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(54) Title: LAUNDRY DETERGENT COMPOSITION

(57) Abstract: The present invention relates to a coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein the particle comprises: (g) from 20wt% to 39wt% deterative surfactant selected from anionic deterative surfactant and/or non-ionic deterative surfactant; (h) from 10wt% to 40wt% inorganic salts selected from sodium carbonate, sodium sesquicarbonate, sodium bicarbonate and any mixtures thereof; (i) optionally, from 10wt% to 40wt% citric acid and/or salts thereof; wherein the deterative surfactant comprises from 51wt% to 100wt% alkyl benzene sulphonate, wherein the coating comprises the inorganic salt (b), and wherein the core comprises the deterative surfactant (a), wherein the alkyl benzene sulphonate has a degree of orientation of at least 92%, preferably at least 95%.



## LAUNDRY DETERGENT COMPOSITION

## FIELD OF THE INVENTION

The present invention relates to a laundry detergent composition having a lenticular size and  
5 shape, and to a process for making such lenticular detergent compositions. The lenticular laundry  
detergent compositions of the present invention, and the lenticular products made by the process  
of the present invention, exhibit improved dissolution profiles.

## BACKGROUND OF THE INVENTION

10 There is a recent trend in the field of laundry detergency to develop compositions that are  
in the form of lenticles. These solid particles are of a shape and size that exhibit improved  
physical performance characteristics compared to the conventional particulate detergent products.  
For example, dosing from the container, especially a bottle, can be improved compared to  
conventional laundry detergent powders. However the larger size of such lenticular particles  
15 means that the surface area to volume ratio is much less than for conventional detergent particles.  
Hence dissolution behaviour can be much worse unless mitigating steps are taken. The lenticular  
particles often have the form of a surfactant-rich core surrounded by a coating layer. Hence there  
is an interest in processes that can change the properties of the surfactant-rich cores through  
process conditions such as shear rate and temperature so as to increase dissolution behaviours.

20 The present invention seeks to provide a lenticular laundry detergent product having an  
improved dissolution profile and a process of making such a lenticular laundry detergent product.

## SUMMARY OF THE INVENTION

The present invention provides a coated detergent particle having perpendicular dimensions  
25 x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein  
the particle comprises:

- (a) from 20wt% to 39wt% deterative surfactant selected from anionic deterative surfactant  
and/or non-ionic deterative surfactant;
- 30 (b) from 10wt% to 40wt% inorganic salts selected from sodium carbonate, sodium  
sesquicarbonate, sodium bicarbonate and any mixtures thereof;
- (c) optionally, from 10wt% to 40wt% citric acid and/or salts thereof;

wherein the deterative surfactant comprises from 51wt% to 100wt% alkyl benzene sulphonate,

wherein the coating comprises the inorganic salt (b), and wherein the core comprises the deterative surfactant (a),

5 wherein the alkyl benzene sulphonate has a degree of orientation of at least 92%, preferably at least 95%.

#### DETAILED DESCRIPTION OF THE INVENTION

**The coated detergent particle:** The coated detergent particle has perpendicular  
10 dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein the particle comprises:

- (a) from 20wt% to 39wt% deterative surfactant selected from anionic deterative surfactant and/or non-ionic deterative surfactant;
- 15 (b) from 10wt% to 40wt% inorganic salts selected from sodium carbonate, sodium sesquicarbonate, sodium bicarbonate and any mixtures thereof;
- (c) optionally, from 10wt% to 40wt% citric acid and/or salts thereof;

20 wherein the deterative surfactant comprises from 51wt% to 100wt% alkyl benzene sulphonate,

wherein the coating comprises the inorganic salt (b), and wherein the core comprises the deterative surfactant (a),

wherein the alkyl benzene sulphonate has a degree of orientation of at least 92%, preferably at least 95%.

25 The coated detergent particle is also referred to herein as the composition.

Typically, at 1wt% dilution in de-ionised water at 20°C, the composition has a pH in the range of from 7.6 to 10.0.

Typically, at 1wt% dilution in de-ionised water at 20°C, the composition has a reserve alkalinity to pH 7.5 of greater than 3.0.

30 Typically, the composition is in the form of a coated laundry detergent particle that is curved.

Typically, the coating comprises the inorganic salt (b), and wherein the core comprises the deterative surfactant (a).

The coated laundry detergent particle may be lenticular (e.g. shaped like a whole dried lentil), an oblate ellipsoid, and where  $z$  and  $y$  are the equatorial diameters and  $x$  is the polar diameter; preferably  $y = z$ .

5 The coated laundry detergent particle may be shaped as a disc. Preferably the coated laundry detergent particle does not have hole; that is to say, the coated laundry detergent particle does not have a conduit that passes through the core: i.e. the coated detergent particle has a topologic genus of zero.

The composition may comprise from 0.05wt% to 4.0wt% soil release polymer.

The composition may comprise from 0.1wt% to 3.0wt% carboxymethylcellulose (CMC).

10 The composition may comprise from 0.1wt% to 5.0wt% calcite.

The composition may comprise from 1wt% to 10wt% carboxylate polymer.

The composition may comprise less than 10wt% total level of silicates and aluminosilicates.

The composition may comprise from 0.001wt% to 0.5wt% hueing dye.

15 The composition may comprise from 0.001wt% to 0.5wt% organic pigment and/or inorganic pigment.

The composition may comprise from 0.2 wt% to 10wt% chelant, preferably phosphonate chelant.

The composition preferably comprises from 10wt% to 40wt% sodium carbonate.

20 **Process:** The process comprises the following steps:

(i) forming a deterative mixture comprising from 40wt% to 90wt% deterative surfactant;

(ii) extruding the deterative mixture of step (i) to form an extruded material;

25 (iii) coating the extruded material with water-soluble inorganic salt in the form of an aqueous solution to form a wet coated extruded material comprising from 1wt% to 40wt% water soluble inorganic salt: and

(iv) removing water from the wet coated extruded material to form the coated detergent particle,

30 wherein, in step (ii) the material in the orifice of the extruder is subjected to a shear rate of at least  $0.033\text{ms}^{-1}$ , preferably least  $0.33\text{ms}^{-1}$ .

**Step (i):** A deterative mixture comprising from 40wt% to 90wt% deterative surfactant is formed. Typically, the deterative surfactant comprises from 15wt% to 85wt% anionic deterative surfactant and from 5wt% to 75wt% non-ionic deterative surfactant.

Typically, the anionic deterative surfactant is selected from alkyl benzene sulphonate, alkyl ether sulphate and/or alkyl sulphate.

Typically, the deterative mixture has a hardness of from 1MPa to 100MPa during the roller compaction step (ii).

A suitable deterative surfactant comprises from 80wt% to 95wt% alkyl benzene sulphonate, and from 6wt% to 10wt% 8% alkyl ethoxylated alcohol having an average degree of ethoxylation of from 5 to 9. Such a deterative surfactant can be prepared by blending the nonionic deterative surfactant into HLAS and rapidly neutralizing the mix with aqueous caustic solution followed by drying in an oven, preferably a vacuum oven until the eRH of the surfactant is less than 10%. This surfactant can then be milled in any suitable device, such as a coffee grinder or mill.

A suitable deterative surfactant comprises at least 51wt% alkyl benzenesulphonate. A suitable deterative surfactant comprises no more than 20wt% non-ionic deterative surfactant.

Typically, the deterative surfactant is dried prior to step (ii). A suitable drying means include an evaporator or drier. A suitable drying means include a wiped film evaporator, fluid bed drier and/or a tube drier.

