



(11) **EP 2 138 565 A1**

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:  
**30.12.2009 Bulletin 2009/53**

(51) Int Cl.:  
**C11D 11/02 (2006.01) C11D 11/04 (2006.01)**

(21) Application number: **08159025.9**

(22) Date of filing: **25.06.2008**

(84) Designated Contracting States:  
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR  
HR HU IE IS IT LI LT LU LV MC MT NL NO PL PT  
RO SE SI SK TR**  
Designated Extension States:  
**AL BA MK RS**

(71) Applicant: **The Procter and Gamble Company  
Cincinnati, Ohio 45202 (US)**

(72) Inventors:  
• **Tantawy, Hossam Hassan  
Northumberland NE61 3JT (GB)**

- **Somerville Roberts, Nigel Patrick  
Newcastle upon Tyne NE4 9UQ (GB)**
- **Howe, Simon  
Tyne and Wear NE34 9HF (GB)**
- **Caldwell, Stuart  
Northumberland NE23 3XL (GB)**

(74) Representative: **Howard, Phillip Jan  
Procter & Gamble  
Technical Centres Limited  
Whitley Road  
Longbenton  
Newcastle upon Tyne NE12 9TS (GB)**

(54) **A spray-drying process**

(57) The present invention relates to a process for preparing a spray-dried powder comprising

- (i) anionic deterative surfactant;
- (ii) from 0wt% to 10wt% zeolite builder;
- (iii) from 0wt% to 10wt% phosphate builder; and
- (iv) optionally from 0wt% to 15wt% silicate salt;

wherein the process comprises the steps of:

- (a) forming an alkaline slurry in a mixer, the slurry comprising:
- (v) from 0wt% to 15wt% anionic deterative surfactant;
- (vi) from 0wt% to 35wt% water; and

(b) transferring the alkaline slurry from the mixer through

at least one pump to a spray pressure nozzle;

- (c) contacting an acid anionic deterative surfactant precursor to the alkaline slurry after the mixer and before the spray pressure nozzle to form a mixture;
- (d) spraying the mixture through the spray pressure nozzle into a spray-drying tower;
- (e) spray-drying the mixture to form a spray-dried powder; and
- (f) optionally, contacting an alkalinity source with the alkaline slurry and/or the acid anionic deterative surfactant precursor, and/or the mixture.

**EP 2 138 565 A1**

**Description**

## FIELD OF THE INVENTION

5 **[0001]** The present invention relates to a spray-drying process.

## BACKGROUND OF THE INVENTION

10 **[0002]** Laundry detergent compositions are typically made by a process that involves the step of spray-drying an aqueous slurry comprising anionic deterative surfactant to form a spray-dried powder. Typically, this spray-drying step is the rate determining step in the production of the laundry detergent powder. This is especially true for low-built formulations that have increased drying loads. With increasing global laundry detergent powder volume demand, many detergent manufacturers' spray-drying towers are running at, or very near, maximum capacity. In order to increase the capacity of their existing spray-drying facilities, detergent manufacturers have to either invest in additional or upgraded

15 spray-drying equipment and/or formulate their spray-dried powder with process aids that increase the formulation complexity of the spray-dried laundry detergent powder.

**[0003]** The inventors have found that by taking at least some of the anionic deterative surfactant from the aqueous slurry in the mixer, and adding its acid precursor at a later stage (i.e. to the alkaline slurry) in the spray-drying process, and by carefully controlling the moisture level of the alkaline slurry in the mixer, the capacity rate of the spray-drying process is significantly increased without the need for investment in additional spray-drying equipment or the need for

20 the incorporation of process aids.

## SUMMARY OF THE INVENTION

25 **[0004]** The present invention relates to a process as defined in claim 1.

