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(54) **SPRAY-DRYING PROCESS**

(57) The present invention relates to a spray-drying process for preparing a spray-dried laundry detergent particle comprising deterative surfactant and magnesium sulphate salt, wherein the process comprises the steps of: (a) contacting magnesium sulphate to water to form a first mixture; (b) contacting deterative surfactant to the first mixture to form a second mixture; and (c) spray-drying the second mixture to form a spray-dried laundry detergent particle, wherein step (a) is carried out in the absence of deterative surfactant.

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Description

FIELD OF THE INVENTION

[0001] The field of the present invention is spray-drying processes to make spray-dried laundry detergent particles.

BACKGROUND OF THE INVENTION

[0002] Spray-drying is a common process for making laundry detergent base particles. Typically, these base particles comprise deterative surfactant, and typically other optional detergent ingredients such as polymers and/or chelants. Typically, an aqueous mixture, commonly known as a crutcher mixture, is prepared by adding the deterative surfactant, and if present any other optional detergent ingredients, with water. The crutcher mixture is then spray-dried to form the spray-dried laundry detergent powder. This spray-dried laundry detergent powder can itself be used as a laundry detergent powder. Alternatively, and as is more common, other detergent particles and ingredients are added to this spray-dried laundry detergent powder to form the laundry detergent powder. These other detergent particles can include bleach particles, enzymes, secondary deterative surfactant particles, polymer particles, chelant particles, perfume particles, aesthetic particles. Other detergent ingredients, such as liquid perfume and/or liquid deterative surfactant, such as non-ionic liquid deterative surfactant, can be added to the particles, such as by spray-on addition.

[0003] The spray-dried laundry detergent needs to have good physical properties, such as good grain stability, good flowability and good hardness properties. Detergent manufacturers often incorporate ingredients into the spray-dried particle to improve these physical properties.

[0004] The incorporation of magnesium sulphate into a spray-dried laundry detergent particle provides good physical properties. This is especially so when the spray-dried laundry detergent particle has a low pH profile. Magnesium sulphate can either be added as a solution, a hydrate or as anhydrous powder. Solutions of $MgSO_4$ are typically low in concentration, to prevent it from crystallization, making it cost prohibitive. Hydrated forms of Magnesium sulphate, where some forms are more stable than another, do exist. One stable form is a heptahydrate, however this material is particularly difficult to handle. It is therefore preferred to use the most concentrated form which is anhydrous Magnesium sulphate because it's not only free flowing powder but it is also the most cost effective. However, the inventors have found the additional of the anhydrous magnesium sulphate into the spray-drying process needs to be carefully controlled. Air entrainment can naturally occur due to addition of different ingredients in the crutcher. However, addition of anhydrous $MgSO_4$ powder seem to exacerbate the negative impact of aeration. Specially, the inventors have found that the anhydrous magnesium sulphate should be contacted to water in the absence of deterative surfactant. In this manner, undesirable aeration that can occur during the dissolution of the magnesium sulphate in the crutcher mixture is reduced. Aeration of the deterative surfactant, and especially anionic deterative surfactants, such as alkyl benzene sulphonate, which is a problem during the formation of the crutcher mixture. This aeration can be such that the materials, and especially the deterative surfactant and water, can overflow out of the mixer during the process.

[0005] The process of the present invention overcomes these problems and provides a spray-dried laundry detergent particle comprising both deterative surfactant and magnesium sulphate. The spray-dried laundry detergent particles have good physical properties and are suitable for use in laundry detergent powder applications.

SUMMARY OF THE INVENTION

[0006] The present invention relates to a spray-drying process for preparing a spray-dried laundry detergent particle comprising deterative surfactant and magnesium sulphate salt, wherein the process comprises the steps of: (a) contacting magnesium sulphate to water to form a first mixture; (b) contacting deterative surfactant to the first mixture to form a second mixture; and (c) spray-drying the second mixture to form a spray-dried laundry detergent particle, wherein step (a) is carried out in the absence of deterative surfactant.

DETAILED DESCRIPTION OF THE INVENTION

[0007] Spray-drying process: The spray-drying process prepares a spray-dried laundry detergent particle comprising deterative surfactant and magnesium sulphate salt. The process comprises the steps of: (a) contacting magnesium sulphate to water to form a first mixture; (b) contacting deterative surfactant to the first mixture to form a second mixture; and (c) spray-drying the second mixture to form a spray-dried laundry detergent particle, wherein step (a) is carried out in the absence of deterative surfactant.

[0008] Ste (a): During step (a), magnesium sulphate is contacted to water to form a first mixture. It is essential that step (a) is carried out in the absence of deterative surfactant.

[0009] Step (b): During step (b), deterative surfactant is contacted to the first mixture to form a second mixture.

[0010] Ste (c): During step (c), the second mixture is spray-dried to form a spray-dried laundry detergent particle. Typically, the spray-drying of the second mixture comprises the steps of transferring the second mixture through a pipe leading through a first pump and then through a second pump to a number of pressure spray nozzles and maybe installed at different levels in the tower. The first pump is typically a low pressure pump, such as a pump that is capable of generating a pressure of from 3×10^5 to 1×10^6 Pa. Typically, the second pump is a high pressure pump, such as a pump that is capable of generating a pressure of from 2×10^6 to 1×10^7 Pa. The pressure in the pipe at the outlet of the first pump may be less than 1×10^6 Pa. Optionally, the second mixture is transferred through a disintegrator, such as disintegrators supplied by Hosakawa Micron. Typically, disintegrators, is positioned between the pumps. The flow rate of the second mixture along the pipes is typically in the range of from 800 kg/hour to >50,000 kg/hour.