Typically, the deterative mixture introduced into step (ii) comprises less than 1.5 wt% water.

Typically, the deterative mixture is cooled prior to step (ii). A suitable cooling means includes a chill roll.

The deterative surfactant may be transferred to a mill and milled to particles of less than 1.5 mm, preferably less than 1 mm, before it is fed to the extrusion step (ii). Typically, a powdered flow aid with a particle diameter of from 0.1µm to 10µm is added to the mill in an amount of 0.5wt% to 5 wt% (wt% based on the deterative mixture), and blended into the surfactant particles during milling.

**Step (ii):** The deterative mixture of step (i) is extruded to form an extruded material.

Typically, the extruder has a void commensurate with the size of the particle before coating.

Typically, the deterative mixture is fed into a twin-screw co-rotating extruder equipped with a die-plate and cutting mechanism.

The extruder is typically cooled during operation.

A suitable extruder is an APV 24.

Another suitable extruder includes a roller compactor.

Extrusion rates of from 2 to 8 kg/hr are suitable, and a die-plate with orifices of from 3 to 7 mm can be used. The die-plate can be equipped with a rotary cutter so as to give an extrudate thickness of from 1mm to 2mm.

5 Typically, the temperature of the deterative mixture does not exceed 45°C, and preferably does not exceed 40°C, during the extrusion step (ii).

Typically, the extruded material produced by step (ii) is an oblate spheroid.

Typically, the diameter of the extruded material produced by step (ii) is greater than 4mm

**Step (iii):** The extruded material of step (ii) is coated with water-soluble inorganic salt in  
10 the form of an aqueous solution to form a wet coated extruded material comprising from 1wt% to 40wt% water soluble inorganic salt.

Typically, the coating material is selected from powdered inorganic material and mixtures of such material and nonionic material with a melting point in the range 40°C to 90°C.

**Step (iv):** Water is removed from the wet coated extruded material to form the coated  
15 detergent particle. Typically, step (iv) is carried out in a fluidized bed and/or a drum.

Typically, the coated detergent particle comprises from 1wt% to 5wt% water.

Typically, the coated detergent particle is curved.

**Deterative surfactant:** A suitable deterative surfactant system typically comprises at least  
20 5% alcohol ether carboxylate as a percentage of the total deterative surfactant system.

A suitable deterative surfactant system typically comprises at least 5% alcohol ethoxylate  
having an average degree of ethoxylation in the range of from 10 to 50 as a percentage of the  
total deterative surfactant system.

Preferably, the deterative surfactant comprises C<sub>8</sub>-C<sub>24</sub> alkyl ethoxylated alcohol having an  
average degree of ethoxylation of from 20 to 50, and preferably the composition comprises from  
25 1wt% to 10wt% C<sub>8</sub>-C<sub>24</sub> alkyl ethoxylated alcohol having an average degree of ethoxylation of  
from 20 to 50. A suitable highly ethoxylated alcohol is Lutensol® AO30 from BASF and/or  
Slovasol® 2430 from Sasol.

**Anionic deterative surfactant:** Suitable anionic deterative surfactants include sulphonate  
and sulphate deterative surfactants.

30 Suitable sulphonate deterative surfactants include methyl ester sulphonates, alpha olefin  
sulphonates, alkyl benzene sulphonates, especially alkyl benzene sulphonates, preferably C<sub>10-13</sub>  
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®.

Suitable sulphate deterative surfactants include alkyl sulphate, preferably C<sub>8-18</sub> alkyl sulphate, or predominantly C<sub>12</sub> alkyl sulphate.

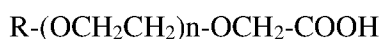
5 A preferred sulphate deterative surfactant is alkyl alkoxyated sulphate, preferably alkyl ethoxyated sulphate, preferably a C<sub>8-18</sub> alkyl alkoxyated sulphate, preferably a C<sub>8-18</sub> alkyl ethoxyated sulphate, preferably the alkyl alkoxyated sulphate has an average degree of alkoxyation of from 0.5 to 20, preferably from 0.5 to 10, preferably the alkyl alkoxyated sulphate is a C<sub>8-18</sub> alkyl ethoxyated sulphate having an average degree of ethoxyation of from 10 0.5 to 10, preferably from 0.5 to 5, more preferably from 0.5 to 3 and most preferably from 0.5 to 1.5.

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

15 Other suitable anionic deterative surfactants include alkyl ether carboxylates.

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

**Alkyl ether carboxylic acid:** A suitable alkyl ether carboxylic acid has the following 20 structure:



wherein,

R is selected from saturated and mono-unsaturated C<sub>10</sub> to C<sub>26</sub> linear or branched alkyl chains, preferably C<sub>12</sub> to C<sub>24</sub> linear or branched alkyl chains, most preferably a C<sub>16</sub> to C<sub>20</sub> linear alkyl 25 chain;

n is selected from 5 to 20, preferably 7 to 13, more preferably 8 to 12, most preferably 9.5 to 10.5; and

The alkyl ether carboxylic acid may be present from 0.5 to 20 wt%, preferably from 2 to 14 wt%, most preferably from 2.5 to 5 wt%. It may be present in acid or salt form, most preferably 30 as its sodium salt.

Suitable materials are sold under the AKYPO® (Kao) and Empicol® C (Huntsman) brand names.

**Non-ionic detergent surfactant:** Suitable non-ionic detergent surfactants are selected from the group consisting of: C<sub>8</sub>-C<sub>18</sub> alkyl ethoxylates, such as, NEODOL® non-ionic surfactants from Shell; C<sub>6</sub>-C<sub>12</sub> alkyl phenol alkoxyates wherein preferably the alkoxyate units are ethyleneoxy units, propyleneoxy units or a mixture thereof; C<sub>12</sub>-C<sub>18</sub> alcohol and C<sub>6</sub>-C<sub>12</sub> alkyl phenol condensates with ethylene oxide/propylene oxide block polymers such as Pluronic® from BASF; alkylpolysaccharides, preferably alkylpolyglycosides; methyl ester ethoxylates; polyhydroxy fatty acid amides; ether capped poly(oxyalkylated) alcohol surfactants; and mixtures thereof.

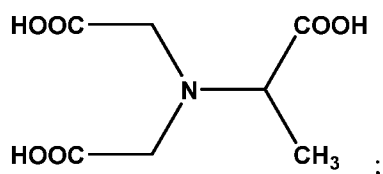
Suitable non-ionic detergent surfactants are alkylpolyglucoside and/or an alkyl alkoxyated alcohol.

Suitable non-ionic detergent surfactants include alkyl alkoxyated alcohols, preferably C<sub>8-18</sub> alkyl alkoxyated alcohol, preferably a C<sub>8-18</sub> alkyl ethoxylated 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 C<sub>8-18</sub> alkyl ethoxylated alcohol having an average degree of ethoxylation of from 1 to 10, preferably from 1 to 7, more preferably from 1 to 5 and most preferably from 3 to 7. The alkyl alkoxyated alcohol can be linear or branched, and substituted or un-substituted.

Suitable nonionic detergent surfactants include secondary alcohol-based detergent surfactants.