## DETAILED DESCRIPTION OF THE INVENTION

Spray-drying process,

30 **[0005]** The process comprises the steps of: (a) forming an alkaline slurry in a mixer; (b) transferring the alkaline slurry from the mixer through at least one pump to a spray pressure nozzle; (c) contacting an acid anionic deterative surfactant precursor to the alkaline slurry after the mixer and before the spray pressure nozzle to form a mixture; (d) spraying the mixture through the spray pressure nozzle into a spray-drying tower; (e) spray-drying the mixture to form a spray-dried powder; and (f) optionally, contacting an alkalinity source with the alkaline slurry and/or the acid anionic deterative surfactant precursor, and/or the mixture. Each of the process steps are described in more detail below.

35

Step (a)

40 **[0006]** In step (a), an alkaline slurry is formed in a mixer. The preferred mixer in step (a) is a crutcher mixer. The alkaline slurry in the mixer is preferably heated, typically in the range of 50°C to 90°C. Saturated steam can be used to heat the slurry in the mixer. Preferably, all of the liquid components that make up the slurry are heated prior to addition to the mixer, and the slurry is preferably maintained at an elevated temperature in the mixer. These temperatures are preferably in the range of 50°C to 90°C.

45 **[0007]** Typically, the residence time of the slurry in the mixer is in the range of from 20 seconds to 600 seconds.

**[0008]** The mixer in step (a) typically has a motor size such that its installed power is in the range of from 50kW to 100kW.

Step (b)

50 **[0009]** In step (b), the alkaline slurry is transferred from the mixer through at least one pump to a spray pressure nozzle. Typically, the alkaline slurry is first transferred to a low pressure line. The low pressure line typically has a pressure in the range of from  $4.0 \times 10^5$  Pa to  $1.2 \times 10^6$  Pa. Typically, the alkaline slurry is then pumped into a high pressure line. The high pressure line typically has a pressure in the range of from  $4.0 \times 10^6$  Pa to  $1.2 \times 10^7$  Pa. Typically, a high pressure pump is used to transfer the alkaline slurry from the low pressure line to the high pressure line. Preferably the

55 high pressure pump is a piston pump.

**[0010]** Typically, the alkaline slurry passes through a second mixer during step (b). The second mixer is preferably a slurry disintegrator. The second mixer is typically operated at 1,000 rpm to 3,000 rpm. This second mixer reduces the particle size of the solid material in the slurry. The particle size of the solid material in the slurry at the end of step (b) is

preferably less than 2.0mm. This mitigates the risk of blocking the spray pressure nozzle.

#### Step (c)

**[0011]** In step (c), an acid anionic deterative surfactant precursor is contacted to the alkaline slurry after the mixer and before the spray pressure nozzle to form a mixture. Preferably, the acid anionic surfactant precursor is contacted to the alkaline slurry in a low pressure line. However, the acid anionic surfactant precursor may be contacted to the alkaline slurry in a high pressure line. Typically, the temperature of the acid anionic deterative surfactant precursor is in the range of from 20°C to 50°C when it is contacted with the alkaline slurry. Typically, the ratio of the flow rate of the alkaline slurry to the flow rate of the acid anionic deterative surfactant precursor is controlled. This control is typically achieved by passing the acid anionic deterative surfactant precursor through a mass flow meter, and monitoring the mass flow rate of the alkaline slurry by a loss in weight system installed on a holding tank into which the alkaline slurry is typically transferred prior to it being pumped into the low pressure line. The ratio of the flow rate of the alkaline slurry to the flow rate of the acid anionic deterative surfactant precursor is typically in the range of from 2.5:1 to 25:1, preferably from 5:1, or from 8:1, and preferably to 20:1, or to 15:1.

#### Step (d)

**[0012]** In step (d), the mixture is sprayed through the spray pressure nozzle into a spray-drying tower. Typically, the mixture is sprayed at a pressure in the range of from  $4.0 \times 10^6$  Pa to  $1.2 \times 10^7$  Pa. Typically, the mixture is sprayed at a mass flow rate in the range of from  $1,000 \text{ kg hr}^{-1}$  to  $70,000 \text{ kg hr}^{-1}$ . Typically, a plurality of nozzles are used in the process, preferably the nozzles are positioned in a circumferential manner at different heights throughout the spray-drying tower. The nozzles are preferably positioned in a counter-current manner with respect to the air flow in the tower.

