem of presence of unreacted sodium hydroxide in the spray-dried particle irrespective of the purity of LAS acid or sodium hydroxide added during the neutralization process.

[0009] WO 98/11193 (Unilever) discloses a process for preparing a granular detergent composition in which an acid

precursor of anionic surfactant is partially neutralized followed by fully neutralizing in a high-speed mixer. The partial neutralization is by using alkali hydroxide followed by complete neutralization with using a weak alkaline material.

[0010] More recently, EP 3146034 B1 (P&G, 2019) discloses a two-stage neutralization process to form detergent granules with improved surfactant activity and good particulate flowability. The process includes a first step of neutralizing 5% to 20% of the acid precursor of the surfactant with a neutralizing agent and then neutralizing the remaining acid precursor with a second neutralizing agent. This discloses an agglomeration process and a dry-neutralization which is different from a spray-drying process for preparing a solid detergent particle.

[0011] Further, EP2870229 B1 (Unilever) discloses a process for preparing a detergent granule having at least 50 wt.% anionic surfactant which includes the step of (i) mixing an anionic surfactant precursor with a source of alkali to form a neutralized paste (ii) adding sodium citrate, up to 5 wt.% alkaline silicate and sodium carbonate to form a slurry (iii) spray drying the slurry to form a detergent granule. This discloses a process in which the source of alkali in a single step process, fully neutralizes the anionic surfactant precursor.

[0012] Similarly, EP2841553 B1 (Unilever) discloses a process for preparing a detergent granule having at least 40 wt.% anionic surfactant which including the step of (i) neutralizing an anionic surfactant precursor with a source of alkali (ii) adding polycarboxylate polymer, sodium sulphate and sodium carbonate to form a slurry (iii) spray drying the slurry to form a detergent granule. This discloses a process in which a source of alkali fully neutralizes the anionic surfactant precursor, in a single step process.

[0013] US 2011/147962 A1 (Cardozo Larry Savio et. al.) discloses a process for preparing a spray-dried detergent powder having the step of forming an aqueous slurry, transferring the aqueous detergent slurry through a pump to a spray nozzle followed by contacting alkoxylated anionic deterative surfactant and then spraying to form a spray-dried powder. This again, discloses the neutralization of the anionic surfactant in a single step process.

[0014] Although two-stage dry neutralization process have been known to produce a detergent granule using a non-tower route, they have not been extensively attempted for preparing a slurry having at least 20 wt.% water for further processing using a spray-drying process to produce a spray-dried detergent particle.

[0015] Thus, there is a need to provide a spray-dried detergent particle and a granular laundry detergent composition including the spray-dried detergent particle which is less harsh on the hands of the user without adversely affecting the cleaning performance and the free-flowing powder properties.

[0016] It is yet another object of the present invention to provide a process for preparing a a spray-dried detergent particle having lower reserve alkalinity and which is less harsh on hands and provides good fabric care performance.

[0017] It is also an object of the present invention to provide a process for preparing a spray-dried detergent particle which provides for incorporating sodium carbonate and sodium silicate at levels which provides good cleaning performance and good powder properties. It is yet another object of the present invention to provide a process for preparing a a spray-dried particle having lower reserve alkalinity and a desired pH for providing good cleaning performance.

Summary of the invention

[0018] The present inventors have found a way to provide a free-flowing spray-dried detergent particle which is gentle on hand, gives good fabric care performance, good fabric cleaning performance and which also retains good powder properties by providing a process of preparing a detergent solution which yields lower reserve alkalinity in the spray-dried particle and has a pH preferably from 10.5 to 11.5 and preferably without impacting the current level of alkaline builders such as sodium carbonate and sodium silicate in the composition.

[0019] It is surprisingly found that one or more of the above mentioned benefits is achievable by a process for preparing a spray-dried detergent particle where a portion of the acid precursor of the anionic surfactant is neutralised first with a alkali metal hydroxide neutralizing agent, followed by further neutralizing the remaining portion of the acid precursor of the anionic surfactant with a silicate neutralizing agent, such that the amount of in-situ neutralized anionic surfactant salt formed by the neutralisation with alkali metal hydroxide neutralizing agent contributes from 10 parts to 98 parts of the total anionic surfactant content in the spray-dried detergent particle.

[0020] While reducing the amount of unreacted sodium hydroxide in the spray-dried detergent particle is required to address the issue of harshness on hands, the present inventors have found that there is a need for a careful balance between the amount of the LAS acid that is reacted with the first neutralizing agent and the second neutralizing agent to ensure that the fully neutralized solution has desired viscosity for proper mixing and spray drying. In the process of the present invention the selection of specific second neutralizing agent and the careful balance between the portion of acid precursor neutralized by the first and the second neutralizing agent gives the spray dried particle the desired mildness on skin while maintaining good powder properties and good cleaning performance.

[0021] Accordingly, in a first aspect the present invention discloses a process for preparing a a spray-dried detergent particle, wherein the process for preparing the spray-dried detergent particle involves the steps of:

- i) mixing an acid precursor of an anionic surfactant and an alkali metal hydroxide neutralizing agent in an aqueous

medium to form a partially neutralized solution, wherein the partially neutralised solution comprises an anionic surfactant salt formed in-situ by the reaction of a portion of acid anionic surfactant precursor with the alkali metal hydroxide neutralizing agent and said anionic surfactant salt forms 10 parts to 98 parts of the total in-situ anionic surfactant content by weight of the spray-dried detergent particle; and,

ii) contacting the partially neutralized solution with a silicate neutralizing agent, wherein the silicate neutralizing agent is provided in an amount sufficient for neutralizing the remaining part of the acid precursor of an anionic surfactant to form a fully neutralized detergent solution;

iii) adding laundry ingredients to the fully neutralized detergent solution to form an aqueous detergent slurry wherein the detergent slurry has a water content ranging from 25 wt.% to 40 wt.%;

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

[0022] Preferably the fully neutralized solution is mixed with one or more laundry ingredients selected from carbonate-based builder; additional surfactant, a carboxylate polymer, fillers selected from the group consisting of alkali metal sulphate, alkali metal chloride, alkaline earth metal carbonate, or mixtures thereof, more preferably the filler is selected from sodium chloride, sodium sulphate, magnesium carbonate, calcium carbonate, calcite mineral, calcium magnesium carbonate (dolomite) to form a detergent slurry;

[0023] As used herein the term "reserve alkalinity" refers to the grams of NaOH/100 grams when titrated till 10 pH with H_2SO_4 . The alkali reserve is determined by an acid/base titration of a solution with 0.5 mol/l sulphuric acid till pH of 10.00.

[0024] The reserve alkalinity of the spray-dried detergent particle to pH 10 is from 1.5 to 5 grams NaOH/100g. Preferably less than 5.0 grams NaOH/100g, preferably less than 4 grams NaOH/100g, still preferably 3.5 grams NaOH/100g, further preferably less than 2 grams NaOH/100g.

[0025] The term "absolute value" refers to the amount of Na LAS in the spray-dried detergent particle formed by neutralizing with the silicate neutralizing agent. For example, in a spray-dried detergent particle where the total NaLAS is 20 wt.% of which 5 wt.% is formed by silicate neutralizing agent, then 5 wt.% is the absolute value of NaLAS formed by neutralizing with silicate neutralizing agent and 15 wt.% is the absolute value of NaLAS neutralized by alkali metal neutralizing agent.

Detailed description of the invention

[0026] According to a first aspect of the present invention disclosed is a process for preparing a spray-dried detergent particle, said process for preparing the spray-dried detergent particle comprising the steps as described herein below.