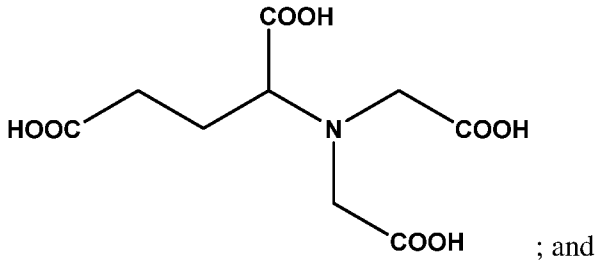
**Amino acid derivative complexing agent:** A suitable amino acid derivative complexing agent is selected from one or more of the following, in any stereoisomer or mixture of stereoisomer form:

(i) methylglycinediacetic acid and salts thereof (MGDA)

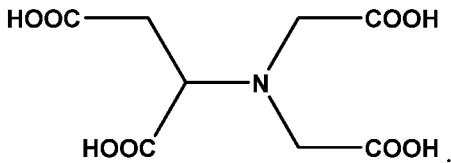


(ii) L-glutamic acid, N,N-diacetic acid and salts thereof (GLDA)

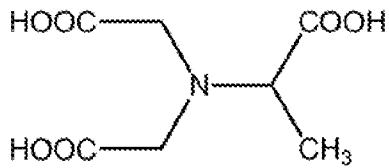




(iii) L-aspartic acid N,N-diacetic acid and salts thereof (ASDA)



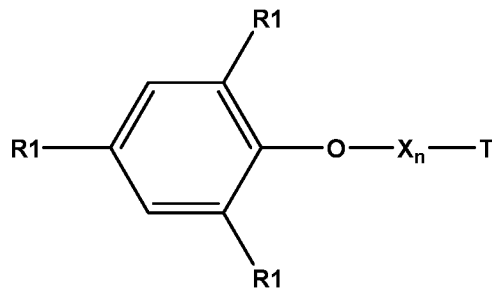
Preferably, the composition comprises from 0.1wt% to 10wt% methylglycinediacetic acid  
5 and salts thereof (MGDA)



It may be preferred to formulate the amino acid derivative complexing agent in acid form. Alternatively, it may be preferred to formulate the amino acid derivative complexing agent in salt form, especially preferred is the sodium salt form.

10 Suitable MGDA salts are produced by BASF. Suitable GLDA salts are produced by Akzo Nobel and Showa Denko. Suitable ASDA salts are produced by Mitsubishi Rayon.

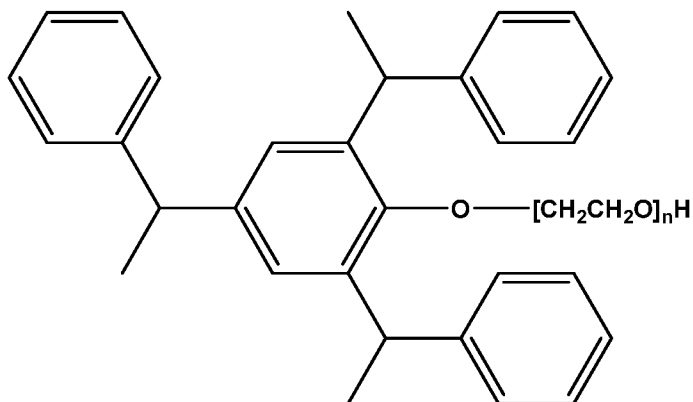
**Alkoxylated polyaryl/polyalkyl phenol:** A suitable alkoxylated polyaryl/polyalkyl phenol has the following structure:



wherein  $R_1$  is selected from linear or branched  $C_3$ - $C_{15}$  alkyl groups and aryl groups, X is selected from ethoxy or propoxy groups, n is from 2 to 70, T is selected from H,  $SO_3^-$ ,  $COO^-$  and  $PO_3^{2-}$

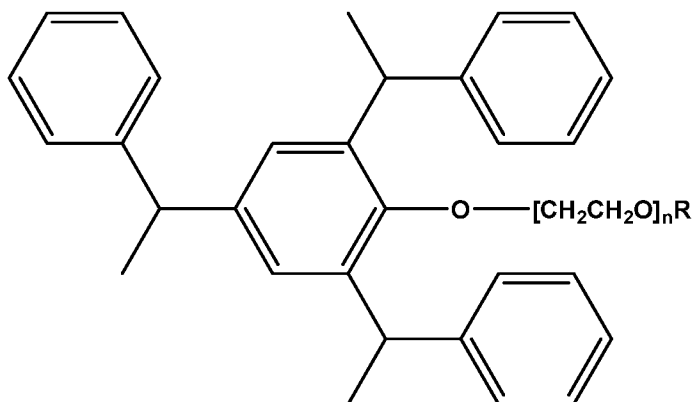
The alkoxyated polyaryl or alkoxyated polyalkyl phenol is preferably selected from groups (i) to (iv):

- 5 (i) Uncharged alkoxyated tristyrylphenols of the following structure:



wherein n is selected from 2 to 70, more preferably n is selected from 10 to 54, most preferably n = 16 or 20.

- (ii) Anionic alkoxyated tristyrylphenols of the following structure

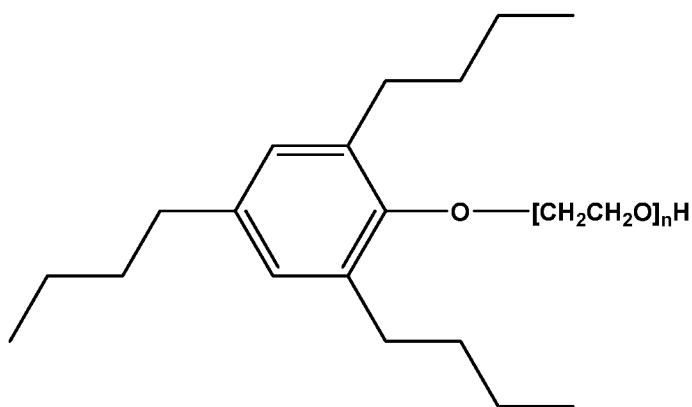


10

wherein R is selected from  $SO_3^-$ ,  $COO^-$  and  $PO_3^{2-}$ , preferably selected from  $SO_3^-$  and  $COO^-$ , wherein n is selected from 2 to 54.

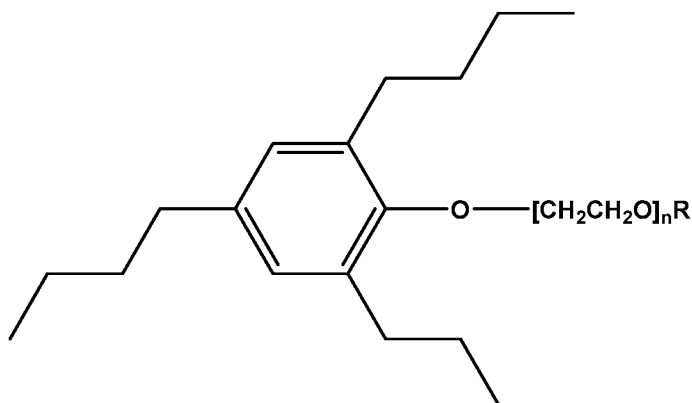
- (iii) Uncharged alkoxyated tri(n-butyl)phenols of the following structure:

10



wherein n is selected from 2 to 50

(iv) Anionic alkoxyated tri(n-butyl)phenols of the following structure:



- 5 wherein R is selected from  $\text{SO}_3^-$ ,  $\text{COO}^-$  and  $\text{PO}_3^{2-}$ , preferably selected from  $\text{SO}_3^-$  and  $\text{COO}^-$ , wherein n is selected from 6 to 50.