#### Step (e)

**[0013]** In step (e), the mixture is spray-dried to form a spray-dried powder. Typically, the air inlet temperature is in the range of from 200°C to 350°C. Typically, the air in-let flow rate is in the range of from 50,000 to  $150,000 \text{ kg m}^{-3}$ .

#### Optional step (f)

**[0014]** In optional step (f), an alkalinity source is contacted with the alkaline slurry and/or the acid anionic deterative surfactant precursor, and/or the mixture. The alkalinity source is preferably contacted to the alkaline slurry substantially simultaneously with the acid anionic deterative surfactant precursor. Typically, the alkalinity is contacted to the alkaline slurry and/or the acid anionic deterative surfactant precursor, and/or the mixture at a temperature above 10°C; this is especially preferred when the alkalinity source comprises sodium hydroxide.

**[0015]** The alkalinity source can be contacted to the alkaline slurry and/or mixture by injecting the alkalinity source into the low pressure line. Alternative, the alkalinity source, can be injected into the high pressure line.

**[0016]** Typically, the ratio of the flow rate of the alkaline slurry to the flow rate of the alkalinity source is controlled. This control is typically achieved by passing the alkalinity source through a mass flow meter. The control of the mass flow rate of the alkaline slurry is described in more detail above.

#### Alkaline slurry

**[0017]** The alkaline slurry typically comprises: (a) from 0wt% to 15wt% anionic deterative surfactant; and (b) from 0wt% to 35wt% water. The alkaline slurry preferably comprises from 0wt%, or from above 0wt%, and preferably to 30wt%, or to 25wt%, or to 20wt%, or to 15wt%, or even to 10wt% water. The alkaline slurry may be substantially anhydrous. The alkaline slurry typically comprises one or more adjunct detergent ingredients. The alkaline slurry preferably comprises carbonate salt, preferably at least 5wt%, or at least 10wt% carbonate salt. Preferably, the alkaline slurry comprises from 0wt% to 10wt%, or from above 0wt%, and preferably to 8wt%, or to 6wt%, or to wt%, or to 2wt% anionic surfactant. The alkaline slurry may even be essentially free of anionic deterative surfactant. By essential free of, it is typically meant comprises no deliberately added.

**[0018]** The alkaline slurry may comprise polymeric material. A preferred polymeric material is a carboxylate polymer. The alkaline slurry may comprise at least 1wt%, or even at least 2wt% polymeric material.

**[0019]** Preferably, the weight ratio of solid inorganic material to solid organic material present in the slurry is in the range of from 10:1 to 10,000:1, preferably at least 35:1. The alkaline slurry may comprise less than 10wt% solid organic material, or less than 5wt% solid organic material. The alkaline slurry may even be essential free of solid organic material. For the purpose of the present invention, organic means any hydrocarbon component.

Spray-dried powder

**[0020]** The spray-dried powder comprises: (i) anionic deterative surfactant; (ii) from 0wt% to 10wt% zeolite builder; (iii) from 0wt% to 10wt% phosphate builder; and (iv) optionally from 0wt% to 15wt% silicate salt. The spray-dried powder typically comprises additional adjunct detergent ingredients. Preferably, the spray-dried powder comprises a carbonate salt.

**[0021]** The spray-dried powder typically has a particle size distribution such that the weight average particle size is in the range of from 300 micrometers to 600 micrometers, and preferably less than 10wt% of the spray-dried powder has a particle size greater than 1,180 micrometers, and preferably less than 10wt% of the spray-dried powder has a particle size of less than 150 micrometers.

**[0022]** Typically, the spray-dried powder has a bulk density in the range of from 100g/l to 700g/l. The spray-dried powder typically has a moisture content of less than 5wt%, preferably less than 4wt%, or even less than 3wt%. Preferably, the spray-dried powder is white.

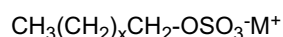
Acid anionic deterative surfactant precursor

**[0023]** The acid anionic deterative surfactant precursor preferably comprises C<sub>8</sub>-C<sub>24</sub> alkyl benzene sulphonic acid. However, any acid anionic deterative surfactant precursor may be used in the present invention.