Step (i): Forming a partially neutralized detergent solution

[0027] According to the first aspect of the present invention disclosed is a process for preparing a spray-dried detergent particle involving the step of mixing a liquid acid precursor of an anionic surfactant and an alkali metal hydroxide neutralizing agent to form a partially neutralized solution. The partially neutralised solution includes an anionic surfactant salt formed in-situ by the reaction of a portion of the acid anionic surfactant precursor with the alkali metal hydroxide neutralizing agent. The neutralized anionic surfactant salt formed in this step provides 10 parts to 98 parts of the total anionic surfactant content by weight of the spray-dried detergent particle.

Acid precursor of anionic surfactant:

[0028] Suitable liquid acid precursor of anionic surfactant are well known to those skilled in the art. Examples of suitable acid precursor of anionic surfactant includes alkyl and aryl sulphonic acids and derivatives thereof, preferably alkylbenzene sulphonic acid, particularly linear alkylbenzene sulphonic acid having an alkyl chain length of C_8 to C_{22} , preferably C_{10} to C_{20} , still preferably C_{11} to C_{15} ; primary and secondary alkyl sulphuric acid, particularly C_{12} to C_{15} primary alkyl sulphuric acid. Preferably, the liquid acid precursor of anionic surfactant is an alkyl benzene sulphonic acid, preferably linear alkylbenzene sulphonic acid (LAS acid or HLAS), more preferably having an alkyl chain length of C_8 to C_{20} , still preferably C_{10} to C_{20} . It is highly preferred that the liquid acid precursor of the anionic surfactant comprises an alkyl benzene sulphonic acid (linear or branched), preferably C_8 to C_{20} linear alkyl benzene sulphonic acid. Preferably the alkyl benzene sulphonic acid is linear or branched, substituted or unsubstituted, C_8 to C_{20} alkyl benzene sulphonate. Highly preferred C_8 to C_{20} alkyl benzene sulphonic are linear C_{10} to C_{13} alkylbenzene sulphonic acid. Especially preferred are linear C_{10} to C_{13} alkylbenzene sulphonic acid obtained, by sulphonating commercially available linear alkyl benzenes (LAB); suitable LAB include low 2-phenyl LAB, such as those supplied by Sasol under the tradename Isochem® or those supplied by Petresa under the tradename Petrelab®, other suitable LAB include high 2-phenyl LAB, such as those supplied by Sasol under the tradename Hyblene®. Preferably, the anionic surfactant precursor is LAS acid which yields

the corresponding linear alkyl benzene sulphonate (LAS) upon neutralisation.

[0029] Also included within the scope of the present invention are the acid precursors which includes alkyl ether sulphuric acid; olefin sulphonic acid (preferably C₈ to C₂₄; alkyl xylene sulphonic acid; alkane sulphonic acid, dialkyl sulphosuccinic acid; fatty acid ester sulphonic acid and mixtures thereof. Preferably the acid precursor of anionic surfactant is a non-soap anionic surfactant acid precursor.

[0030] Preferably the acid precursor of anionic surfactant has a purity of 85% to 99%, that is it comprises from 90% to 98% acid precursor of anionic surfactant, the remaining ingredients include liquid carrier (water or other inorganic solvent), sulphuric acid, non-detergent organic matter such as unreacted linear alkyl benzene. More preferably the acid precursor of anionic surfactant has a purity from 90% to 98%. Preferably the liquid acid precursor of anionic surfactant is aqueous.

[0031] The acid precursor of the anionic surfactant is preferably provided in a liquid form that is pumpable and suitable for a spray-drying process. Specifically, such acid precursor of anionic surfactant is characterized by a viscosity ranging from about 0.1 Pa.S to about 10 Pa.S, preferably from about 0.1 Pa.S to about 5 Pa.S, more preferably from about 0.2 Pa.S to about 0.5 Pa.S, when measured at 50°C and at a shear rate of 25s⁻¹. Preferably the amount of acid precursor of anionic surfactant present in step (i) is sufficient to provide a spray-dried detergent particle having from 5 wt.% to 50 wt.% of the in-situ formed salt of the anionic surfactant, still preferably from 7 wt.% to 50 wt.%, and most preferably from 10 wt.% to 50 wt.% in-situ formed anionic surfactant by weight of the spray dried detergent particle.

Alkali metal hydroxide neutralizing agent:

[0032] The alkali metal hydroxide neutralizing agent employed during the first neutralization step in accordance with the process of the present invention for partially neutralizing the liquid acid precursor of the anionic surfactant is preferably selected from sodium hydroxide or potassium hydroxide, most preferably the alkali metal hydroxide neutralizing agent according to the present invention is sodium hydroxide.

[0033] Typically, the sodium hydroxide is available in the form of an aqueous solution. Moreover, the reaction of an alkali metal hydroxide and acid precursor also yields some water as a by-product. Preferably, the aqueous sodium hydroxide solution has a concentration in the range from 40 wt.% to 50 wt.%. Preferably the sodium hydroxide has a concentration of at least a 30 wt.% in the aqueous solution. In some cases, anhydrous sodium hydroxide flakes may be used but with extensive safety precautions from chemical spillages or exothermic reactions.

[0034] The mixing of a liquid acid precursor of an anionic surfactant and the alkali metal hydroxide neutralizing agent may be carried out in any suitable mixer. This is typically done in a crutcher mixer with an efficient solid to liquid mixing profile. The most preferred order of addition for this process involves adding water and the alkali metal hydroxide with continuous agitation. The temperature of this solution can be increased optionally by direct or indirect steam heating. This is followed by addition of the liquid acid precursor of the anionic surfactant to the crutcher mixer, with a controlled rate of addition while the reaction mass is under continuous agitation. Preferably it is required to maintain the temperature of the reaction mix in such a manner so as to prevent overflow or boil out of the reaction mass in the crutcher. The temperature of the partially neutralized solution formed in step (i) is typically maintained above 50°C, preferably above 60°C and most preferably above 70°C, but preferably below 95 °C and most preferably below 85 °C. It can be preferred that the temperature of the partially neutralized solution so formed is carefully monitored and controlled by the degree of partial neutralization, either alone or in combination with additional heating and cooling means if necessary. The total reaction time for the partial neutralization of the liquid acid precursor of anionic surfactant in step (i) can range from 1 minute to 10 minutes preferably depending on the anionic active content present in the detergent solution. In some cases the reaction time can be higher and will not have any impact on the process. Preferably the mixing time of the acid precursor of anionic surfactant and the alkali metal hydroxide neutralizing agent to attain the neutralization is from 1 to 5 minutes, preferably 1 to 3 minutes, still preferably for 1 to 2 minutes. Typically, the neutralization time is less than 5 minutes, preferably less than 3 minutes and still preferably less than 2 minutes.

[0035] The alkali metal hydroxide neutralizing agent added is in an amount sufficient to react with a portion of the acid precursor of anionic surfactant which yields from 10 parts to 98 parts of the total in-situ formed anionic surfactant present in the spray-dried detergent particle, more preferably from 21 parts to 98 parts of the total in-situ formed anionic surfactant present in the spray dried detergent particle. More preferably the alkali metal hydroxide neutralizing agent added is in an amount sufficient to react with a portion of the acid precursor of anionic surfactant which yields at least 21 parts, still preferably at least 22 parts, still preferably at least 23 parts, most preferably at least 24 parts of the total in-situ formed anionic surfactant present in the spray-dried detergent particle, but typically not more than 95 parts, still preferably not more than 90 parts, still further preferably not more than 85 parts, still more preferably not more than 80 parts and most preferably not more than 50 parts of the total in-situ formed anionic surfactant present in the spray-dried detergent particle.