Such compounds are available from industrial suppliers, for example Solvay under the Soprophor trade name, from Clariant under the Emulsogen trade name, Aoki Oil Industrial Co. under the Blaunon trade name, from Stepan under the Makon trade name, and from TOTO  
 10 Chemical Industry Co. under the Sorpol trade name. Specific examples of suitable compounds are Emulsogen® TS160, Hostapal® BV conc., Sapogenat® T110 or Sapogenat® T139, all from Clariant.

The alkoxyated polyaryl/polyalkyl phenol may be present at levels of 0.5-20wt%, preferably 1-15wt%, most preferably 3-10wt%.

- 15 **Amylase variant:** A suitable amylase variant comprises:

(a) a deletion and/or a substitution at two or more positions corresponding to positions R181 , G182, H183 and G184 of the mature polypeptide of SEQ ID NO: 1 , and

(b) a substitution at one or more positions said substitutions selected from the group consisting of:

- 5 I206Y;F;Q;P;R;V;C;G;A;C;D;E;H;K;L;M;N;S;T, particularly I206Y;F;C;L;H;S,  
N195F;Y;H;K;L,  
L63Q;P;R;V;F;C;G;A;C;D;E;H;K;I;M;N;S;T;Y, particularly L63V,  
A113M;R;W;I;L,  
M116F;Y;I;W;L,
- 10 R118P;Q;V;F;C;G;A;C;D;E;H;I;K;L;M;N;S;T;Y, particularly R118P;Q;V;F;C;G,  
N128C;  
Q129P;R;V;F;C;G;A;C;D;E;H;I;K;L;M;N;S;T;Y, particularly Q129E,  
G133N,  
A139Q;P;R;V;F;C;G;C;D;E;H;I;K;L;M;N;S;T;Y, particularly A139T,
- 15 R142H;V;L;Q;I,  
A186E;N;Q;S,  
E190P;R;V;F;C;G;A;C;D;Q;H;I;K;L;M;N;S;T;Y, particularly E190P,  
A204Q;P;R;V;F;C;G;C;D;E;H;I;K;L;M;N;S;T;Y, particularly A204T,  
H210M;D;C;A;Q;S;F;N;E;T,
- 20 P211Q;R;V;F;C;G;A;C;D;E;H;I;K;L;M;N;S;T;Y, particularly P211L;M;S;Q;G;V;W;A;H;T;R;  
E212T;R;S;V;L;Y;R;T;G;  
V213Q;P;R;F;C;G;A;C;D;E;H;I;K;L;M;N;S;T;Y, particularly V213T;A;G;S;C;L;P,

V214Q;P;R;F;C;G;A;C;D;E;H;I;K;L;M;N;S;T;Y, particularly V214T;I,

L217M;Q;V;I;H, particularly L217V,

Y243Q;P;R;F;C;G;A;C;D;E;H;I;K;L;M;N;S;T;V, particularly Y243F,

S244Q;P;R;F;C;G;A;C;D;E;H;I;K;L;M;N;Y;T;V, particularly S244Q,

5 T246Q;P;R;F;C;G;A;C;D;E;H;I;K;L;M;N;Y;S;V, particularly T246Q;M,

N260E, Q280S, N311R, F343W, D418C, S419M, S420Q;R and Y482W,

where the positions correspond to the positions of SEQ ID NO 1 and wherein the amylase variant has at least 95%, such as at least 96%, or at least 97%, or at least 98%, or at least 99% but less than 100% sequence identity to the polypeptide having the amino acid sequence of SEQ ID NO:

10 1 and wherein the variant has alpha-amylase activity.

One preferred amylase variant comprises a sequence corresponding to SEQ ID NO: 1 with the following mutations: H183\*+G184\*+I405L+A421H+A422P+A428T.

A suitable amylase is commercially available from Novozymes under the Amplify® brand name, for example as a liquid raw material as Amplify® 12L.

15 **Lipase:** A suitable lipase is a variant of SEQ ID NO:2 comprising:

(a) substitutions T231R and N233R

and

(b) at least three further substitutions selected from D27R, N33Q, G38A, D96E, D111A, G91Q, G163K, E210Q, D254S, I255A, and P256T;

20 where the positions correspond to the positions of SEQ ID NO 2 and wherein the lipase variant has at least 95% but less than 100% sequence identity to the polypeptide having the amino acid sequence of SEQ ID NO: 2 and wherein the variant has lipase activity.

One preferred lipase is a variant of SEQ ID NO: 2 comprising the following substitutions: T231R, N233R, D27R, G38A, D96E, D111A, G163K, D254S and P256T

25 One preferred lipase is a variant of SEQ ID NO: 2 comprising the following substitutions: T231R, N233R, N33Q, G91Q, E210Q, I255A.

Suitable lipases are commercially available from Novozymes, for example as Lipex Evity 100L (a liquid raw material) and Lipex Evity 105T (a granulate). These lipases have different structures to the products Lipex 100L, Lipex 100T and Lipex Evity 100T which are outside the scope of this particular lipase definition.

5       **Metalloprotease:** Metalloproteases can be derived from animals, plants, bacteria or fungi. Suitable metalloprotease can be selected from the group of neutral metalloproteases and *Myxobacter* metalloproteases. Suitable metalloproteases can include collagenases, hemorrhagic toxins from snake venoms and thermolysin from bacteria.

10       Preferred thermolysin enzyme variants include an M4 peptidase, more preferably the thermolysin enzyme variant is a member of the PepSY~Peptidase\_M4~Peptidase\_M4\_C family.

Suitable metalloprotease variants can have at least 50% identity to the thermolysin set forth in SEQ ID NO: 3. In some embodiments, the thermolysin enzyme variant is from a genus selected from the group consisting of *Bacillus*, *Geobacillus*, *Alicyclobacillus*, *Lactobacillus*, *Exiguobacterium*, *Brevibacillus*, *Paenibacillus*, *Herpetosiphon*, *Oceanobacillus*, *Shewanella*,  
 15 *Clostridium*, *Staphylococcus*, *Flavobacterium*, *Stigmatella*, *Myxococcus*, *Vibrio*, *Methanosarcina*, *Chryseobacterium*, *Streptomyces*, *Kribbella*, *Janibacter*, *Nocardioides*, *Xanthamonas*, *Micromonospora*, *Burkholderia*, *Dehalococcoides*, *Croceibacter*, *Kordia*, *Microscilla*, *Thermoactinomyces*, *Chloroflexus*, *Listeria*, *Plesiocystis*, *Haliscomenobacter*, *Cytophaga*, *Hahella*, *Arthrobacter*, *Brachybacterium*, *Clavibacter*, *Microbacterium*, *Intrasporangium*,  
 20 *Frankia*, *Meiothermus*, *Pseudomonas*, *Ricinus*, *Catenulispora*, *Anabaena*, *Nostoc*, *Halomonas*, *Chromohalobacter*, *Bordetella*, *Variovorax*, *Dickeya*, *Pectobacterium*, *Citrobacter*, *Enterobacter*, *Salmonella*, *Erwinia*, *Pantoea*, *Rahnella*, *Serratia*, *Geodermatophilus*, *Gemmata*, *Xenorhabdus*, *Photorhabdus*, *Aspergillus*, *Neosartorya*, *Pyrenophora*, *Saccharopolyspora*, *Nectria*, *Gibberella*, *Metarhizium*, *Waddlia*, *Cyanothece*, *Cellulphaga*, *Providencia*, *Bradyrhizobium*, *Agrobacterium*,  
 25 *Mucilaginibacter*, *Serratia*, *Sorangium*, *Streptosporangium*, *Renibacterium*, *Aeromonas*, *Reinekea*, *Chromobacterium*, *Moritella*, *Haliangium*, *Kangiella*, *Marinomonas*, *Vibrionales*, *Listonella*, *Salinivibrio*, *Photobacterium*, *Alteromonadales*, *Legionella*, *Teredinibacter*, *Reinekea*, *Hydrogenivirga* and *Pseudoalteromonas*. In some embodiments, the thermolysin enzyme variant is from a genus selected from the group consisting of *Bacillus*,  
 30 *Geobacillus*, *Alicyclobacillus*, *Lactobacillus*, *Exiguobacterium*, *Brevibacillus*, *Paenibacillus*, *Herpetosiphon*, *Oceanobacillus*, *Shewanella*, *Clostridium*, *Staphylococcus*, *Flavobacterium*, *Stigmatella*, *Myxococcus*, *Vibrio*, *Methanosarcina*, *Chryseobacterium*, and *Pseudoalteromonas*.