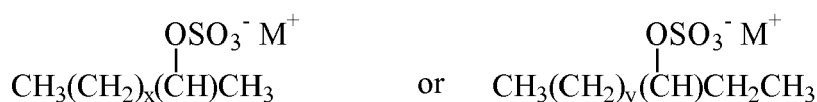
Anionic deterative surfactant

**[0024]** The anionic deterative surfactant preferably comprises alkyl benzene sulphonate. Preferably the anionic deterative surfactant comprises at least 50%, preferably at least 55%, or at least 60%, or at least 65%, or at least 70%, or even at least 75%, by weight of the anionic deterative surfactant, of alkyl benzene sulphonate. Preferably the alkyl benzene sulphonate is a linear or branched, substituted or unsubstituted, C<sub>8-18</sub> alkyl benzene sulphonate. This is the optimal level of the C<sub>8-18</sub> alkyl benzene sulphonate to provide a good cleaning performance. The C<sub>8-18</sub> alkyl benzene sulphonate can be a modified alkylbenzene sulphonate (MLAS) as described in more detail in WO 99/05243, WO 99/05242, WO 99/05244, WO 99/05082, WO 99/05084, WO 99/05241, WO 99/07656, WO 00/23549, and WO 00/23548. Highly preferred C<sub>8-18</sub> alkyl benzene sulphonates are linear C<sub>10-13</sub> alkylbenzene sulphonates. Especially preferred are linear C<sub>10-13</sub> alkylbenzene sulphonates that are obtainable, preferably 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®.

**[0025]** The anionic deterative surfactant may preferably comprise other anionic deterative surfactants. A preferred adjunct anionic deterative surfactant is a non-alkoxylated anionic deterative surfactant. The non-alkoxylated anionic deterative surfactant can be an alkyl sulphate, an alkyl phosphate, an alkyl phosphonate, an alkyl carboxylate or any mixture thereof. The non-alkoxylated anionic surfactant can be selected from the group consisting of; C<sub>10</sub>-C<sub>20</sub> primary, branched-chain, linear-chain and random-chain alkyl sulphates (AS), typically having the following formula:



wherein, M is hydrogen or a cation which provides charge neutrality, preferred cations are sodium and ammonium cations, wherein x is an integer of at least 7, preferably at least 9; C<sub>10</sub>-C<sub>18</sub> secondary (2,3) alkyl sulphates, typically having the following formulae:



wherein, M is hydrogen or a cation which provides charge neutrality, preferred cations include sodium and ammonium cations, wherein x is an integer of at least 7, preferably at least 9, y is an integer of at least 8, preferably at least 9; C<sub>10</sub>-C<sub>18</sub> alkyl carboxylates; mid-chain branched alkyl sulphates as described in more detail in US 6,020,303 and US 6,060,443; methyl ester sulphonate (MES); alpha-olefin sulphonate (AOS); and mixtures thereof.

**[0026]** Another preferred anionic deterative surfactant is an alkoxylated anionic deterative surfactant. The presence of

an alkoxyated anionic deterative surfactant in the spray-dried powder provides good greasy soil cleaning performance, gives a good sudsing profile, and improves the hardness tolerance of the anionic deterative surfactant system. It may be preferred for the anionic deterative surfactant to comprise from 1% to 50%, or from 5%, or from 10%, or from 15%, or from 20%, and to 45%, or to 40%, or to 35%, or to 30%, by weight of the anionic deterative surfactant system, of an alkoxyated anionic deterative surfactant.

**[0027]** Preferably, the alkoxyated anionic deterative surfactant is a linear or branched, substituted or unsubstituted C<sub>12-18</sub> alkyl alkoxyated sulphate having an average degree of alkoxylation of from 1 to 30, preferably from 1 to 10. Preferably, the alkoxyated anionic deterative surfactant is a linear or branched, substituted or unsubstituted C<sub>12-18</sub> alkyl ethoxyated sulphate having an average degree of ethoxylation of from 1 to 10. Most preferably, the alkoxyated anionic deterative surfactant is a linear unsubstituted C<sub>12-18</sub> alkyl ethoxyated sulphate having an average degree of ethoxylation of from 3 to 7.