[0011] A suitable spray nozzle is a spray systems T4C8 nozzle. Preferably the second mixture is at a temperature of from 60 °C to 130 °C. Suitable spray drying towers are concurrent or counter current spray drying towers, where the latter maybe operated as a swirl tower. Preferably, the inlet air temperature to the spray drying tower is in the range of from 220 °C to 350 °C. Preferably, the exhaust air temperature to the spray drying tower is in the range of from 60 °C to 100 °C.

[0012] The spray-dried laundry detergent powder may be subjected to cooling, for example an air lift. Typically, the spray-dried laundry detergent powder is subjected to particle size classification to remove oversize material (> 1.8 mm) to form a spray-dried laundry detergent powder which is free flowing. Fine material (< 0.15 mm) is elutriated with the exhaust air in the spray drying tower and collected in a post tower dust containment system.

[0013] First mixture: The first mixture comprises magnesium sulphate. Preferably the first mixture comprises at least 12% magnesium sulphate. Other ingredients may be present in the first mixture, except for deterative surfactant. However, it may be preferred for any optional ingredients to be included in the second mixture, i.e. a preferred order of addition could be to add the deterative surfactant to the first mixture before adding any other ingredients.

[0014] Second mixture: The second mixture comprises magnesium sulphate. Preferably the second mixture comprises at least 2.5 wt% magnesium sulphate. The second mixture comprises deterative surfactant. Preferably the second mixture comprises at least 6.0 wt% deterative surfactant.

[0015] The second mixture, which can also be known as a crutcher mixture, may also contain other detergent ingredients that are suitable for inclusion into the spray-dried laundry detergent particle. Suitable ingredients are described in more detail below, but include polymers, chelants, hueing dyes, brighteners, colourants and pigments. Preferably, the second mixture comprises carboxylate polymer.

[0016] Spray-dried laundry detergent particle: The spray-dried laundry detergent particle comprises magnesium sulphate. Preferably the spray-dried laundry detergent particle comprises at least 2% magnesium sulphate. The spray-dried laundry detergent particle comprises deterative surfactant. Preferably the detergent particle comprises at least 8% deterative surfactant.

[0017] The spray-dried laundry detergent particle, upon dissolution in de-ionized water at a concentration of 1g/1 and at a temperature of 20°C, has an equilibrium pH in the range of from 4.0 to 8.5.

[0018] The particle may comprise other detergent ingredients. These ingredients are described in more detail below. It may be preferred that the particle is substantially free of silicate salt, such as sodium silicate salt. It may be preferred that the particle is substantially free of carbonate salt, such as sodium carbonate salt. It may be preferred that the particle is substantially free of zeolite. It may be preferred that the particle is substantially free of phosphate builder. Preferably, the particle comprises: (a) from 12wt% to 30wt% deterative surfactant; and (b) from 2wt% to 10wt% magnesium sulphate. The particle may comprise: (c) from 4wt% to 10wt% monomeric organic carboxylic acid, such as citric acid, or salt thereof; (d) from 0.5wt% to 3wt% silica; (e) from 0.5wt% to 5wt% polymer; (f) from 35wt% to 80wt% sodium sulphate; and (g), from 0wt% to 6wt% water.

[0019] The particle may comprise alkalinity agents, a preferred alkalinity agent is NaOH.

[0020] Preferably the particle comprises magnesium sulphate in amorphous form.

[0021] The particle may comprise at least 2.0wt% magnesium sulphate.

[0022] The particle may comprise at least 8.0wt% deterative surfactant.

[0023] Detergent ingredients: Suitable detergent ingredients are selected from: deterative surfactant, such as anionic deterative surfactants, non-ionic deterative surfactants, cationic deterative surfactants, zwitterionic deterative surfactants and amphoteric deterative surfactants; polymers, such as carboxylate polymers, soil release polymer, anti-redeposition polymers, cellulosic polymers and care polymers; bleach, such as sources of hydrogen peroxide, bleach activators, bleach catalysts and pre-formed peracids; photobleach, such as such as zinc and/or aluminium sulphonated phthalocyanine; enzymes, such as proteases, amylases, cellulases, lipases; zeolite builder; phosphate builder; co-builders, such as citric acid and citrate; carbonate, such as sodium carbonate and sodium bicarbonate; sulphate salt, such as sodium sulphate; silicate salt such as sodium silicate; chloride salt, such as sodium chloride; brighteners; chelants; hueing agents; dye transfer inhibitors; dye fixative agents; perfume; silicone; fabric softening agents, such as clay; flocculants, such as polyethyleneoxide; suds suppressors; and any combination thereof.

[0024] Suitable particles may have a low buffering capacity. Such particles typically have a reserve alkalinity to pH

9.5 of less than S.OgNaOH/100g. These low buffered laundry detergent particles typically comprise low levels of carbonate salt.

[0025] Deterative Surfactant: Suitable deterative surfactants include anionic deterative surfactants, non-ionic deterative surfactant, cationic deterative surfactants, zwitterionic deterative surfactants and amphoteric deterative surfactants. Suitable deterative surfactants may be linear or branched, substituted or un-substituted, and may be derived from petrochemical material or biomaterial.

[0026] Anionic deterative surfactant: Suitable anionic deterative surfactants include sulphonate and sulphate deterative surfactants.