Preferably the thermolysin enzyme is from the genus *Bacillus*.

Preferred metalloproteases include thermolysin, matrix metalloproteinases and those metalloproteases derived from *Bacillus subtilis*, *Bacillus thermoproteolyticus*, *Geobacillus stearothermophilus* or *Geobacillus sp.*, or *Bacillus amyloliquefaciens*, as described in US PA 2008/0293610A1.

5 A specially preferred metalloprotease belongs to the family EC3.4.24.27.

Further suitable metalloproteases are the thermolysin variants described in WO2014/71410.

In one aspect the metalloprotease is a variant of a parent protease, said parent protease having at least 60%, or 80%, or 85% or 90% or 95% or 96% or 97% or 98% or 99% or even 100% identity to SEQ ID NO:3 including those with substitutions at one or more of the following

10 sets of positions versus SEQ ID NO:3:

(a) 2, 26, 47, 53, 87, 91, 96, 108, 118, 154, 179, 197, 198, 199, 209, 211, 217, 219, 225, 232, 256, 257, 259, 261, 265, 267, 272, 276, 277, 286, 289, 290, 293, 295, 298, 299, 300, 301, 303, 305, 308, 311 and 316;

(b) 1, 4, 17, 25, 40, 45, 56, 58, 61, 74, 86, 97, 101, 109, 149, 150, 158, 159, 172, 181, 15 214, 216, 218, 221, 222, 224, 250, 253, 254, 258, 263, 264, 266, 268, 271, 273, 275, 278, 279, 280, 282, 283, 287, 288, 291, 297, 302, 304, 307 and 312;

(c) 5, 9, 11, 19, 27, 31, 33, 37, 46, 64, 73, 76, 79, 80, 85, 89, 95, 98, 99, 107, 127, 129, 131, 137, 141, 145, 148, 151, 152, 155, 156, 160, 161, 164, 168, 171, 176, 180, 182, 187, 188, 205, 206, 207, 210, 212, 213, 220, 227, 234, 235, 236, 237, 242, 244, 246, 20 248, 249, 252, 255, 270, 274, 284, 294, 296, 306, 309, 310, 313, 314 and 315;

(d) 3, 6, 7, 20, 23, 24, 44, 48, 50, 57, 63, 72, 75, 81, 92, 93, 94, 100, 102, 103, 104, 110, 117, 120, 134, 135, 136, 140, 144, 153, 173, 174, 175, 178, 183, 185, 189, 193, 201, 223, 230, 238, 239, 241, 247, 251, 260, 262, 269, and 285;

(e) 17, 19, 24, 25, 31, 33, 40, 48, 73, 79, 80, 81, 85, 86, 89, 94, 109, 117, 140, 141, 150, 25 152, 153, 158, 159, 160, 161, 168, 171, 174, 175, 176, 178, 180, 181, 182, 183, 189, 205, 206, 207, 210, 212, 213, 214, 218, 223, 224, 227, 235, 236, 237, 238, 239, 241, 244, 246, 248, 249, 250, 251, 252, 253, 254, 255, 258, 259, 260, 261, 262, 266, 268, 269, 270, 271, 272, 273, 274, 276, 278, 279, 280, 282, 283, 294, 295, 296, 297, 300, 302, 306, 310 and 312;

(f) 1, 2, 127, 128, 180, 181, 195, 196, 197, 198, 199, 211, 223, 224, 298, 299, 300, and 30 316

all relative to SEQ ID NO:3.

In a further aspect the metalloprotease protease is a variant of a parent protease, said parent protease having at least 60%, or 80%, or 85% or 90% or 95% or 96% or 97% or 98% or 99% or

even 100% identity to SEQ ID NO:3 including those with substitutions at one or more of the following sets of positions versus SEQ ID NO:3:

- (a) I001L, T002A, T002C, T002I, T002K, T002M, T004K, T004L, T004M, T004Y, Q017L, N037K, F040K, F040L, K045A, K045G, K045M, T049E, T049M, T049Y, L050P, S053C, S053L, A056M, A058E, A058L, Q061L, F063C, A064D, A064E, S065A, S065D, S065E, S065P, S065Y, V087C, V087K, V087L, V087M, V087N, V087Q, V087W, V087Y, N096K, N096L, N096Y, R101H, Q108L, Q108M, G109E, G109M, G109R, G109W, S118A, S118D, S118M, S118Q, S118R, S118T, SI 18V, Q128A, Q128L, Q128Y, I131L, I137L, T149N, G154A, G154H, G154K, G154M, G154Y, L155M, I164A, N181S, G196A, G196W, I197C, S198A, S198K, G199A, G199Y, A209C, A209M, H216A, Y217C, Y217L, T222K, N227A, I244L, Q246D, V256N, L263A, L263M, T272K, Q273N, Y274M, P277A, P277D, P277Y, L284A, L284M, L284Y, A286K, A286L, A286M, A286N, A286Y, A287C, A288L, A288M, V289A, S291A, S291T, T293A, T293I, T293K, T293L, T293M, T293Y, L295A, L295K, L295M, L295W, Y296M, G297N, S298A, S298G, S298K, S298M, S298R, T299A, T299K, S300D, S300N, Q301K, E302A, V303A, V303P, V303Y, A304E, A304K, A304Y, S305A, S305K, S305M, V306L, V306T, A309C, F310M, D311A, D311K, D311L, D311M, D311V, D311W, D311Y, and A312C;
- (b) T002Q, T004V, V007I, V009I, R01 IK, I020L, I020V, S025A, S025C, S025K, S025M, S025R, T026C, T026D, Y027C, Y027L, N037L, F040A, A044C, K045F, K045H, K045Q, K045Y, Y046C, R047D, R047E, R047G, R047L, R047M, R047Q, R047T, T049L, T049N, T049Q, T049V, S053A, S053N, S053V, A056E, Q061C, Q061I, A064T, S065L, S065T, S065W, A073F, A073L, A073M, A073W, H074C, H074F, H074M, H074N, H074Q, H074W, T080L, T080N, K085S, N086D, V087R, V087T, L091A, L091N, L091R, L091W, L091Y, S092L, Y093C, N096G, N096H, N096Q, N096R, N096S, N096W, N097E, N097M, A099R, A099S, R101C, R101L, R101S, S102N, S107G, Q108I, Q108K, Q108N, G109S, S118E, M120L, Q128I, Q128K, T129L, T129M, I131W, S134P, G136S, I137E, I137T, I137V, V140D, V148A, V148Q, T149D, T149S, T152G, G154C, G154N, L155I, N159S, N159Y, I164C, I168L, I171G, Y179F, A180S, G189A, Y193F, G196H, G196L, G196Y, I197F, S198M, S198N, S198R, S198W, S201A, A209G, A209I, A209K, A209P, A209R, A209Y, Y211E, Y211R, P214A, P214R, Y217A, Y217F, Y217M, Y217N, K219A, K219E, K219R, K219S, R220A, Y221A, Y221F, Y221G, Y221M, T222A, T222M, Q225C, Q225E, Q225K, Q225L, Q225S, I232L, I232R, I232S, I232T,