**[0028]** The alkoxyated anionic deterative surfactant, when present with an alkyl benzene sulphonate may also increase the activity of the alkyl benzene sulphonate by making the alkyl benzene sulphonate less likely to precipitate out of solution in the presence of free calcium cations. Preferably, the weight ratio of the alkyl benzene sulphonate to the alkoxyated anionic deterative surfactant is in the range of from 1:1 to less than 5:1, or to less than 3:1, or to less than 1.7:1, or even less than 1.5:1. This ratio gives optimal whiteness maintenance performance combined with a good hardness tolerance profile and a good sudsing profile. However, it may be preferred that the weight ratio of the alkyl benzene sulphonate to the alkoxyated anionic deterative surfactant is greater than 5: 1, or greater than 6:1, or greater than 7:1, or even greater than 10:1. This ratio gives optimal greasy soil cleaning performance combined with a good hardness tolerance profile, and a good sudsing profile.

**[0029]** Suitable alkoxyated anionic deterative surfactants are: Texapan LEST™ by Cognis; Cosmacol AES™ by Sasol; BES151™ by Stephan; Empicol ESC70/U™; and mixtures thereof.

**[0030]** Preferably, the anionic deterative surfactant comprises from 0% to 10%, preferably to 8%, or to 6%, or to 4%, or to 2%, or even to 1%, by weight of the anionic deterative surfactant, of unsaturated anionic deterative surfactants such as alpha-olefin sulphonate. Preferably the anionic deterative surfactant is essentially free of unsaturated anionic deterative surfactants such as alpha-olefin sulphonate. By "essentially free of" it is typically meant "comprises no deliberately added". Without wishing to be bound by theory, it is believed that these levels of unsaturated anionic deterative surfactants such as alpha-olefin sulphonate ensure that the anionic deterative surfactant is bleach compatible.

**[0031]** Preferably, the anionic deterative surfactant comprises from 0% to 10%, preferably to 8%, or to 6%, or to 4%, or to 2%, or even to 1%, by weight of alkyl sulphate. Preferably the anionic deterative surfactant is essentially free of alkyl sulphate. Without wishing to be bound by theory, it is believed that these levels of alkyl sulphate ensure that the anionic deterative surfactant is hardness tolerant.

#### Zeolite builder

**[0032]** The spray-dried powder typically comprises from 0% to 10wt% zeolite builder, preferably to 9wt%, or to 8wt%, or to 7wt%, or to 6wt%, or to 5wt%, or to 4wt%, or to 3wt%, or to 2wt%, or to 1wt%, or to less than 1% by weight of the spray-dried powder, of zeolite builder. It may even be preferred for the spray-dried powder to be essentially free from zeolite builder. By essentially free from zeolite builder it is typically meant that the spray-dried powder comprises no deliberately added zeolite builder. This is especially preferred if it is desirable for the spray-dried powder to be very highly soluble, to minimise the amount of water-insoluble residues (for example, which may deposit on fabric surfaces), and also when it is highly desirable to have transparent wash liquor. Zeolite builders include zeolite A, zeolite X, zeolite P and zeolite MAP.

#### Phosphate builder

**[0033]** The spray-dried powder typically comprises from 0% to 10wt% phosphate builder, preferably to 9wt%, or to 8wt%, or to 7wt%, or to 6wt%, or to 5wt%, or to 4wt%, or to 3wt%, or to 2wt%, or to 1wt%, or to less than 1% by weight of the spray-dried powder, of phosphate builder. It may even be preferred for the spray-dried powder to be essentially free from phosphate builder. By essentially free from phosphate builder it is typically meant that the spray-dried powder comprises no deliberately added phosphate builder. This is especially preferred if it is desirable for the composition to have a very good environmental profile. Phosphate builders include sodium tripolyphosphate.