[0036] The process according to the first aspect of the present invention forms a partially neutralized solution in step (i), wherein the partially neutralized solution comprises:

- i) a salt of anionic surfactant (formed by the reaction between a portion of the acid precursor of the anionic surfactant and the alkali metal hydroxide);
- ii) the remaining part of the liquid acid precursor of anionic surfactant; and,
- iii) at least 35 wt. % water.

[0037] In effect, the partially neutralized detergent solution is then an aqueous mixture of the surfactant acid (acid precursor of the anionic surfactant) and neutralised salt form of the anionic surfactant.

Step ii: Forming the fully neutralized detergent solution

[0038] According to the first aspect of the present invention disclosed is a process for preparing a spray dried detergent particle involving the step (ii) of contacting the partially neutralized solution with a silicate neutralizing agent. In the process, the silicate neutralizing agent is provided in an amount sufficient for neutralizing the remaining part of the liquid acid precursor of an anionic surfactant to form a fully neutralized detergent solution.

[0039] Preferably the silicate neutralizing agent is provided in a stoichiometrically excess amount in relation to the remaining part of the liquid acid precursor of an anionic surfactant to form a fully neutralized detergent solution. More preferably the ratio of the silicate neutralizing agent to the anionic surfactant salt formed from the neutralization of the remaining part of the liquid acid precursor in the spray dried detergent particle is from 5 to 30.

[0040] Preferably the amount of silicate neutralizing agent is in excess of the amount of anionic surfactant salt formed in the spray-dried particle from the remaining liquid acid precursor surfactant available in the partially neutralized solution. Preferably the amount of the liquid acid precursor of anionic surfactant remaining in the partially neutralized solution is in an amount sufficient to provide from 1 wt. % to 7 wt. % of the total in-situ anionic surfactant salt content (absolute value) present in the spray-dried detergent particle.

[0041] Preferably the anionic surfactant salt formed by the neutralization of the remaining part of the acid precursor of an anionic surfactant with the silicate neutralizing agent comprises from 1 wt. % to 7 wt. % of the total anionic surfactant salt present in the spray-dried detergent particle (absolute value), still preferably from 1 wt. % to 6 wt. % and still further preferably from 1 wt. % to 5 wt. % of the total anionic surfactant salt (in absolute value) present in the spray-dried detergent particle.

[0042] The mixing of the partially neutralized solution and silicate neutralizing agent may be carried out in any suitable mixer as described in detail hereinabove in the discussion for mixing liquid acid precursor of anionic surfactant and the alkali metal hydroxide neutralizing agent in step (i).

[0043] Silicate neutralizing agent is employed during the second neutralization stage according to the process of the first aspect of present invention, to neutralize the remaining liquid acid anionic surfactant precursor to form the fully neutralizing detergent solution. The silicate neutralizing agent can be any silicate alkali component that is capable of reacting in-situ with the acid precursor of the anionic surfactant to form the anionic surfactant in an appropriate salt form. Non-limiting examples of the silicate neutralizing agent includes alkali metal silicates, alkaline earth metal silicates or mixtures thereof. Preferably alkali metal silicate. Preferably the silicate may be selected from sodium silicate, potassium silicate, sodium-potassium double silicate, lithium silicate or mixtures thereof are particularly preferred. More preferably silicate neutralizing agent is sodium silicate. The silicate neutralizing agent may be liquid or solid form, preferably they may be crystalline silicates or soluble amorphous silicates.

[0044] The silicate neutralizing agent is preferably a liquid silicate neutralizing agent having an approximate ratio of alkali oxide to silicon dioxide (M_2O to SiO_2) of 1:1.6 to 1:3.3, preferably 1:1.6 to 1:2.85, still preferably from 1:2 to 1:2.85 and most preferably 1:2 to 1:2.7. Orthosilicate, having the formula M_4SiO_4 , is the most alkaline having a M_2O to SiO_2 ratio of 2:1. Metasilicate, M_2SiO_3 , has a M_2O to SiO_2 ratio of 1:1. The so called "water glass" silicates, which are soluble in water, have a M_2O to SiO_2 ratio of 1:1.6 to 1:3.3, preferably silicate with a M_2O to SiO_2 ratio of from 1:2.0 to 1:2.7 where M is an alkaline earth metal, alkali metal, preferably alkali metal. Most preferably the M is sodium (Na).

[0045] Preferably the reaction time involving the mixing of the partially neutralized solution and the silicate neutralizing agent is from 1 to 5 minutes, preferably 1 to 3 minutes, still preferably for 1 to 2 minutes to obtain the fully neutralized detergent solution.

[0046] The process according to the first aspect of the present invention forms a fully neutralized detergent solution after step (ii), wherein the fully neutralized detergent solution comprises:

- i) in-situ formed salt of anionic surfactant;
- ii) at least 40 wt. % water; and,
- iii) silicate salt.

[0047] Preferably the fully neutralized detergent solution is an aqueous solution having at least 40 wt. % water, still preferably having at least 45 wt. % water, still preferably having at least 50 wt. % water and typically from 40 wt. % to

65 wt.% water..

[0048] Preferably the pH of the fully neutralized detergent solution when measured using a 1 wt.% solution in distilled water at 25°C ranges from 10.5 to 11.5, preferably ranging from 10.8 to 11.2.

5 Further steps in the process for preparing a spray-dried detergent particle

[0049] After the full neutralization of the acid anionic surfactant precursor, the fully neutralized detergent solution may be spray dried to form the spray dried particle. Alternately preferred laundry ingredients which are thermally stable may be added to the fully neutralized detergent solution to form an aqueous detergent slurry before spray-drying. The term aqueous detergent slurry used herein refers to the aqueous mixture obtained by adding the below described laundry ingredients such as builders, filler, optional ingredients to the fully neutralized detergent solution.

Builder:

[0050] Advantageously the laundry ingredient may be a builder which may be added post full neutralization of the acid anionic precursor. The builders may be preferably selected from precipitation builder, complexing builder, sequestering builders, ion-exchange builder or mixtures thereof. Preferably the sequestering builder materials added to the fully neutralized detergent solution before spray drying include inorganic sequestering material and/or organic sequestering material. Example of the inorganic sequestering material include alkali metal polyphosphates, such as sodium tripolyphosphate. Example of the organic sequestering material include ethylene diamine tetra-acetic acid.

[0051] Examples of precipitating builder materials include sodium carbonate. The preferred complexing builder includes diethylenetriamine-pentaacetic acid, alkyl- or alkenylsuccinic acid, nitrilotriacetic acid as suitable examples. Examples of ion-exchange builder materials include the various types of water-insoluble crystalline or amorphous aluminosilicates, of which zeolites are the best known representatives, e.g. zeolite A, zeolite B (also known as zeolite P), zeolite C, zeolite X, zeolite Y and also the zeolite P-type as described in EP-A-0,384,070.

[0052] Preferably the builder is a carbonate salt builder. Examples of the carbonate salt builder includes alkaline earth metal and alkali metal carbonates or mixtures thereof. The carbonate salt is preferably an alkali metal carbonate, alkaline earth metal carbonate or mixtures thereof. Preferred alkali metal carbonates are sodium and/or potassium carbonate of which sodium carbonate is particularly preferred. 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 salt.

[0053] Optionally the aqueous detergent 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.

[0054] 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.%.