[0027] Suitable sulphonate deterative surfactants include methyl ester sulphonates, alpha olefin sulphonates, alkyl benzene sulphonates, especially alkyl benzene sulphonates, preferably C₁₀₋₁₃ alkyl benzene sulphonate. Suitable alkyl benzene sulphonate (LAS) is obtainable, preferably obtained, by sulphonating commercially available linear alkyl benzene (LAB); suitable LAB includes low 2-phenyl LAB, other suitable LAB include high 2-phenyl LAB, such as those supplied by Sasol under the tradename Hyblene®.

[0028] Suitable sulphate deterative surfactants include alkyl sulphate, preferably C₈₋₁₈ alkyl sulphate, or predominantly C₁₂ alkyl sulphate.

[0029] A preferred sulphate deterative surfactant is alkyl alkoxyated sulphate, preferably alkyl ethoxyated sulphate, preferably a C₈₋₁₈ alkyl alkoxyated sulphate, preferably a C₈₋₁₈ alkyl ethoxyated sulphate, preferably the alkyl alkoxyated sulphate has an average degree of alkoxylation of from 0.5 to 20, preferably from 0.5 to 10, preferably the alkyl alkoxyated sulphate is a C₈₋₁₈ alkyl ethoxyated sulphate having an average degree of ethoxylation of from 0.5 to 10, preferably from 0.5 to 5, more preferably from 0.5 to 3 and most preferably from 0.5 to 1.5.

[0030] The alkyl sulphate, alkyl alkoxyated sulphate and alkyl benzene sulphonates may be linear or branched, substituted or un-substituted, and may be derived from petrochemical material or biomaterial.

[0031] Other suitable anionic deterative surfactants include alkyl ether carboxylates.

[0032] Suitable anionic deterative surfactants may be in salt form, suitable counter-ions include sodium, calcium, magnesium, amino alcohols, and any combination thereof. A preferred counterion is sodium.

[0033] Non-ionic deterative surfactant: Suitable non-ionic deterative surfactants are selected from the group consisting of: C₈-C₁₈ alkyl ethoxylates, such as, NEODOL® non-ionic surfactants from Shell; C₆-C₁₂ alkyl phenol alkoxyates wherein preferably the alkoxyate units are ethyleneoxy units, propyleneoxy units or a mixture thereof; C₁₂-C₁₈ alcohol and C₆-C₁₂ alkyl phenol condensates with ethylene oxide/propylene oxide block polymers such as Pluronic® from BASF; alkyl polysaccharides, preferably alkyl polyglycosides; methyl ester ethoxylates; polyhydroxy fatty acid amides; ether capped poly(oxyalkylated) alcohol surfactants; and mixtures thereof.

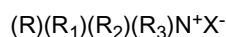
[0034] Suitable non-ionic deterative surfactants are alkyl polyglucoside and/or an alkyl alkoxyated alcohol.

[0035] Suitable non-ionic deterative surfactants include alkyl alkoxyated alcohols, preferably C₈₋₁₈ alkyl alkoxyated alcohol, preferably a C₈₋₁₈ alkyl ethoxyated alcohol, preferably the alkyl alkoxyated alcohol has an average degree of alkoxylation of from 1 to 50, preferably from 1 to 30, or from 1 to 20, or from 1 to 10, preferably the alkyl alkoxyated alcohol is a C₈₋₁₈ alkyl ethoxyated alcohol having an average degree of ethoxylation of from 1 to 10, preferably from 1 to 7, more preferably from 1 to 5 and most preferably from 3 to 7. The alkyl alkoxyated alcohol can be linear or branched, and substituted or un-substituted.

[0036] Suitable nonionic deterative surfactants include secondary alcohol-based deterative surfactants.

[0037] Cationic deterative surfactant: Suitable cationic deterative surfactants include alkyl pyridinium compounds, alkyl quaternary ammonium compounds, alkyl quaternary phosphonium compounds, alkyl ternary sulphonium compounds, and mixtures thereof.

[0038] Preferred cationic deterative surfactants are quaternary ammonium compounds having the general formula:



wherein, R is a linear or branched, substituted or unsubstituted C₆₋₁₈ alkyl or alkenyl moiety, R₁ and R₂ are independently selected from methyl or ethyl moieties, R₃ is a hydroxyl, hydroxymethyl or a hydroxyethyl moiety, X is an anion which provides charge neutrality, preferred anions include: halides, preferably chloride; sulphate; and sulphonate.

[0039] Zwitterionic deterative surfactant: Suitable zwitterionic deterative surfactants include amine oxides and/or betaines.

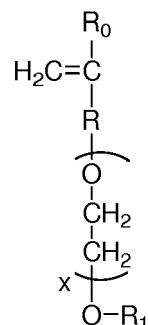
[0040] Polymer: Suitable polymers include carboxylate polymers, soil release polymers, anti-redeposition polymers, cellulosic polymers, care polymers and any combination thereof.

[0041] Carboxylate polymer: The composition may comprise a carboxylate polymer, such as a maleate/acrylate random copolymer or polyacrylate homopolymer. Suitable carboxylate polymers include: polyacrylate homopolymers having a molecular weight of from 4,000 Da to 9,000 Da; maleate/acrylate random copolymers having a molecular weight of from 50,000 Da to 100,000 Da, or from 60,000 Da to 80,000 Da.