#### Silicate salt

**[0034]** The spray-dried powder optionally comprises from 0% to 20wt% silicate salt, preferably from 1wt%, or from 2wt%, or from 3wt%, and preferably to 15wt%, or to 10wt%, or even to 5% silicate salt. Silicate salts include amorphous silicates and crystalline layered silicates (e.g. SKS-6). A preferred silicate salt is sodium silicate.

Carbonate salt

**[0035]** The spray-dried powder typically comprises carbonate salt, typically from 1% to 50%, or from 5% to 25% or from 10% to 20%, by weight of the spray-dried powder, of carbonate salt. A preferred carbonate salt is sodium carbonate and/or sodium bicarbonate. A highly preferred carbonate salt is sodium carbonate. Preferably, the spray-dried powder may comprise from 10% to 40%, by weight of the spray-dried powder, of sodium carbonate. However, it may also be preferred for the spray-dried powder to comprise from 2% to 8%, by weight of the spray-dried powder, of sodium bicarbonate. Sodium bicarbonate at these levels provides good alkalinity whilst minimizing the risk of surfactant gelling which may occur in surfactant-carbonate systems. If the spray-dried powder comprises sodium carbonate and zeolite, then preferably the weight ratio of sodium carbonate to zeolite is at least 15:1.

**[0036]** High levels of carbonate improve the cleaning performance of the composition by increasing the pH of the wash liquor. This increased alkalinity: improves the performance of the bleach, if present; increases the tendency of soils to hydrolyse, which facilitates their removal from the fabric; and also increases the rate, and degree, of ionization of the soils to be cleaned (n.b. ionized soils are more soluble and easier to remove from the fabrics during the washing stage of the laundering process). In addition, high carbonate levels improve the flowability of the spray-dried powder.

Alkalinity source

**[0037]** The alkalinity source preferably comprises sodium hydroxide. The alkalinity source preferably comprises carbonate salt. The alkalinity source preferably comprises silicate salt.

Adjunct detergent ingredients

**[0038]** Suitable adjunct ingredients include: deterative surfactants such as anionic deterative surfactants, nonionic deterative surfactants, cationic deterative surfactants, zwitterionic deterative surfactants, amphoteric deterative surfactants; preferred nonionic deterative surfactants are C<sub>8-18</sub> alkyl alkoxyated alcohols having an average degree of alkoxylation of from 1 to 20, preferably from 3 to 10, most preferred are C<sub>12-18</sub> alkyl ethoxyated alcohols having an average degree of alkoxylation of from 3 to 10; preferred cationic deterative surfactants are mono-C<sub>6-18</sub> alkyl mono-hydroxyethyl di-methyl quaternary ammonium chlorides, more preferred are mono-C<sub>8-10</sub> alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride, mono-C<sub>10-12</sub> alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride and mono-C<sub>10</sub> alkyl mono-hydroxyethyl di-methyl quaternary ammonium chloride; 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 ingredient such as a carbonate salt, a sulphate salt, a silicate salt, borosilicate, or mixtures, including mixed salts, thereof; bleach activator such as tetraacetylethylene 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 peroxyacaproic acid, nonylamido peroxyadipic acid or dibenzoyl peroxide; 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; filler salts such as sulphate salts, preferably sodium sulphate; 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 co-polymer 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 alkoxyated polyamines and ethoxyated ethyleneimine polymers; anti-redeposition components such as carboxymethyl cellulose and polyesters; perfumes; sulphamic acid or salts thereof; citric acid or salts thereof; and dyes such as orange dye, blue dye, green dye, purple dye, pink dye, or any mixture thereof.

## EXAMPLES

**[0039]** While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

**Example 1. A spray-dried laundry detergent powder and process of making it.**Aqueous alkaline slurry composition.

[0040]

Component	Aqueous slurry (parts)
Sodium Silicate	8.5
Acrylate/maleate copolymer	3.2
Hydroxyethane di(methylene phosphonic acid)	0.6
Sodium carbonate	8.8
Sodium sulphate	42.9
Water	19.7
Miscellaneous, such as magnesium sulphate, and one or more stabilizers	1.7
<b>Aqueous alkaline slurry parts</b>	<b>85.4</b>

Preparation of a spray-dried laundry detergent powder.