I232V, I232Y, S234A, S234C, G235A, I236C, I244A, I244M, Q246C, V256S,  
 G257K, G257R, I258A, I258C, I258K, I258Q, I258V, G259N, G259S, G259T,  
 L263H, L263K, L263N, L263V, G264A, G264N, G264P, G264Q, G264S, G264T,  
 K265N, I266C, I266M, I266T, I266V, F267A, F267C, F267H, F267I, F267K,  
 5 F267L, F267M, F267T, F267Y, R269K, A270G, L271H, T272A, Q273E, Q273G,  
 L275C, L275Q, L275S, L275T, T276A, T276L, T276V, T276Y, P277E, P277F,  
 P277G, P277H, P277N, P277R, P277V, P277W, S279G, R285Y, A286C, A286Q,  
 A286R, A286T, A288N, V289L, V289M, V289Y, Q290A, Q290H, Q290N, S291V,  
 T293N, T293V, T293W, D294N, L295F, L295G, Y296W, G297D, S298E, S298N,  
 10 S298P, T299N, S300A, S300G, S300T, Q301M, Q301S, Q301T, Q301V, E302D,  
 E302Q, V303G, V303K, V303L, V303R, V303W, A304R, A304S, A304T, A304W,  
 S305H, S305T, S305V, V306I, Q308A, Q308L, F310C, F310W, D311F, D311G,  
 D311I, D311Q, D311S, D311T, V313C, G314Q, V315L, V315T, K316A, and  
 K316M;

15 (c) I001K, I001M, I001V, T002F, T002L, T002P, T002S, T002V, T002W, T002Y,  
 T004E, S005D, S005N, S005P, T006C, R011I, Q017I, Q017W, Q017Y, S025D,  
 S025F, T026K, T026L, T026R, T026V, T026Y, Y027W, Q031A, Q031K, Q031V,  
 N033S, N033T, N037D, N037Q, N037R, F040E, F040G, F040M, F040Q, F040S,  
 F040Y, K045E, K045L, K045S, Y046L, R047A, R047C, R047H, R047K, R047N,  
 20 T048E, T049A, T049D, T049F, T049H, T049I, T049S, S053F, S053H, S053I,  
 S053M, S053Q, S053T, S053W, A056K, A056Q, A056V, A056W, Q061M, S065I,  
 S065M, S065Q, S065V, D072F, H074E, H074L, Y076H, Y076L, Y076M, Y076Q,  
 V079L, V079Q, V079T, T080I, Y081F, K085E, N086L, N086S, V087D, V087E,  
 V087G, V087I, V087S, L091D, L091E, L091F, L091K, L091M, L091P, L091Q,  
 25 L091S, Y093T, G095A, G095D, G095H, G095M, G095N, G095S, N096C, N096D,  
 N096I, N096V, N097K, A098C, A098E, A098H, A098R, A099E, A099K, A099P,  
 S107D, Q108C, Q108E, Q108F, Q108H, G127C, G127D, G127E, Q128C, Q128D,  
 Q128E, Q128R, Q128S, T129I, T129R, S134A, I137P, A141S, T145A, T145C,  
 T145E, T145G, T145M, T145N, T145Q, V148L, V148N, V148Y, T149M, T149V,  
 30 Y151K, T152S, A153T, G154L, G154Q, G154S, G154T, L155C, Q158A, Q158K,  
 Q158M, Q158N, N159R, N159W, S161A, S161N, S161P, S161T, I164L, I164N,  
 I164S, I164T, I164V, I171C, I171E, I171F, I171L, I171S, F172G, F172L, F172M,  
 F172Q, F172S, F172V, F172W, F172Y, G173A, G173C, T174C, V176L, V176N,  
 N181L, G196D, G196E, G196T, I197D, I197K, I197L, I197T, I197V, I197W,

I197Y, S198C, S198E, S198F, S198G, S198H, S198I, S198P, S198Q, S198T,  
S198V, G199C, G199E, G199F, G199H, G199Q, G199S, G199T, G199W, M205L,  
A209D, A209E, A209L, A209S, A209T, A209V, Y211A, Y211C, Y211D, Y211F,  
Y211G, Y211H, Y211I, Y211L, Y211N, Y211Q, Y211S, Y211T, D213N, D213S,  
5 P214C, P214G, P214K, P214S, H216C, H216E, H216S, H216T, Y217Q, Y217S,  
Y217T, Y217V, Y217W, S218K, S218L, S218Y, K219D, K219F, K219G, K219H,  
K219I, K219M, K219N, K219Q, K219T, R220K, R220V, Y221K, Y221N, Y221Q,  
Y221R, Y221S, Y221T, Y221V, T222C, T222D, T222L, T222Y, T224K, T224M,  
Q225D, Q225G, Q225H, Q225I, Q225P, Q225V, Q225W, I232C, I232E, I232F,  
10 I232K, I232M, I232N, I232Q, I232W, S234D, G235M, I236M, Y242C, Y242F,  
Y242N, Y242V, I244T, I244V, Q246E, Q246N, Q246T, G247A, G247S, T249K,  
T249M, T249N, H250A, H250C, G252K, G252Y, V253N, V253T, S254A, S254M,  
S254R, S254Y, V255L, V255P, V256L, V256T, G257C, G257D, G257E, G257L,  
G257N, G257P, G257Q, G257S, G257T, G257Y, I258E, I258L, I258M, I258N,  
15 G259A, G259C, G259E, G259F, G259H, G259L, G259M, G259W, D261A, D261N,  
L263C, L263I, L263Q, L263T, K265A, K265C, K265D, K265M, K265P, K265Q,  
K265S, I266A, I266F, I266L, I266S, F267E, F267G, F267N, F267S, F267V,  
F267W, Y268M, Y268Q, Y268V, A270C, A270F, A270I, A270L, A270S, L271A,  
L271D, L271F, L271I, T272E, T272L, T272V, T272W, Q273A, Q273H, Q273Y,  
20 Y274F, Y274H, L275I, L275M, L275V, T276C, T276F, T276I, T276P, T276Q,  
T276W, P277Q, P277S, P277T, T278G, S279A, S279D, S279I, S279L, S279M,  
S279N, S279Q, S279T, N280A, N280C, N280D, N280E, S282K, S282N, L284V,  
L284W, R285K, A286D, A286E, A286F, A286G, A286H, A286I, A286S, A287I,  
A287L, A287N, A287V, A287Y, A288C, A288I, A288S, A288T, A288V, V289C,  
25 V289E, V289F, V289G, V289I, V289N, V289S, V289W, Q290C, Q290D, Q290F,  
Q290G, Q290L, Q290W, S291E, T293C, T293E, T293F, T293G, T293H, T293Q,  
T293S, L295C, L295I, L295N, Y296N, G297A, G297M, G297R, G297Y, S298C,  
S298T, S298W, S298Y, T299C, T299F, T299L, T299M, T299R, T299W, S300C,  
S300K, S300M, S300R, S300Y, Q301E, Q301H, Q301P, Q301R, V303C, V303H,  
30 A304C, A304D, A304L, A304N, S305G, S305I, S305L, S305N, S305W, S305Y,  
V306A, V306S, K307A, K307C, K307G, K307I, K307M, K307N, K307Q, K307R,  
K307W, K307Y, Q308C, Q308D, Q308F, Q308G, Q308I, Q308M, A309G, A309S,  
D311C, D311E, A312G, A312M, A312V, V313T, G314A, G314E, G314H, G314M,  
G314S, G314W, V315A, V315C, V315I, V315M, K316D, K316E, K316F, K316G,

K316H, K316L, K316N, K316P, K316Q, K316R, K316S, K316V, K316W and K316Y.