[0042] Another suitable carboxylate polymer is a co-polymer that comprises: (i) from 50 to less than 98 wt% structural

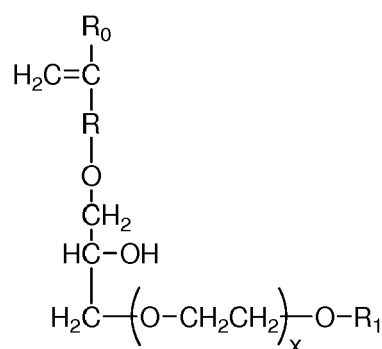
units derived from one or more monomers comprising carboxyl groups; (ii) from 1 to less than 49 wt% structural units derived from one or more monomers comprising sulfonate moieties; and (iii) from 1 to 49 wt% structural units derived from one or more types of monomers selected from ether bond-containing monomers represented by formulas (I) and (II):

formula (I):



wherein in formula (I), R_0 represents a hydrogen atom or CH_3 group, R represents a CH_2 group, CH_2CH_2 group or single bond, X represents a number 0-5 provided X represents a number 1-5 when R is a single bond, and R_1 is a hydrogen atom or C_1 to C_{20} organic group;

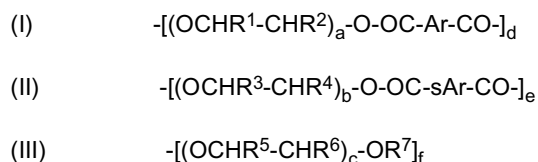
formula (II)



wherein in formula (II), R_0 represents a hydrogen atom or CH_3 group, R represents a CH_2 group, CH_2CH_2 group or single bond, X represents a number 0-5, and R_1 is a hydrogen atom or C_1 to C_{20} organic group.

It may be preferred that the polymer has a weight average molecular weight in the range of from 30kDa to 70kDa.

[0043] Soil release polymer: The composition may comprise a soil release polymer. A suitable soil release polymer has a structure as defined by one of the following structures (I), (II) or (III):



wherein:

a, b and c are from 1 to 200;

d, e and f are from 1 to 50;

Ar is a 1,4-substituted phenylene;

sAr is 1,3-substituted phenylene substituted in position 5 with SO_3Me ;

Me is Li, K, Mg/2, Ca/2, Al/3, ammonium, mono-, di-, tri-, or tetraalkylammonium wherein the alkyl groups are C_1 - C_{18} alkyl or C_2 - C_{10} hydroxyalkyl, or mixtures thereof; R^1 , R^2 , R^3 , R^4 , R^5 and R^6 are independently selected from H or

C_1 - C_{18} n- or iso-alkyl; and

R^7 is a linear or branched C_1 - C_{18} alkyl, or a linear or branched C_2 - C_{30} alkenyl, or a cycloalkyl group with 5 to 9 carbon atoms, or a C_8 - C_{30} aryl group, or a C_6 - C_{30} arylalkyl group. Suitable soil release polymers are sold by Clariant under the TexCare® series of polymers, e.g. TexCare® SRN240 and TexCare® SRA300. Other suitable soil release polymers are sold by Solvay under the Repel-o-Tex® series of polymers, e.g. Repel-o-Tex® SF2 and Repel-o-Tex® Crystal.

[0044] Anti-redeposition polymer: Suitable anti-redeposition polymers include polyethylene glycol polymers and/or polyethyleneimine polymers.

[0045] Suitable polyethylene glycol polymers include random graft co-polymers comprising: (i) hydrophilic backbone comprising polyethylene glycol; and (ii) hydrophobic side chain(s) selected from the group consisting of: C_4 - C_{25} alkyl group, polypropylene, polybutylene, vinyl ester of a saturated C_1 - C_6 mono-carboxylic acid, C_1 - C_6 alkyl ester of acrylic or methacrylic acid, and mixtures thereof. Suitable polyethylene glycol polymers have a polyethylene glycol backbone with random grafted polyvinyl acetate side chains. The average molecular weight of the polyethylene glycol backbone can be in the range of from 2,000 Da to 20,000 Da, or from 4,000 Da to 8,000 Da. The molecular weight ratio of the polyethylene glycol backbone to the polyvinyl acetate side chains can be in the range of from 1:1 to 1:5, or from 1:1.2 to 1:2. The average number of graft sites per ethylene oxide unit can be less than 0.02, or less than 0.016, the average number of graft sites per ethylene oxide unit can be in the range of from 0.010 to 0.018, or the average number of graft sites per ethylene oxide unit can be less than 0.010, or in the range of from 0.004 to 0.008.

[0046] Suitable polyethylene glycol polymers are described in WO08/007320.

[0047] A suitable polyethylene glycol polymer is Sokalan HP22.

[0048] Cellulosic polymer: Suitable cellulosic polymers are selected from alkyl cellulose, alkyl alkoxyalkyl cellulose, carboxyalkyl cellulose, alkyl carboxyalkyl cellulose, sulphaalkyl cellulose, more preferably selected from carboxymethyl cellulose, methyl cellulose, methyl hydroxyethyl cellulose, methyl carboxymethyl cellulose, and mixtures thereof.

[0049] Suitable carboxymethyl celluloses have a degree of carboxymethyl substitution from 0.5 to 0.9 and a molecular weight from 100,000 Da to 300,000 Da.

Suitable carboxymethyl celluloses have a degree of substitution greater than 0.65 and a degree of blockiness greater than 0.45, e.g. as described in WO09/154933.