[0041] An alkaline aqueous slurry having the composition as described above is prepared in a slurry making vessel (crutcher). The moisture content of the above slurry is 23.1 %. Any ingredient added above in liquid form is heated to 70°C, such that the aqueous slurry is never at a temperature below 70°C. Saturated steam at a pressure of  $6.0 \times 10^5$  Pa is injected into the crutcher to raise the temperature to 90°C. The slurry is then pumped into a low pressure line (having a pressure of  $5.0 \times 10^5$  Pa).

[0042] Separately, 11.4 parts of  $C_8$ - $C_{24}$  alkyl benzene sulphononic acid (HLAS), and 3.2 parts of a 50w/w% aqueous sodium hydroxide solution are pumped into the low pressure line. The mixture is then pumped by a high pressure pump into a high pressure line (having an exit pressure of  $8.0 \times 10^6$  Pa). The mixture is then sprayed at a rate of 1,640kg/hour at a pressure of  $8.0 \times 10^6$  Pa and at a temperature of 90°C  $\pm 2^\circ$ C through a spray pressure nozzle into a counter current spray-drying tower with an air inlet temperature of 300°C. The mixture is atomised and the atomised slurry is dried to produce a solid mixture, which is then cooled and sieved to remove oversize material ( $>1.8$ mm) to form a spray-dried powder, which is free-flowing. Fine material ( $<0.15$ mm) is elutriated with the exhaust the exhaust air in the spray-drying tower and collected in a post tower containment system. The spray-dried powder has a moisture content of 2.5wt%, a bulk density of 510 g/l and a particle size distribution such that greater than 80wt% of the spray-dried powder has a particle size of from 150 to 710 micrometers. The composition of the spray-dried powder is given below.

Spray-dried laundry detergent powder composition

[0043]

Component	%w/w Spray Dried Powder
Sodium silicate salt	10.0
$C_8$ - $C_{24}$ alkyl benzene sulphonate	15.1
Acrylate/maleate copolymer	4.0
Hydroxyethane di(methylene phosphonic acid)	0.7
Sodium carbonate	11.9
Sodium sulphate	53.7
Water	2.5
Miscellaneous, such as magnesium sulphate, and one or more stabilizers	2.1
<b>Total Parts</b>	<b>100.00</b>

A granular laundry detergent composition.**[0044]**

5	<b>Component</b>	<b>%w/w granular laundry detergent composition</b>
	Spray-dried powder of example 1 (described above)	59.38
	91.6wt% active linear alkyl benzene sulphonate flake supplied by Stepan under the tradename Nacconol 90G®	0.22
10	Citric acid	5.00
	Sodium percarbonate (having from 12% to 15% active AvOx)	14.70
	Photobleach particle	0.01
15	Lipase (11.00mg active/g)	0.70
	Amylase (21.55mg active/g)	0.33
	Protease (56.00mg active/g)	0.43
20	Tetraacetyl ethylene diamine agglomerate (92wt% active)	4.35
	Suds suppressor agglomerate (11.5wt% active)	0.87
	Acrylate/maleate copolymer particle (95.7wt% active)	0.29
	Green/Blue carbonate speckle	0.50
25	Sodium Sulphate	9.59
	Solid perfume particle	0.63
	Ethoxylated C <sub>12</sub> -C <sub>18</sub> alcohol having an average degree of ethoxylation of 7 (AE7)	3.00
30	<b>Total Parts</b>	<b>100.00</b>

**[0045]** The above laundry detergent composition was prepared by dry-mixing all of the above particles (all except the AE7) in a standard batch mixer. The AE7 in liquid form is sprayed on the particles in the standard batch mixer. Alternatively, the AE7 in liquid form is sprayed onto the spray-dried powder of example 1. The resultant powder is then mixed with all of the other particles in a standard batch mixer.