Further suitable metalloproteases are the NprE variants described in WO2007/044993, WO2009/058661 and US 2014/0315775.

5 In one aspect the protease is a variant of a parent protease, said parent protease having at least 45%, or 60%, or 80%, or 85% or 90% or 95% or 96% or 97% or 98% or 99% or even 100% identity to SEQ ID NO:4 including those with substitutions at one or more of the following sets of positions versus SEQ ID NO:4:

S23, Q45, T59, S66, S129, F130, M138, V190, S199, D220, K211, and G222,

10 Another suitable metalloprotease is a variant of a parent protease, said parent protease having at least 60%, or 80%, or 85% or 90% or 95% or 96% or 97% or 98% or 99% or even 100% identity to SEQ ID NO:4 including those with substitutions at one or more of the following sets of positions versus SEQ ID NO:4:

Q45E, T59P, S66E, S129I, S129V, F130L, M138I, V190I, S199E, D220P, D220E, K211V,

15 K214Q, G222C, M138L/D220P, F130L/D220P, S129I/D220P, V190I/D220P, M138L/V190I/D220P, S129I/V190I, S129V/V190I, S129V/D220P, S129I/F130L/D220P, T004V/S023N, T059K/S66Q/S129I, T059R/S66N/S129I, S129I/F130L/M138L/V190I/D220P and T059K/S66Q/S129V.

Especially preferred metalloproteases for use herein belong to EC classes EC 3.4.22 or EC3.4.24, more preferably they belong to EC classes EC3.4.22.2, EC3.4.24.28 or EC3.4.24.27. The most preferred metalloprotease for use herein belong to EC3.4.24.27.

Suitable commercially available metalloprotease enzymes include those sold under the trade names Neutrase® by Novozymes A/S (Denmark), the Corolase® range including Corolase® 2TS, Corolase® N, Corolase® L10, Corolase® LAP and Corolase® 7089 from AB  
25 Enzymes, Protex 14L and Protex 15L from DuPont (Palo Alto, California), those sold as thermolysin from Sigma and the Thermoase range (PC10F and C100) and thermolysin enzyme from Amano enzymes.

A preferred metalloprotease is selected from the M4 Metalloprotease Family.

**Builder system:** A suitable water-soluble builder system comprising one or more  
30 aminocarboxylates, selected from: methylglycine diacetic acid (MGDA) and/or alkali metal or ammonium salts thereof; N,N-dicarboxymethyl glutamic acid (GLDA) and/or alkali metal or ammonium salts thereof; Aspartic acid N,N-diacetic acid (ASDA) and/or alkali metal or ammonium salts thereof; Ethylene diamine-N,N'-disuccinic acid (EDDS) and/or alkali metal or ammonium salt thereof; 2-hydroxy propylene diamine-N,N'-disuccinic acid (HPDDS), and/or

alkali metal or ammonium salt thereof; ethylenediamine-N,N'-diglutamic acid (EDDG and/or alkali metal or ammonium salt thereof; ethylenediamine-N,N'-bis-(orthohydroxyphenyl)acetic acid (EDDHA) and/or alkali metal or ammonium salt thereof; N-hydroxyethyl ethylenediamine-N,N',N'-triacetic acid (HEDTA) alkali metal or ammonium salts thereof; iminodisuccinate, hydroxyethyl iminodiacetate, and ethylene iminodisuccinate and the respective alkali metal or ammonium salts; and any combination thereof.

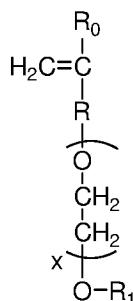
**Phosphonate chelant:** A suitable phosphonate chelant is selected from: 1-hydroxyethane-1,1-diphosphonic acid (HEDP); Diethylene triamine pentamethylene phosphonic acid (DTPMP, CW-Base); 2-phosphonobutane-1,2,4-tricarboxylic acid (PBTC); Amino trimethylene phosphonic acid (ATMP); Ethylenediamine tetramethylene phosphonic acid (EDTMP); Diethylenetriamine pentamethylene phosphonic acid (DTPMP); Aminotrimethylene phosphonic acid (ATMP); salts of the aforementioned materials; and any combination thereof.

**Carboxylate polymer:** The composition may comprise a carboxylate polymer, such as a maleate/acrylate random copolymer, maleic-olefin copolymers 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. Examples of the foregoing include Acusol 410N, Acusol 445N (polyacrylic acid, Na salt); Acusol 450N and Acusol 480N (modified polyacrylic acid, Na salt); Acusol 479N, Acusol 490N, and Acusol 505N (acrylic acid/maleic acid, Na salt); Acusol 460N (maleic acid/olefin, Na salt); Sokolan CP5 and Sokolan CP12S (maleic acid/acrylic acid, Na salt); and Sokolan CP 9 (maleic acid/olefin, Na salt). The Acusol series are available from Rohm & Haas, Philadelphia, PA and the Sokolan series are available from BASF (Germany and New Jersey).

Suitable carboxylate polymers can contain other monomers including modified acrylic, fumaric, maleic, itaconic, aconitic, mesaconic, citraconic and methylenemalonic acid or their salts, modified maleic anhydride, acrylamide, alkylene, vinylmethyl ether, styrene and any mixtures thereof. Suitable carboxylate polymers can also containing 2-acrylamido-2-methyl-1-propanesulfonic acid, 2-methacrylamido-2-methyl-1-propanesulfonic acid, 3-methacrylamido-2-hydroxy-propanesulfonic acid, allylsulfonic acid, methallylsulfonic acid, 2-hydroxy-3-(2-propenyloxy)propanesulfonic acid, 2-methyl-2-propenen-1-sulfonic acid, styrenesulfonic acid, vinylsulfonic acid, 3-sulfopropyl acrylate, 3-sulfopropylmethacrylate, sulfomethylacrylamide, sulfomethylmethacrylamide and water soluble salts thereof.

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):

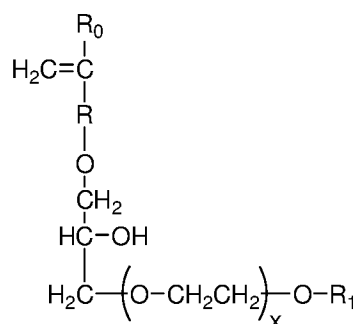
formula (I):



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)

15



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 of at least 50kDa, or even at least 70kDa.

**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):



5



10 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<sub>3</sub>Me;

15 Me is Li, K, Mg/2, Ca/2, Al/3, ammonium, mono-, di-, tri-, or tetraalkylammonium wherein the alkyl groups are C<sub>1</sub>-C<sub>18</sub> alkyl or C<sub>2</sub>-C<sub>10</sub> hydroxyalkyl, or mixtures thereof;

R<sup>1</sup>, R<sup>2</sup>, R<sup>3</sup>, R<sup>4</sup>, R<sup>5</sup> and R<sup>6</sup> are independently selected from H or C<sub>1</sub>-C<sub>18</sub> n- or iso-alkyl; and

R<sup>7</sup> is a linear or branched C<sub>1</sub>-C<sub>18</sub> alkyl, or a linear or branched C<sub>2</sub>-C<sub>30</sub> alkenyl, or a cycloalkyl group with 5 to 9 carbon atoms, or a C<sub>8</sub>-C<sub>30</sub> aryl group, or a C<sub>6</sub>-C<sub>30</sub> arylalkyl group.