[0050] Care polymers: Suitable care polymers include cellulosic polymers that are cationically modified or hydrophobically modified. Such modified cellulosic polymers can provide anti-abrasion benefits and dye lock benefits to fabric during the laundering cycle. Suitable cellulosic polymers include cationically modified hydroxyethyl cellulose.

[0051] Other suitable care polymers include dye lock polymers, for example the condensation oligomer produced by the condensation of imidazole and epichlorhydrin, preferably in ratio of 1:4:1. A suitable commercially available dye lock polymer is Polyquart® FDI (Cognis).

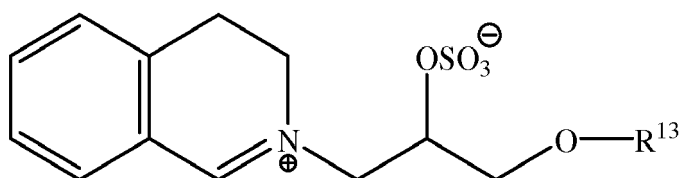
[0052] Other suitable care polymers include amino-silicone, which can provide fabric feel benefits and fabric shape retention benefits.

[0053] Bleach: Suitable bleach includes sources of hydrogen peroxide, bleach activators, bleach catalysts, pre-formed peracids and any combination thereof. A particularly suitable bleach includes a combination of a source of hydrogen peroxide with a bleach activator and/or a bleach catalyst.

[0054] Source of hydrogen peroxide: Suitable sources of hydrogen peroxide include sodium perborate and/or sodium percarbonate.

[0055] Bleach activator: Suitable bleach activators include tetra acetyl ethylene diamine and/or alkyl oxybenzene sulphonate.

[0056] Bleach catalyst: The composition may comprise a bleach catalyst. Suitable bleach catalysts include oxaziridinium bleach catalysts, transition metal bleach catalysts, especially manganese and iron bleach catalysts. A suitable bleach catalyst has a structure corresponding to general formula below:



wherein R^{13} is selected from the group consisting of 2-ethylhexyl, 2-propylheptyl, 2-butyloctyl, 2-pentylnonyl, 2-hexyldecyl, n-dodecyl, n-tetradecyl, n-hexadecyl, n-octadecyl, iso-nonyl, isodecyl, iso-tridecyl and iso-pentadecyl.

[0057] Pre-formed peracid: Suitable pre-form peracids include phthalimido-peroxycaproic acid.

[0058] Enzymes: Suitable enzymes include lipases, proteases, cellulases, amylases and any combination thereof.

[0059] Protease: Suitable proteases include metalloproteases and/or serine proteases. Examples of suitable neutral or alkaline proteases include: subtilisins (EC 3.4.21.62); trypsin-type or chymotrypsin-type proteases; and metalloproteases. The suitable proteases include chemically or genetically modified mutants of the aforementioned suitable proteases.

[0060] Suitable commercially available protease enzymes include those sold under the trade names Alcalase®, Savinase®, Primase®, Durazym®, Polarzyme®, Kannase®, Liquanase®, Liquanase Ultra®, Savinase Ultra®, Ovozyme®, Neutrase®, Everlase® and Esperase® by Novozymes A/S (Denmark), those sold under the tradename Maxatase®, Maxacal®, Maxapem®, Preferenz P® series of proteases including Preferenz® P280, Preferenz® P281, Preferenz® P2018-C, Preferenz® P2081-WE, Preferenz® P2082-EE and Preferenz® P2083-A/J, Properase®, Purafect®, Purafect Prime®, Purafect Ox®, FN3®, FN4®, Excellase® and Purafect OXP® by DuPont, those sold under the tradename Opticlean® and Optimase® by Solvay Enzymes, those available from Henkel/ Kemira, namely BLAP (sequence shown in Figure 29 of US 5,352,604 with the following mutations S99D + S101 R + S103A + V104I + G159S, hereinafter referred to as BLAP), BLAP R (BLAP with S3T + V4I + V199M + V205I + L217D), BLAP X (BLAP with S3T + V4I + V205I) and BLAP F49 (BLAP with S3T + V4I + A194P + V199M + V205I + L217D) - all from Henkel/Kemira; and KAP (Bacillus alkalophilus subtilisin with mutations A230V + S256G + S259N) from Kao.

[0061] A suitable protease is described in WO11/140316 and WO11/072117.

[0062] Amylase: Suitable amylases are derived from AA560 alpha amylase endogenous to Bacillus sp. DSM 12649, preferably having the following mutations: R118K, D183*, G184*, N195F, R320K, and/or R458K. Suitable commercially available amylases include Stainzyme®, Stainzyme® Plus, Natalase, Termamyl®, Termamyl® Ultra, Liquezyme® SZ, Duramyl®, Everest® (all Novozymes) and Spezyme® AA, Preferenz S® series of amylases, Purastar® and Purastar® Ox Am, Optisize® HT Plus (all Du Pont).

A suitable amylase is described in WO06/002643.

[0063] Cellulase: Suitable cellulases include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are also suitable. Suitable cellulases include cellulases from the genera *Bacillus*, *Pseudomonas*, *Humicola*, *Fusarium*, *Thielavia*, *Acremonium*, e.g., the fungal cellulases produced from *Humicola insolens*, *Myceliophthora thermophila* and *Fusarium oxysporum*.

[0064] Commercially available cellulases include Celluzyme®, Carezyme®, and Carezyme® Premium, Celluclean® and Whitezyme® (Novozymes A/S), Revitalenz® series of enzymes (Du Pont), and Biotouch® series of enzymes (AB Enzymes). Suitable commercially available cellulases include Carezyme® Premium, Celluclean® Classic. Suitable cellulases are described in WO07/144857 and WO10/056652.