**[0046]** The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40 mm" is intended to mean "about 40 mm".

**Claims**

**1.** A process for preparing a spray-dried powder comprising

- (i) anionic deterative surfactant;
  - (ii) from 0wt% to 10wt% zeolite builder;
  - (iii) from 0wt% to 10wt% phosphate builder; and
  - (iv) optionally from 0wt% to 15wt% silicate salt;
- wherein the process comprises the steps of:

(a) forming an alkaline slurry in a mixer, the slurry comprising:

- (v) from 0wt% to 15wt% anionic deterative surfactant;
- (vi) from 0wt% to 35wt% water; and

(b) transferring the alkaline slurry from the mixer through at least one pump to a spray pressure nozzle;



(c) contacting an acid anionic deterative surfactant precursor to the alkaline slurry after the mixer and before the spray pressure nozzle to form a mixture;  
 (d) spraying the mixture through the spray pressure nozzle into a spray-drying tower;  
 (e) spray-drying the mixture to form a spray-dried powder; and  
 (f) optionally, contacting an alkalinity source with the alkaline slurry and/or the acid anionic deterative surfactant precursor, and/or the mixture.

2. A process according to claim 1, wherein the alkaline slurry comprises from 0wt% to 25wt% water.
3. A process according to any preceding claim, wherein the alkaline slurry comprises from 0wt% to 5wt% anionic deterative surfactant.
4. A process according to any preceding claim, wherein an alkalinity source is added to the alkaline slurry substantially simultaneously with the acid anionic deterative surfactant precursor.
5. A process according to any preceding claim, wherein the acid anionic deterative surfactant precursor comprises C<sub>8</sub>-C<sub>24</sub> alkyl benzene sulphonic acid.
6. A process according to any preceding claim, wherein the alkalinity source comprises sodium hydroxide.
7. A process according to any preceding claim, wherein the alkalinity source comprises carbonate salt.
8. A process according to any preceding claim, wherein the alkalinity source comprises silicate salt.



## EUROPEAN SEARCH REPORT

Application Number  
EP 08 15 9025

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	WO 96/03488 A (HENKEL KGAA [DE]) 8 February 1996 (1996-02-08) * page 5, lines 9-15 * * page 6, lines 22-27 * * claims; examples *	1-8	INV. C11D11/02 C11D11/04
A	US 2005/256023 A1 (IMAIZUMI YOSHINOBU [JP] ET AL) 17 November 2005 (2005-11-17) * claims; examples *	1-8	
A	US 2005/101503 A1 (CALDWELL STUART A [GB] ET AL) 12 May 2005 (2005-05-12) * paragraphs [0019], [0020]; claims *	1-8	
			TECHNICAL FIELDS SEARCHED (IPC)
			C11D
The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 11 February 2009	Examiner Péntek, Eric
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

 3  
EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 08 15 9025

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.  
The members are as contained in the European Patent Office EDP file on  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

11-02-2009

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 9603488	A	08-02-1996	AT 167700 T	15-07-1998
			DE 4425968 A1	01-02-1996
			DK 772674 T3	06-04-1999
			EP 0772674 A1	14-05-1997
			ES 2117433 T3	01-08-1998
-----				
US 2005256023	A1	17-11-2005	AU 2003263591 A1	29-03-2004
			CN 1678726 A	05-10-2005
			DE 60315668 T2	05-06-2008
			EP 1534812 A1	01-06-2005
			ES 2289353 T3	01-02-2008
			WO 2004022688 A1	18-03-2004
			KR 20050057125 A	16-06-2005
-----				
US 2005101503	A1	12-05-2005	EP 1529833 A1	11-05-2005
			WO 2005047442 A1	26-05-2005
-----				

**REFERENCES CITED IN THE DESCRIPTION**

*This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.*

**Patent documents cited in the description**

- WO 9905243 A [0024]
- WO 9905242 A [0024]
- WO 9905244 A [0024]
- WO 9905082 A [0024]
- WO 9905084 A [0024]
- WO 9905241 A [0024]
- WO 9907656 A [0024]
- WO 0023549 A [0024]
- WO 0023548 A [0024]
- US 6020303 A [0025]
- US 6060443 A [0025]