[0055] Preferably the aqueous detergent slurry includes an acrylic maleic copolymer. 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 acrylic-maleic copolymer, suitably present in an amount of from 1 wt.% to 5 wt.%.

[0056] Inorganic phosphate 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.%. 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. Most preferably the slurry includes 0 wt.% inorganic phosphate builder. Preferably the spray-dried detergent particle includes 0 wt.% inorganic phosphate builder.

[0057] Similarly, the 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.

Filler:

[0058] Advantageously the laundry ingredient may be a filler, the filler may be added to the fully neutralized detergent solution to form an aqueous slurry. Preferably a filler may be added to the aqueous detergent slurry before or after the carbonate builder addition, preferably before the carbonate builder addition but before spray-drying. The filler acts as a balancing ingredient and can be a neutral inorganic salt, mineral or mixtures thereof. Preferably the filler is selected from the group consisting of alkali metal sulphate, alkali metal chloride, alkaline earth metal carbonate or mixtures thereof. Still preferably the fillers are selected from the group consisting of sodium sulphate, magnesium sulphate, calcium magnesium carbonate (dolomite), calcite, sodium chloride or calcium carbonate, magnesium carbonate or mixtures thereof. Preferably the filler is sodium sulphate, sodium chloride, calcium carbonate (calcite) or mixtures thereof. More preferably the filler may be preferably sulphate, carbonate salt, sodium chloride or a mixture thereof.

Pre-neutralized surfactant:

[0059] Other pre-neutralized surfactant may be preferably added to the fully neutralized detergent solution these include but are not limited to LAS salt, primary alkyl sulphate (PAS), secondary alkyl sulphate (SAS), sodium lauryl ether sulphate (SLES) or combinations thereof. Preferably the pre-neutralized surfactant is added as a co-surfactant and preferably is present in an amount ranging from 0 wt.% to 20 wt.% by weight of the spray dried particle. Preferably the co-surfactant is PAS, SLES or combinations thereof.

Optional ingredients:

[0060] Further optional ingredients may also be advantageously added to the aqueous detergent 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, dyes, anti-redeposition agents such as sodium carboxy methyl cellulose, slurry stabilizers such as homopolymers of acrylic acid and copolymers of acrylic acid and maleic acid; ethylene and maleic anhydride, and of vinyl methyl ether and maleic anhydride, usually in salt form; viscosity modifiers, hydrotrope, defoaming agents, antioxidants, colourants, shading dyes and combinations thereof.

[0061] Aqueous detergent slurry is obtained by adding laundry ingredients preferably carbonate salt builder and/or filler to the fully neutralized detergent solution, and preferably the aqueous detergent slurry comprises:

- i) in-situ formed salt of anionic surfactant;
- ii) sodium carbonate;
- iii) silicate salt;
- iv) filler;
- v) preferably a pre-neutralized co-surfactant selected preferably from PAS, SLES or mixtures thereof; and,
- vi) water.

[0062] The aqueous detergent slurry has a water content from 25 wt.% to 40 wt.%, preferably 27 wt.% to 40 wt.%, still preferably 30 wt.% to 35 wt.%.

[0063] Preferably the aqueous detergent slurry has a pH ranging from 10.5 to 11.5, when measured using a 1 wt.% solution in distilled water at 25°C. Preferably a pH ranging from 10.8 to 11.2.

[0064] The reserve alkalinity of the aqueous detergent slurry/spray-dried detergent particle is 3% to 20% lower than that of a similar formulation made with the conventional process i.e., a single step neutralisation with a strong alkali metal hydroxide, NaOH. More preferably the reserve alkalinity of the spray-dried detergent particle prepared in accordance with the present invention has a reserve alkalinity which is at least 3 % lower than the reserve alkalinity of spray-dried detergent particle prepared using a single step neutralization using sodium hydroxide as the neutralizing agent (herein referred to as the conventional process), still preferably lower by at least 4%, still further preferably lower by at least 5%, still further preferably lower by at least 8% but preferably lower by not more than 19%, still preferably lower by not more than 18%, still further preferably lower by not more than 15% as compared to the reserve alkalinity of a spray-dried detergent particle prepared using a single step neutralization using sodium hydroxide as the neutralizing agent (herein referred to as the conventional process).

[0065] Preferably the viscosity of the aqueous detergent slurry is from 1 Pa.s to 20 Pa.s (1000 centipoise to 20,000 centipoise) at a temperature of 65°C to 85°C or even somewhat higher at a shear rate of 17 to 18 sec⁻¹. The aqueous detergent slurry is sufficiently fluid to pump it out of the mixing vessel to a spray tower. The viscosity of the slurry may preferably be adjusted by manipulating the slurry moisture content or by addition of an organic or inorganic additive. Non-limiting examples of the viscosity adjusters are nonionic surfactant, hydrotropes, polyethylene glycol, polypropylene glycol, and inorganic salts.

Spray-dried detergent particle

[0066] The aqueous detergent slurry obtained by addition of other laundry ingredients is spray-dried to form a spray-dried detergent particle.

[0067] 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.

[0068] The pump system includes one or more pump, the pump is preferably a high-pressure pump. In a preferred embodiment, the pump system includes a first pump which is typically a low-pressure pump, such as a pump that is capable of generating a pressure of from 1 to 10 bar (1×10^5 to 1×10^6 Pa), this pressure ensures proper flooding of the second pump. Typically, the second pump is a high-pressure pump, such as a pump that is capable of generating a pressure ranging from 20 bar to 200 bar (2×10^6 Pa to 2×10^7 Pa). Optionally, the aqueous detergent slurry is 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. Preferably disintegrator is positioned between the pumps. The flow rate of the aqueous detergent slurry along the pipes is typically in the range from 800 kg/hour to more than 75,000 kg/hour.

[0069] 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.

[0070] 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.

[0071] 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.

[0072] 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 150°C to 500°C depending on the evaporation capacity and sizing of the tower. Preferably the tower exhaust air temperature can range from, 50°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. Preferably the spray-dried powder exiting the spray-drying tower has a temperature of less than 150°C, and still preferably less than to 100°C.

[0073] Preferably, the spray-drying zone is under a slight vacuum. Preferably the vacuum is controlled by the speed and/or damper setting of the inlet and outlet air fans. The negative pressure in the spray- drying tower can be measured by any available means. Typically pressure sensors are present in the spray-drying zone (inside the spray-drying tower). Preferably the vacuum pressure in the spray drying zone ranges from -10Nm^{-2} to -600Nm^{-2} , preferably -10Nm^{-2} to -300Nm^{-2} to improve the cooling of the spray-dried particle. Typically, the maximum pressure one can use is determined by the structural strength of the spray-drying tower and care must be taken not to exceed this maximum vacuum so that no undue stress is placed on the spray- drying tower.

[0074] The spray-dried detergent particle collected at the bottom of the tower may be subjected to cooling and conditioning by using an air lift or any similar process. Preferably a flow aid such as zeolite is added to the spray-dried detergent particle before air-lifting. Preferably, the spray-dried detergent particle is subjected 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.

[0075] The spray-dried detergent particle may itself be used as fully formulated laundry composition. Typically, further laundry ingredients are dry-added to the spray-dried detergent particle to form a fully formulated laundry detergent composition, this dry addition of further laundry ingredient to the spray-dried particle (generally referred as base powder) is generally termed as post-dosing.

[0076] According to a second aspect of the present invention disclosed is a spray-dried detergent particle obtainable by a process of the first aspect, wherein the spray-dried particle has a reserve alkalinity at pH 10 ranging from 1.5 to 5 grams NaOH/100g.