[0065] Lipase: Suitable lipases include those of bacterial, fungal or synthetic origin, and variants thereof. Chemically modified or protein engineered mutants are also suitable. Examples of suitable lipases include lipases from *Humicola* (synonym *Thermomyces*), e.g., from *H. lanuginosa* (*T. lanuginosus*).

[0066] The lipase may be a "first cycle lipase", e.g. such as those described in WO06/090335 and WO13/116261. In one aspect, the lipase is a first-wash lipase, preferably a variant of the wild-type lipase from *Thermomyces lanuginosus* comprising T231R and/or N233R mutations. Preferred lipases include those sold under the tradenames Lipex®, Lipolex® and Lipoclean® by Novozymes, Bagsvaerd, Denmark.

[0067] Other suitable lipases include: Lip1 139, e.g. as described in WO2013/171241; and TfuLip2, e.g. as described in WO2011/084412 and WO2013/033318.

[0068] Other enzymes: Other suitable enzymes are bleaching enzymes, such as peroxidases/oxidases, which include those of plant, bacterial or fungal origin and variants thereof. Commercially available peroxidases include Guardzyme® (Novozymes A/S). Other suitable enzymes include choline oxidases and perhydrolases such as those used in Gentle Power Bleach™.

[0069] Other suitable enzymes include pectate lyases sold under the tradenames X-Pect®, Pectaway® (from Novozymes A/S, Bagsvaerd, Denmark) and PrimaGreen® (DuPont) and mannanases sold under the tradenames Mannaway® (Novozymes A/S, Bagsvaerd, Denmark), and Mannastar® (Du Pont).

[0070] Zeolite builder: The particle may comprise zeolite builder. The composition may comprise from 0wt% to 5wt% zeolite builder, or 3wt% zeolite builder. The composition may even be substantially free of zeolite builder; substantially free means "no deliberately added". Typical zeolite builders include zeolite A, zeolite P and zeolite MAP.

[0071] Phosphate builder: The particle may comprise phosphate builder. The composition may comprise from 0wt% to 5wt% phosphate builder, or to 3wt%, phosphate builder. The composition may even be substantially free of phosphate builder; substantially free means "no deliberately added". A typical phosphate builder is sodium tri-polyphosphate.

[0072] Carbonate salt: The particle may comprise carbonate salt. The composition may comprise from 0wt% to 10wt% carbonate salt, or to 5wt% carbonate salt. The composition may even be substantially free of carbonate salt; substantially free means "no deliberately added". Suitable carbonate salts include sodium carbonate and sodium bicarbonate.

[0073] Silicate salt: The particle may comprise silicate salt. The composition may comprise from 0wt% to 10wt% silicate salt, or to 5wt% silicate salt. A preferred silicate salt is sodium silicate, especially preferred are sodium silicates having a Na₂O:SiO₂ ratio of from 1.0 to 2.8, preferably from 1.6 to 2.0.

[0074] Sulphate salt: A suitable sulphate salt is sodium sulphate.

[0075] Brightener: Suitable fluorescent brighteners include: di-styryl biphenyl compounds, e.g. 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-2hydroxyethyl)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 C.I. Fluorescent Brightener 260, which may be used in its beta or alpha crystalline forms, or a mixture of these forms.

[0076] Chelant: The particle may also comprise a chelant selected from: diethylene triamine pentaacetate, diethylene triamine penta(methyl phosphonic acid), ethylene diamine-N'-N'-disuccinic acid, ethylene diamine tetraacetate, ethylene diamine tetra(methylene phosphonic acid) and hydroxyethane di(methylene phosphonic acid). A preferred chelant is ethylene diamine-N'-N'-disuccinic acid (EDDS) and/or hydroxyethane diphosphonic acid (HEDP). The composition preferably comprises ethylene diamine-N'-N'-disuccinic acid or salt thereof. Preferably the ethylene diamine-N'-N'-disuccinic acid is in S,S enantiomeric form. Preferably the composition comprises 4,5-dihydroxy-m-benzenedisulfonic acid disodium salt. Preferred chelants may also function as calcium carbonate crystal growth inhibitors such as: 1-hydroxyethanedi-phosphonic acid (HEDP) and salt thereof; N,N-dicarboxymethyl-2-aminopentane-1,5-dioic acid and salt thereof; 2-phosphonobutane-1,2,4-tricarboxylic acid and salt thereof; and combination thereof.

[0077] Hueing agent: Suitable hueing agents include small molecule dyes, typically falling into the Colour Index (C.I.) classifications of Acid, Direct, Basic, Reactive (including hydrolysed forms thereof) or Solvent or Disperse dyes, for example classified as Blue, Violet, Red, Green or Black, and provide the desired shade either alone or in combination. Preferred such hueing agents include Acid Violet 50, Direct Violet 9, 66 and 99, Solvent Violet 13 and any combination thereof.

[0078] Many hueing agents are known and described in the art which may be suitable for the present invention, such as hueing agents described in WO2014/089386.

[0079] Suitable hueing agents include phthalocyanine and azo dye conjugates, such as described in WO2009/069077.

[0080] Suitable hueing agents may be alkoxyated. Such alkoxyated compounds may be produced by organic synthesis that may produce a mixture of molecules having different degrees of alkoxylation. Such mixtures may be used directly to provide the hueing agent, or may undergo a purification step to increase the proportion of the target molecule. Suitable hueing agents include alkoxyated bis-azo dyes, such as described in WO2012/054835, and/or alkoxyated thiophene azo dyes, such as described in WO2008/087497 and WO2012/166768.