[0077] Preferably the spray-dried detergent particle according to the second aspect of the present invention includes:

- i) from 5 wt.% to 50 wt.% in-situ neutralized anionic surfactant;
- ii) optionally from 0 wt.% to 20 wt.% co-surfactant, preferably selected from PAS, SLES or mixtures thereof;

- iii) from 8 wt.% to 20 wt.% silicate salt, preferably with a $\text{SiO}_2/\text{Na}_2\text{O}$ weight ratio from 1:1.6 to 3.3,
- iv) preferably from 8 wt.% to 70 wt.% carbonate salt;
- v) optionally, from 2 wt.% to 10 wt.% magnesium sulphate;
- vi) optionally, from 0.5 wt.% to 5 wt.% polymer;
- vii) optionally, from 1 wt.% to 65 wt.% filler, preferably selected from sodium sulphate, sodium carbonate, sodium chloride, alkaline earth metal carbonate or mixtures thereof; and
- viii) optionally, from 0 wt.% to 6 wt.% water.

[0078] Preferably the amount of water present in the spray dried particle is from 0 wt.% to 4.5 wt.%, preferably 0 wt.% to 4 wt.%. The spray-dried detergent particle formed by spray drying the fully neutralized detergent solution or the aqueous slurry preferably comprises from 7 wt.% to 50 wt.% in-situ formed anionic surfactant salt.

[0079] Preferably the spray-dried detergent particle has a pH ranging from 10.5 to 11.5 when measured using a 1 wt.% solution with distilled water at room temperature. Preferably the spray-dried detergent particle has a reserve alkalinity which is 3% to 20% lower than that of a similar formulation made with the conventional process i.e., a single step neutralisation with a strong alkali metal hydroxide, NaOH.

[0080] 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 micrometers to 600 micrometers.

[0081] The spray-dried detergent particle preferably comprises from 7 wt.% to 50 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.

[0082] 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.

[0083] Preferably the composition of the spray-dried detergent particle includes from 8 wt.% to 20 wt.% alkali metal silicate builder, preferably from 8 wt.% to 70 wt.% alkali metal carbonate builder, still preferably from 8 wt.% to 30 wt.% alkali metal carbonate builder. In addition to this the spray dried detergent particle may further include cellulose derivatives, acetates, polyacrylates, acrylate-maleate copolymers, magnesium sulfate and mixtures thereof.

Laundry detergent composition

[0084] The spray-dried detergent particle may be used as a fully formulated laundry detergent composition or may be additionally combined with other optional benefit 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.

[0085] 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:

[0086] Suitable fluorescent brighteners include dis-styryl biphenyl compounds example Tinopal[®] CBS-X, di-amino stilbene di-sulfonic acid compounds, e.g. Tinopal[®] DMS pure Xtra and Blankophor[®] HRH, and Pyrazoline compounds, e.g. Blankophor[®] SN, and coumarin compounds, e.g. Tinopal[®] SWN. Preferred brighteners are: sodium 2 (4-styryl)-3-sulfophenyl)-2H-naphthol(1,2-d]triazole, disodium 4,4'-bis{[(4-anilino-6-(N methyl-N-2 hydroxyethyl)amino 1,3,5- triazin-2-yl)]amino}stilbene-2-2' disulfonate, disodium 4,4'-bis{[(4-anilino-6-morpholino-1,3,5-triazin-2-yl)]amino} stilbene-2-2'disulfonate, and disodium 4,4'- bis(2-sulfostyryl)biphenyl. A suitable fluorescent brightener is S C.I. Fluorescent Brightener 260, which may be used in its beta or alpha crystalline forms, or a mixture of these forms.

Enzymes:

[0087] 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.

[0088] 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, lipoxxygenases, ligninases, pullulanases, tannases, pentosanases, mannanases, G-glucanases, arabinosidases, hyaluronidase, chondroitinase, laccase, and amylases, or mixtures thereof. A typical combination is an enzyme combination that includes 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

[0089] 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.

[0090] 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 deterative surfactants (as are further defined above).

EXAMPLES

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

[0091] A batch of 1 tonne of spray-dried detergent particle was prepared using the ingredients as provided in Table 1. To prepare the spray-dried detergent particle according to the present invention, initially an aqueous detergent slurry (Ex 1) was prepared by adding required amount of water and caustic acid (NaOH, alkali metal hydroxide neutralizing agent) to the crutcher mixer. Next LAS acid (liquid acid precursor of anionic surfactant) was added and homogeneously mixed. After 2 minutes of mixing a portion of LAS acid (which formed 75 parts of the total anionic surfactant present in the spray dried detergent particle) was neutralized to form the salt form (NaLAS). Thereafter, required excess amount sodium silicate was added to neutralize the remainder of the LAS acid (this constitutes 5 wt.% NaLAS present in the spray dried detergent particle shown on table 2, the rest of the 15 wt.% Na LAS is formed from the first neutralizing step). The mixing was continued for another 2 to 5 minutes to ensure the LAS acid is fully neutralized to form the fully neutralized detergent solution. Next, to this solution having fully neutralized LAS (in the form of NaLAS) acrylic acid- maleic acid copolymer was added followed by the addition of laundry ingredients which included sodium sulphate (filler, balancing ingredient) and then sodium carbonate, fluorescers and antiredeposition agent (SCMC) were added to the solution to form the aqueous detergent slurry having a water content of 29 wt.%. A comparative aqueous slurry (Ex A) was prepared according to the conventional method of neutralization which employs only caustic acid as shown in Table 1 below. The process involved in the preparation of the comparative aqueous detergent slurry was similar to the preparation of the aqueous detergent slurry according to the present invention, with the difference that the LAS acid was neutralized only with NaOH and the sodium silicate was added after the fully neutralization of the LAS acid.

Table 1

Ingredients	Ex A (in Kg)	Ex 1 (in Kg)
Water	201.8	213.3
NaOH	73.4	55.0
LAS Acid	214.1	213.8
Sodium silicate	274.5	274.1
Polymer	3.9	3.9

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(continued)

Ingredients	Ex A (in Kg)	Ex 1 (in Kg)
Sodium sulphate	450.3	458.3
Flourosccers	0.3	0.3
Antiredeposition agent	4.2	4.2
Sodium carbonate	157.0	157.3
Total	1000	1000
Reserve alkalinity	2.2	1.8

[0092] Next the aqueous detergent slurries (Ex 1, Ex A) were each spray dried in a counter-current spray drying tower. The aqueous detergent slurry is maintained at a temperature of 80°C and pumped under a pressure of $3 \times 10^6 \text{ Nm}^{-2}$, into a counter current spray-drying tower with an air inlet temperature of around 300°C.

[0093] The specific conditions maintained in the spray-drying tower is as follows:

Parameter	Setting
Total air flow rate during the spray drying process, when measured at ambient temperature and atmospheric pressure.	10,000 m ³ /hr
Negative pressure in the tower	49 Nm ⁻²
Slurry flow rate	1160 Kg hr ⁻¹
Tower outlet temperature	150°C

[0094] The aqueous slurry is atomised, and the atomised slurry is dried to produce a solid mixture, which is then cooled and sieved to remove oversize material (> 2mm) to form a spray- dried free-flowing particle in the form of a powder. The spray-dried (powder) detergent particle has a moisture content of 2 wt.%, a bulk density of 375g/L and a particle size distribution such that greater than 90 wt.% of the spray-dried detergent particle (powder) has a particle size such that greater than 1.4 mm is around 3 wt.% and less than 180 micrometers is less than 15 wt.%.

Measurement of pH

[0095] pH was measured by preparing 1% aqueous solution of the spray dried particle. 20 grams of the spray-dried detergent particle (W) was dissolved in 1980 mL of distilled water and stirred for 30 minutes. Measure and record the pH of the solution.

Measurement of Reserve alkalinity:

[0096] The spray dried detergent particle of Ex A and Ex 1 were prepared using same method of slurry making and spray-drying but differed in that in the process of preparing the spray dried particle of Ex A the entire acid precursor of the anionic surfactant was neutralized using only NaOH as the neutralization agent and in the spray-dried detergent particle of Ex 1 the acid precursor of the anionic surfactant was prepared using a first neutralization step using NaOH followed by neutralization with sodium silicate.

[0097] The two spray-dried detergent particle were then analysed for their reserve alkalinity using the following method.

[0098] The reserve alkalinity was determined by an acid titration of a 1% solution of the spray-dried detergent particle with 0.5 mol/l sulphuric acid till pH of 10.00. First a 1% aqueous solution of the spray dried particle was prepared by dissolving 20 grams of the spray-dried detergent particle (W) in 1980 mL of distilled water. The prepared aqueous solution (2000 mL) was then titrated with 0.5 mol/l sulphuric acid (t) till a pH of 10 was reached. The amount of titrant (V) was noted.

[0099] The Reserve alkalinity (amount of NaOH per 100 grams) of the spray-dried detergent particle was then calculated using the formula:

$$\text{Amount of grammes of NaOH per 100 gram sample} = \frac{2 * V * t * 40.00 * 100}{W * 1000} = \frac{V * t * 8}{W}$$

[0100] Irritancy index: The irritancy index indicates degree of harshness of the spray-dried detergent particle on the skin. A lower irritancy index value indicates that the formulation is milder on skin, while higher value indicate that the formulation is harsher on the skin. The irritancy index is determined using the formula: $\text{pH} + (\text{Reserve alkalinity})/6$.

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.

[0101] The composition of the spray-dried detergent particle obtained by spray-drying the aqueous detergent slurry is given below.

Table 2

Ingredients	Spray-dried detergent particle	
	Ex A (wt%)	Ex 1 (wt%)
Na LAS	20	20
Sodium silicate	12	12
Polymer	0.15	0.15
Sodium sulphate	48.80	48.80
Sodium carbonate	15	15
Flourosccers	0.03	0.03
Antiredeposition agent	0.2	0.2
Moisture content	3	3
Impurities (NDOM and other salt)	0.8	0.8
Total	100	100
parts of the total NaLAS in the spray dried particle formed in first neutralization step (NaOH)	100	75
Absolute value of NaLAS by silicate neutralizing agent	0	5
Parameters		
Reserve alkalinity*	4.5	3.9
pH*	11.2	11.2
Irritancy index	11.95	11.85
Compression test value (in Kg) (after 8 weeks of storage under 45°C and 85 RH)	0.6	0.2
* measured by preparing a 1 wt.% solution of the spray-dried detergent particle in distilled water at room temperature.		

[0102] The data on table 2 shows that the spray-dried detergent particle obtained from a process where the liquid acid detergent precursor is neutralized according to the present invention by employing alkali metal hydroxide neutralizing agent followed by silicate neutralizing agent provides the spray-dried detergent particle (Ex 1) having lowered reserve alkalinity (13.3% lesser reserve alkalinity as compared to the conventional powder of Ex A) whilst maintaining similar pH as that of the conventionally prepared spray dried particle (Ex A, fully neutralized using NaOH), thus providing the

spray-dried particle according to the present invention with lowered harshness on hand and/or fabric while maintaining good cleaning performance and powder properties (as shown by a lowered tendency to caking).

5 Claims

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

10 i) mixing an acid precursor of an anionic surfactant and an alkali metal hydroxide neutralizing agent in an aqueous medium to form a partially neutralized solution, wherein the partially neutralised solution comprises an anionic surfactant salt formed in-situ by the reaction of a portion of acid anionic surfactant precursor with the alkali metal hydroxide neutralizing agent and said anionic surfactant salt forms 10 parts to 98 parts of the total anionic surfactant content by weight of the spray-dried detergent particle; and,

15 ii) contacting the partially neutralized solution with a silicate neutralizing agent, wherein the silicate neutralizing agent is provided in an amount sufficient for neutralizing the remaining part of the acid precursor of an anionic surfactant to form a fully neutralized detergent solution,

iii) adding laundry ingredient to the fully neutralized detergent solution to form an aqueous detergent slurry wherein the detergent slurry has a water content ranging from 25 wt.% to 40 wt.%;

20 iv) spray-drying the aqueous detergent slurry to form a spray-dried detergent particle.

2. A process according to claim 1 wherein the anionic surfactant salt formed by the neutralization of the remaining part of the acid precursor of an anionic surfactant with the silicate neutralizing agent comprises from 1 wt.% to 7 wt.% anionic surfactant salt in absolute value by weight of the spray dried detergent particle.

25 3. A process according to claim 1 or 2 wherein the silicate neutralizing agent is provided in a stoichiometrically excess amount in relation to the remaining part of the liquid acid precursor of an anionic surfactant to form the fully neutralized detergent solution.

30 4. A process according to claim 1 to 3 wherein the ratio of silicate neutralizing agent to the anionic surfactant salt formed from by the neutralization of the remaining part of the liquid acid precursor in the spray dried particle is from 5 to 30.

35 5. A process according to any one of the preceding claims wherein the silicate neutralizing agent is preferably an alkali metal or alkaline earth metal salt of silicate, more preferably sodium silicate.

6. A process according to any one of the preceding claims wherein the silicate neutralizing agent has a molar ratio of $M_2O: SiO_2$ from 1:1.6 to 1:3.3, preferably 1:1.6 to 1:2.4, still preferably 1:2 to 1:2.85 wherein M is an alkali metal.

40 7. A process according to any one of the preceding claims wherein the liquid acid precursor of an anionic surfactant is an alkyl benzene sulphonic acid, preferably C_{10} to C_{20} linear alkyl benzene sulphonic acid.

45 8. A process according to any one of the preceding claims wherein prior to spray-drying to form a spray-dried particle, the fully neutralized detergent solution is converted to an aqueous detergent slurry comprising the step of: mixing the fully neutralized detergent solution with one or more laundry ingredients selected from (i) carbonate builder salt; (ii) additional pre-neutralized co-surfactant, (iii) a carboxylate polymer, (iv) fillers selected from the group consisting of alkali metal sulphate, alkali metal chloride, alkaline earth metal carbonate or mixtures thereof, more preferably the filler is selected from sodium sulphate, sodium chloride, calcium carbonate, magnesium carbonate, dolomite, calcite or mixtures thereof

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

i) in-situ formed anionic surfactant salt;

ii) sodium carbonate;

55 iii) alkali metal silicate salt;

iv) filler selected from the group consisting of alkali metal sulphate, alkali metal chloride, alkaline earth metal carbonate or mixtures thereof, more preferably the filler is selected from sodium sulphate, sodium chloride, calcium carbonate, magnesium carbonate, dolomite, calcite or mixtures thereof;

v) 25 wt.% to 40 wt.% water.