[0081] The hueing agent may be incorporated into the detergent composition as part of a reaction mixture which is the result of the organic synthesis for a dye molecule, with optional purification step(s). Such reaction mixtures generally comprise the dye molecule itself and in addition may comprise un-reacted starting materials and/or by-products of the organic synthesis route. Suitable hueing agents can be incorporated into hueing dye particles, such as described in WO 2009/069077.

[0082] Dye transfer inhibitors: Suitable dye transfer inhibitors include polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, polyvinylpyrrolidone, polyvinylloxazolidone, polyvinylimidazole and mixtures thereof. Preferred are poly(vinyl pyrrolidone), poly(vinylpyridine betaine), poly(vinylpyridine N-oxide), poly(vinyl pyrrolidone-vinyl imidazole) and mixtures thereof. Suitable commercially available dye transfer inhibitors include PVP-K15 and K30 (Ashland), Sokalan® HP165, HP50, HP53, HP59, HP56K, HP56, HP66 (BASF), Chromabond® S-400, S403E and S-100 (Ashland).

[0083] Perfume: Suitable perfumes comprise perfume materials selected from the group: (a) perfume materials having a ClogP of less than 3.0 and a boiling point of less than 250°C (quadrant 1 perfume materials); (b) perfume materials having a ClogP of less than 3.0 and a boiling point of 250°C or greater (quadrant 2 perfume materials); (c) perfume materials having a ClogP of 3.0 or greater and a boiling point of less than 250°C (quadrant 3 perfume materials); (d) perfume materials having a ClogP of 3.0 or greater and a boiling point of 250°C or greater (quadrant 4 perfume materials); and (e) mixtures thereof.

[0084] It may be preferred for the perfume to be in the form of a perfume delivery technology. Such delivery technologies further stabilize and enhance the deposition and release of perfume materials from the laundered fabric. Such perfume delivery technologies can also be used to further increase the longevity of perfume release from the laundered fabric. Suitable perfume delivery technologies include: perfume microcapsules, pro-perfumes, polymer assisted deliveries, molecule assisted deliveries, fiber assisted deliveries, amine assisted deliveries, cyclodextrin, starch encapsulated accord, zeolite and other inorganic carriers, and any mixture thereof. A suitable perfume microcapsule is described in WO2009/101593.

[0085] Silicone: Suitable silicones include polydimethylsiloxane and amino-silicones. Suitable silicones are described in WO05075616.

EXAMPLES

[0086] Aqueous detergent mixtures were prepared using 445L stainless steel crutcher and a 1335L drop tank vessel. The crutcher has bottom sail blade, a screw, a draft tube and a baffle which helps in the solid-liquid dispersion. The crutcher is electrically heat traced and was set to 85°C throughout the experiment. 350kg batch size was used to make the slurry composition below.

[0087] In example 1A, the sequence of addition was followed as described on the table. Water was first added into the crutcher. The crutcher mixer was turned on and set to 120 RPM. Anhydrous Magnesium Sulphate was then added followed by the other ingredients as per the table. The mixer speed was increased to 190 RPM before Sodium Sulphate was added to the batch. Additional 1-minute mixing was employed after all the Sodium Sulphate. Several batches of this formula were produced and spray dried. The resulting blown powder was free flowing with good physical properties. The composition of the resulting blown powder is shown below.

[0088] In example 1B, the sequence of addition was changed as described below. Water was added first. The crutcher mixer was turned on and set to 120RPM. C12 -14 Alkyl Benzene Sulphonate Paste was added next followed by the other ingredients as described on the table. After zeolite was added, the anhydrous Magnesium Sulphate was added next. The RPM was increased from 120 to 190 RPM. Sodium sulphate was added last. Before all the Sodium sulphate has been added, the crutcher mix overflowed and the operation was stopped and did not proceed for spray drying. The resulting mix was not the complete formula planned so the batch was scrapped while the surrounding area and equipment were cleaned.

Examples 1A and 1B- Slurry composition of Low pH composition

[0089]

Sequence of Addition							
Example 1A-Invention	Example 1B-Comparative	Material Name	RM Active	Active RM Solids	Water	Miscellaneous	% Addition
6	5	Zeolite 4A	79%	2.2959%	0.5812%	0.0291%	2.9062%
4	3	Citric acid	100%	5.1021 %	0.0000%	0.0000%	5.1021 %
5	4	Water Soluble Sodium Polyacrylate Polymer	40%	0.7041 %	1.0561%	0.0000%	1.7602%
3	2	C12 -14 Alkyl Benzene Sulphonate Paste	45%	9.1837 %	10.6122%	0.6122%	20.4082%
2	6	Anhydrous MgSO4 Powder	98%	6.1607 %	0.1257%	0.0000%	6.2864%
7	7	Na2SO4	100%	50.9122%	0.0000%	0.0000%	50.9122%
1	1	Additional Water	100%	0.0000%	12.6247%	0.0000%	12.6247%
		Total			25.0000%		100.0000%
No Overflow	Overflowed	Slurry mixture in the Crutcher					
Completed	Unable to proceed	Spray Drying					

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Low pH Blown Powder Composition

[0090]