10. A spray dried detergent particle obtainable by spray-drying an aqueous slurry according to any one of the preceding claims 1 to 9 wherein the spray-dried detergent particle has a reserve alkalinity at pH 10 ranging from 1.5 to 5 grams NaOH/100g, where reserve alkalinity refers to the grams of NaOH/100 grams when titrated till 10 pH with 0.5 mol/L H_2SO_4 .
11. A spray dried detergent particle according to claim 10 wherein the spray-dried detergent particle formed by spray drying the aqueous detergent slurry comprises from 7 wt.% to 50 wt.% in-situ formed anionic surfactant salt.
12. A spray dried detergent particle according to claim 10 or 11, wherein the spray-dried detergent particle comprises:
- i) from 7 wt.% to 50 wt.% in-situ formed anionic surfactant;
 - ii) optionally from 0 wt.% to 20 wt.% co-surfactant, preferably selected from PAS, SLES or mixtures thereof;
 - iii) from 8 wt.% to 20 wt.% silicate salt, preferably with a weight ratio of SiO_2 : M_2O from 1:6 to 1:3.3;
 - iv) preferably from 8 wt.% to 70 wt.% carbonate-based builder;
 - v) laundry ingredients preferably selected from (i) additional anionic surfactant salt, (ii) fillers selected from the group consisting of alkali metal sulphate, alkali metal chloride, alkaline earth metal carbonate or mixtures thereof, more preferably the filler is selected from sodium sulphate, sodium chloride, calcium carbonate (calcite), magnesium carbonate, calcium magnesium carbonate or combination thereof.

Patentansprüche

1. Verfahren zur Herstellung eines sprühgetrockneten Waschmittelpartikels, wobei das Verfahren zur Herstellung des sprühgetrockneten Waschmittelpartikels die Schritte umfasst:
- i) Mischen eines Säurevorläufers eines anionischen Tensids und eines Alkalimetallhydroxid-Neutralisierungsmittels in einem wässrigen Medium, um eine teilweise neutralisierte Lösung zu bilden, wobei die teilweise neutralisierte Lösung ein anionisches Tensidsalz umfasst, das in situ durch die Reaktion eines Teils des Säurevorläufers des anionischen Tensids mit dem Alkalimetallhydroxid-Neutralisierungsmittel gebildet wird, und das anionische Tensidsalz 10 bis 98 Teile des Gesamtgehalts an anionischem Tensid, bezogen auf das Gewicht des sprühgetrockneten Waschmittelpartikels, bildet, und
 - ii) Kontaktieren der teilweise neutralisierten Lösung mit einem Silikat-Neutralisierungsmittel, wobei das Silikat-Neutralisierungsmittel in einer Menge bereitgestellt wird, die ausreicht, um den verbleibenden Teil des Säurevorläufers eines anionischen Tensids zu neutralisieren, um eine vollständig neutralisierte Waschmittellösung zu bilden,
 - iii) Hinzufügen eines Waschmittelbestandteils zu der vollständig neutralisierten Waschmittellösung, um eine wässrige Waschmittelaufschlämmung zu bilden, wobei die Waschmittelaufschlämmung einen Wassergehalt in dem Bereich von 25 bis 40 Gew.-% aufweist;
 - iv) Sprühtrocknen der wässrigen Waschmittelaufschlämmung zur Bildung eines sprühgetrockneten Waschmittelpartikels.
2. Verfahren nach Anspruch 1, wobei das durch Neutralisieren des verbleibenden Teils des Säurevorläufers eines anionischen Tensids mit dem Silikat-Neutralisierungsmittel gebildete anionische Tensidsalz 1 bis 7 Gew.-% anionisches Tensidsalz in absolutem Wert, bezogen auf das Gewicht des sprühgetrockneten Waschmittelpartikels, umfasst.
3. Verfahren nach Anspruch 1 oder 2, wobei das Silikat-Neutralisierungsmittel in einer stöchiometrischen Überschussmenge im Verhältnis zum verbleibenden Teil des flüssigen Säurevorläufers eines anionischen Tensids bereitgestellt wird, um die vollständig neutralisierte Waschmittellösung zu bilden.
4. Verfahren nach den Ansprüchen 1 bis 3, wobei das Verhältnis des Silikat-Neutralisierungsmittels zu dem anionischen Tensidsalz, das durch Neutralisierung des verbleibenden Teils des flüssigen Säurevorläufers in dem sprühgetrockneten Partikel gebildet wird, 5 bis 30 beträgt.
5. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Silikat-Neutralisierungsmittel bevorzugt ein Alkalimetall- oder Erdalkalimetallsalz von Silikat, bevorzugter Natriumsilikat, ist.

6. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei das Silikat-Neutralisierungsmittel ein Molverhältnis von $M_2O:SiO_2$ von 1:1,6 bis 1:3,3, bevorzugt von 1:1,6 bis 1:2,4, noch bevorzugt von 1:2 bis 1:2,85 aufweist, wobei M ein Alkalimetall ist.

7. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei der flüssige Säurevorläufer eines anionischen Tensids eine Alkylbenzolsulfonsäure, bevorzugt eine lineare C_{10} - bis C_{20} -Alkylbenzolsulfonsäure, ist.

8. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei vor dem Sprühtrocknen zur Bildung eines sprühgetrockneten Partikels die vollständig neutralisierte Waschmittellösung in eine wässrige Waschmittelaufschlammung umgewandelt wird, umfassend den Schritt: Mischen der vollständig neutralisierten Waschmittellösung mit einem oder mehreren Waschmittelbestandteilen, ausgewählt unter (i) Carbonatbuildersalz; (ii) zusätzlichem vorneutralisierten Co-Tensid, (iii) einem Carboxylatpolymer, (iv) Füllstoffen, ausgewählt aus der Gruppe, bestehend aus Alkalimetallsulfat, Alkalimetallchlorid, Erdalkalimetallcarbonat oder Mischungen davon, wobei der Füllstoff bevorzugter unter Natriumsulfat, Natriumchlorid, Calciumcarbonat, Magnesiumcarbonat, Dolomit, Calcit oder Mischungen davon ausgewählt wird.

9. Verfahren nach irgendeinem der vorhergehenden Ansprüche, wobei die wässrige Waschmittelaufschlammung umfasst:

- i) in situ gebildetes anionisches Tensidsalz;
- ii) Natriumcarbonat;
- iii) Alkalimetallsilikatsalz;
- iv) Füllstoff, ausgewählt aus der Gruppe, bestehend aus Alkalimetallsulfat, Alkalimetallchlorid, Erdalkalimetallcarbonat oder Mischungen davon, wobei der Füllstoff bevorzugter unter Natriumsulfat, Natriumchlorid, Calciumcarbonat, Magnesiumcarbonat, Dolomit, Calcit oder Mischungen davon ausgewählt wird;
- v) 25 bis 40 Gew.-% Wasser.

10. Sprühgetrocknetes Waschmittelpartikel, erhältlich durch Sprühtrocknen einer wässrigen Aufschlammung nach irgendeinem der vorhergehenden Ansprüche 1 bis 9, wobei das sprühgetrocknete Waschmittelpartikel bei einem pH-Wert von 10 eine Reservealkalität in dem Bereich von 1,5 bis 5 Gramm NaOH/100 Gramm aufweist, wobei sich die Reservealkalität auf die Grammmenge von NaOH/100 Gramm bezieht, wenn mit 0,5 mol/l H_2SO_4 bis zu einem pH-Wert von 10 titriert wird.

11. Sprühgetrocknetes Waschmittelpartikel nach Anspruch 10, wobei das durch Sprühtrocknen der wässrigen Waschmittelaufschlammung gebildete sprühgetrocknete Waschmittelpartikel 7 bis 50 Gew.-% in situ gebildetes anionisches Tensidsalz umfasst.