5	Material Name	BP Composition
	SiO ₂ from Zeolite 4A	0.99
	Citric Acid	3.6467
10	Monosodium Citrate	2.9667
	Disodium Citrate	0.0533
	Water Soluble Sodium Polyacrylate Polymer	0.9200
	C12 -14 Alkyl Benzene Sulphonate Paste	12.0000
15	Anhydrous MgSO ₄ Powder	8.0500
	Na ₂ SO ₄	66.5254
	Water	2.0000
20	Miscellaneous	2.8480
	Total	100.0000

Examples of Different Sequence of Addition using simplified Crutcher mixes

25 **[0091]** Aqueous detergent mixtures were prepared using a 445L (0.445 m³) stainless steel crutcher and a 1335L (1.335m³) drop tank vessel. The crutcher has bottom sail blade, a screw, a draft tube and a baffle which helps in the solid-liquid dispersion. The crutcher is electrically heat traced and was set to 60C throughout the experiment. Batch size used for these experiments is 138.57kg to allow sufficient volume for expansion during the experiment. Water was added first into the crutcher. The mixer was then turned on to 120 RPM before adding the succeeding materials one after the other as described on the tables below. Before discharging the mixture to the drop tank, an additional 1-minute mixing was employed to ensure homogeneity of the mixture. As soon as the mixture was discharged to the receiving vessel, a sample was taken from the bottom of the receiving vessel for density measurements.

Example 2A Simplified Mixture with Modified Sequence of Addition - Invention

[0092]

40	Addition Sequence	Raw Material	Activity	Material Temperature, °C	% Addition	Quantity, kg
	1	Water	100% liquid	60	31.8800	44.1761
	2	Anhydrous MgSO ₄ Powder	98 solids%	Ambient	16.04	22.23
45	3	C12 -14 Alkyl Benzene Sulphonate Paste	45% active solids	60	52.0800	72.1673
					100.00	138.57

Example 2B - Simplified Mixture with Incorrect Sequence of Addition - Comparative

[0093]

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Addition Sequence	Raw Material	Activity	Material Temperature, °C	% Addition	Quantity, kg
1	Water	100% liquid	60	31.8800	44.1761
2	C12 -14 Alkyl Benzene Sulphonate Paste	45% active solids	60	52.0800	72.1673
3	Anhydrous MgSO ₄ Powder	98% solids	Ambient	16.0400	22.2266
				100.0000	138.5700

	Actual density of the Mixture, kg/m ³	Calculated mixture Volume, m ³	Theoretical Density, kg/m ³	% Vol Expansion
Example 2A	1032	0.1342	1173	12%
Example 2B	622	0.2228	1173	47%

$$\% \text{ Vol Expansion} = \frac{(D_t - D_a)}{D_t} \times 100$$

D_t

Where

D_a = Actual measured density, kg/m³

D_t = Theoretical Density calculated based on the following densities of materials in kg/m³

Raw Material	Activity	Absolute density, kg/m ³
C12 -14 Alkyl Benzene Sulphonate Paste	45 active solids%	1100
Water	100% liquid	1000
Anhydrous MgSO ₄ Powder	100%	2662.8

D_t = 1 / (%Weight of Water * 1000 + %Weight of C12 -14 Alkyl Benzene Sulphonate Paste * 1100 + %Weight of Anhydrous MgSO₄ Powder * 2662.8), rounded off to the nearest integer

Slurry/Paste Density measurement

[0094] The density of the aqueous mixtures was measured by taking the sample after it was transferred from crutcher to the drop tank. The sample was poured into a pre-tared cylindrical stainless-steel container with a volume of 500ml.

Using a straight edge or pallet knife and without further disturbing the samples, excess liquid was removed from the top of the container ensuring the surface of the sample on the same level as the top of the container. Sample weight is measured and density is calculated as follows

$$D = W \times 2$$

Where D= density of the mixture, g/L or kg/m³

W= weight of the sample in a 500ml container, grams

[0095] 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 spray-drying process for preparing a spray-dried laundry detergent particle comprising deterative surfactant and magnesium sulphate salt, wherein the process comprises the steps of:
 - (a) contacting magnesium sulphate to water to form a first mixture;
 - (b) contacting deterative surfactant to the first mixture to form a second mixture; and
 - (c) spray-drying the second mixture to form a spray-dried laundry detergent particle,
 wherein step (a) is carried out in the absence of deterative surfactant.
2. A process according to claim 1, wherein the first mixture comprises at least 12% wt% magnesium sulphate.
3. A process according to any preceding claim, wherein the second mixture comprises at least 2.5 wt% magnesium sulphate.
4. A process according to any preceding claim, wherein the second mixture comprises at least 6.0 wt% deterative surfactant.
5. A process according to any preceding claim, wherein the spray-dried laundry detergent particle comprises at least 2.0 wt% magnesium sulphate.
6. A process according to any preceding claim, wherein the spray-dried laundry detergent particle comprises at least 8.0 wt% deterative surfactant.
7. A process according to any preceding claim, wherein the deterative surfactant is an anionic deterative surfactant.
8. A process according to claim 7, wherein the deterative surfactant is an anionic deterative surfactant comprises alkyl-benzene sulphonate.
9. A process according to any preceding claim, wherein the spray-dried laundry detergent particle, upon dissolution in de-ionized water at a concentration of 1g/l and at a temperature of 20°C, has an equilibrium pH in the range of from 4.0 to 8.5.



EUROPEAN SEARCH REPORT

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Place of search Munich		Date of completion of the search 20 July 2018	Examiner Hillebrecht, Dieter
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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