12. Sprühgetrocknetes Waschmittelpartikel nach Anspruch 10 oder 11, wobei das sprühgetrocknete Waschmittelpartikel umfasst:

- i) 7 bis 50 Gew.-% in situ gebildetes anionisches Tensid;
- ii) optional 0 bis 20 Gew.-% Co-Tensid, vorzugsweise ausgewählt unter PAS, SLES oder Mischungen davon;
- iii) 8 bis 20 Gew.-% Silikatsalz, bevorzugt mit einem Gewichtsverhältnis von $SiO_2:M_2O$ von 1:6 bis 1:3,3;
- iv) bevorzugt 8 bis 70 Gew.-% Builder auf Carbonatbasis;
- v) Waschmittelbestandteile, bevorzugt ausgewählt unter (i) zusätzlichem anionischem Tensidsalz, (ii) Füllstoffen, ausgewählt aus der Gruppe, bestehend aus Alkalimetallsulfat, Alkalimetallchlorid, Erdalkalimetallcarbonat oder Mischungen davon, wobei der Füllstoff bevorzugter unter Natriumsulfat, Natriumchlorid, Calciumcarbonat (Calcit), Magnesiumcarbonat, Calciummagnesiumcarbonat oder einer Kombination davon ausgewählt ist.

Revendications

1. Procédé pour préparer une particule détergente séchée par pulvérisation, ledit procédé pour préparer la particule détergente séchée par pulvérisation comprenant les étapes de :

- i) mélange d'un précurseur acide d'un tensioactif anionique et d'un agent neutralisant hydroxyde de métal alcalin dans un milieu aqueux pour former une solution partiellement neutralisée, dans lequel la solution partiellement neutralisée comprend un sel tensioactif anionique formé in situ par la réaction d'une portion de précurseur de

tensioactif anionique acide avec l'agent neutralisant hydroxyde de métal alcalin et ledit sel tensioactif anionique forme 10 parties à 98 parties de la teneur totale en tensioactif anionique en poids de la particule détergente séchée par pulvérisation ; et

ii) mise en contact de la solution partiellement neutralisée avec un agent neutralisant silicate, dans laquelle l'agent neutralisant silicate est fourni en une quantité suffisante pour neutraliser la partie restante du précurseur acide d'un tensioactif anionique pour former une solution détergente complètement neutralisée,

iii) addition d'ingrédient pour le linge à la solution détergente complètement neutralisée pour former une bouillie détergente aqueuse, dans laquelle la bouillie détergente a une teneur en eau située dans la plage allant de 25 % en poids à 40 % en poids ;

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

2. Procédé selon la revendication 1, dans lequel le sel tensioactif anionique formé par la neutralisation de la partie restante du précurseur acide d'un tensioactif anionique avec l'agent neutralisant silicate comprend 1 % en poids à 7 % en poids de sel tensioactif anionique en valeur absolue en poids de la particule détergente séchée par pulvérisation.

3. Procédé selon la revendication 1 ou 2, dans lequel l'agent neutralisant silicate est fourni en une quantité en excès stoechiométrique en relation avec la partie restante du précurseur acide liquide d'un tensioactif anionique pour former la solution détergente complètement neutralisée.

4. Procédé selon les revendications 1 à 3, dans lequel le rapport de l'agent neutralisant silicate au sel tensioactif anionique formé par la neutralisation de la partie restante du précurseur acide liquide dans la particule séchée par pulvérisation est de 5 à 30.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'agent neutralisant silicate est de préférence un sel silicate de métal alcalin ou de métal alcalino-terreux, mieux encore le silicate de sodium.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'agent neutralisant silicate a un rapport molaire M_2O/SiO_2 de 1/1,6 à 1/3,3, de préférence de 1/1,6 à 1/2,4, mieux encore de 1/2 à 1/2,85, dans lequel M est un métal alcalin.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel le précurseur acide liquide d'un tensioactif anionique est un acide alkylbenzènesulfonique, de préférence un acide (alkyle linéaire en C_{10} à C_{20})benzènesulfonique.

8. Procédé selon l'une quelconque des revendications précédentes, dans lequel, avant le séchage par pulvérisation pour former une particule séchée par pulvérisation, la solution détergente complètement neutralisée est convertie en une bouillie détergente aqueuse comprenant l'étape de : mélange de la solution détergente complètement neutralisée avec un ou plusieurs ingrédients pour le linge choisis parmi (i) un sel adjuvant carbonate ; (ii) un co-tensioactif pré-neutralisé additionnel, (iii) un polymère de carboxylate, (iv) des charges choisies dans le groupe constitué par un sulfate de métal alcalin, un chlorure de métal alcalin, un carbonate de métal alcalino-terreux ou leurs mélanges, mieux encore la charge est choisie parmi le sulfate de sodium, le chlorure de sodium, le carbonate de calcium, le carbonate de magnésium, la dolomite, la calcite ou leurs mélanges.

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

i) un sel tensioactif anionique formé in situ ;

ii) du carbonate de sodium ;

iii) un sel silicate de métal alcalin ;

iv) une charge choisie dans le groupe constitué par un sulfate de métal alcalin, un chlorure de métal alcalin, un carbonate de métal alcalino-terreux ou leurs mélanges, mieux encore la charge est choisie parmi le sulfate de sodium, le chlorure de sodium, le carbonate de calcium, le carbonate de magnésium, la dolomite, la calcite ou leurs mélanges ;

v) 25 % en poids à 40 % en poids d'eau.

10. Particule détergente séchée par pulvérisation pouvant être obtenue par séchage par pulvérisation d'une bouillie

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aqueuse selon l'une quelconque des revendications 1 à 9, laquelle particule détergente séchée par pulvérisation a une alcalinité de réserve à pH 10 située dans la plage allant de 1,5 à 5 grammes de NaOH pour 100 g, où l'alcalinité de réserve se réfère aux grammes de NaOH pour 100 grammes lors d'une titration jusqu'à un pH de 10 avec 0,5 mol/l de H₂SO₄.

11. Particule détergente séchée par pulvérisation selon la revendication 10, laquelle particule détergente séchée par pulvérisation formée par séchage par pulvérisation de la bouillie détergente aqueuse comprend 7 % en poids à 50 % en poids de sel tensioactif anionique formé in situ.

12. Particule détergente séchée par pulvérisation selon la revendication 10 ou 11, laquelle particule détergente séchée par pulvérisation comprend :

- i) 7 % en poids à 50 % en poids de tensioactif anionique formé in situ ;
- ii) éventuellement 0 % en poids à 20 % en poids de co-tensioactif, de préférence choisi parmi le PAS, le SLES ou leurs mélanges ;
- iii) 8 % en poids à 20 % en poids de sel silicate, de préférence avec un rapport en poids SiO₂/M₂O de 1/6 à 1/3,3 ;
- iv) de préférence 8 % en poids à 70 % en poids d'adjuvant à base de carbonate ;
- v) des ingrédients pour le linge de préférence choisis parmi (i) un sel tensioactif anionique additionnel, (ii) des charges choisies dans le groupe constitué par un sulfate de métal alcalin, un chlorure de métal alcalin, un carbonate de métal alcalino-terreux ou leurs mélanges, mieux encore la charge est choisie parmi le sulfate de sodium, le chlorure de sodium, le carbonate de calcium (calcite), le carbonate de magnésium, le carbonate de calcium-magnésium ou leurs combinaisons.

REFERENCES CITED IN THE DESCRIPTION

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