20 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.

25 **Anti-redeposition polymer:** Suitable anti-redeposition polymers include polyethylene glycol polymers and/or polyethyleneimine polymers.30 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<sub>4</sub>-C<sub>25</sub> alkyl group, polypropylene, polybutylene, vinyl ester of a saturated C<sub>1</sub>-C<sub>6</sub> mono-carboxylic acid, C<sub>1</sub>-C<sub>6</sub> 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 units can be less than 1, or less than 0.8, the average number of graft sites per ethylene oxide units can be in the range of from 0.5 to 0.9, or the average number of graft sites per ethylene oxide units can be in the range of from 0.1 to 0.5, or from 0.2 to 0.4. A suitable polyethylene glycol polymer is Sokalan HP22. Suitable

5 polyethylene glycol polymers are described in WO08/007320.

**Cellulosic polymer:** Suitable cellulosic polymers are selected from alkyl cellulose, alkyl alkoxyalkyl cellulose, carboxyalkyl cellulose, alkyl carboxyalkyl cellulose, sulphoalkyl cellulose, more preferably selected from carboxymethyl cellulose, methyl cellulose, methyl hydroxyethyl cellulose, methyl carboxymethyl cellulose, and mixtures thereof.

10 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.

**Care polymers:** Suitable care polymers include cellulosic polymers that are cationically  
15 modified and/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. Suitable care polymers also include guar polymers that are cationically and/or hydrophobically modified. Other suitable care polymers include dye lock polymers, for example the condensation oligomer produced by the  
20 condensation of imidazole and epichlorhydrin, preferably in ratio of 1:4:1. A suitable commercially available dye lock polymer is Polyquart® FDI (Cognis).

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

**Alkoxyated polyalkyleneimine:** The composition may comprise an alkoxyated  
25 polyalkyleneimine, wherein said alkoxyated polyalkyleneimine has a polyalkyleneimine core with one or more side chains bonded to at least one nitrogen atom in the polyalkyleneimine core, wherein said alkoxyated polyalkyleneimine has an empirical formula (I) of  $(PEI)_a-(EO)_b-R_1$ , wherein a is the average number-average molecular weight ( $MW_{PEI}$ ) of the polyalkyleneimine core of the alkoxyated polyalkyleneimine and is in the range of from 100 to 100,000 Daltons,  
30 wherein b is the average degree of ethoxylation in said one or more side chains of the alkoxyated polyalkyleneimine and is in the range of from 5 to 40, and wherein  $R_1$  is independently selected from the group consisting of hydrogen,  $C_1$ - $C_4$  alkyls, and combinations thereof.

The composition may comprise an alkoxyated polyalkyleneimine, wherein said alkoxyated polyalkyleneimine has a polyalkyleneimine core with one or more side chains

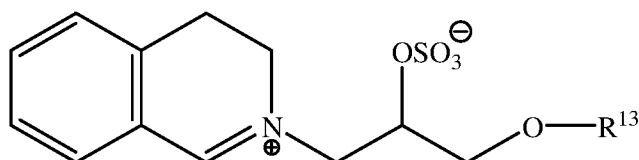
bonded to at least one nitrogen atom in the polyalkyleneimine core, wherein the alkoxyated polyalkyleneimine has an empirical formula (II) of  $(PEI)_o-(EO)_m(PO)_n-R_2$  or  $(PEI)_o-(PO)_n(EO)_m-R_2$ , wherein  $o$  is the average number-average molecular weight ( $MW_{PEI}$ ) of the polyalkyleneimine core of the alkoxyated polyalkyleneimine and is in the range of from 100 to 100,000 Daltons, wherein  $m$  is the average degree of ethoxylation in said one or more side chains of the alkoxyated polyalkyleneimine which ranges from 10 to 50, wherein  $n$  is the average degree of propoxylation in said one or more side chains of the alkoxyated polyalkyleneimine which ranges from 1 to 50, and wherein  $R_2$  is independently selected from the group consisting of hydrogen, C<sub>1</sub>-C<sub>4</sub> alkyls, and combinations thereof.

10 **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.

**Source of hydrogen peroxide:** Suitable sources of hydrogen peroxide include sodium perborate and/or sodium percarbonate.

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

**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:



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

**Pre-formed peracid:** Suitable pre-form peracids include phthalimido-peroxycaproic acid. However, it is preferred that the composition is substantially free of pre-formed peracid. By: 30 “substantially free” it is meant: “no deliberately added”.

**Enzymes:** Suitable enzymes include lipases, proteases, cellulases, amylases and any combination thereof.



**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.

5 Suitable commercially available protease enzymes include those sold under the trade names Alcalase®, Savinase®, Primase®, Durazym®, Polarzyme®, Kannase®, Liquezyme®, Liquezyme 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, 10 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 15 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.

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

20 **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 25 Purastar® Ox Am, Optisize® HT Plus (all Du Pont).

A suitable amylase is described in WO06/002643.

**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 30 fungal cellulases produced from *Humicola insolens*, *Myceliophthora thermophila* and *Fusarium oxysporum*.

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.

**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  
5 suitable lipases include lipases from *Humicola* (synonym *Thermomyces*), e.g., from *H. lanuginosa* (*T. lanuginosus*).

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.  
10 Preferred lipases include those sold under the tradenames Lipex®, Lipolex® and Lipoclean® by Novozymes, Bagsvaerd, Denmark.

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

**Other enzymes:** Other suitable enzymes are bleaching enzymes, such as  
15 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™.

Other suitable enzymes include pectate lyases sold under the tradenames X-Pect®,  
20 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).

**Identity:** When used herein identity or sequence identity refers to the relatedness between two amino acid sequences.

25 For purposes of the present invention, the degree of sequence identity between two amino acid sequences is determined using the Needleman-Wunsch algorithm (Needleman and Wunsch, 1970, *J. Mol. Biol.* 48: 443-453) as implemented in the Needle program of the EMBOSS package (EMBOSS: The European Molecular Biology Open Software Suite, Rice *et al.*, 2000, *Trends Genet.* 16: 276-277), preferably version 3.0.0 or later. The optional parameters used are gap open  
30 penalty of 10, gap extension penalty of 0.5, and the EBLOSUM62 (EMBOSS version of BLOSUM62) substitution matrix. The output of Needle labeled “longest identity” (obtained using the –nobrief option) is used as the percent identity and is calculated as follows:

$$(\text{Identical Residues} \times 100) / (\text{Length of Alignment} - \text{Total Number of Gaps in Alignment}).$$

**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.

5 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 C.I. Fluorescent Brightener 260, which may be used in its beta or alpha crystalline forms, or a  
10 mixture of these forms.

**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  
15 include Acid Violet 50, Direct Violet 9, 66 and 99, Solvent Violet 13 and any combination thereof.

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.

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

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  
25 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.

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  
30 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.

**Reserve alkalinity:** Typically, the composition at 1wt% dilution in deionized water at 20°C, has a reserve alkalinity to pH 7.5 of less than 3.0gNaOH/100g, preferably less than 2.5gNaOH/100g, or even less than 2.0gNaOH/100g.

As used herein, the term “reserve alkalinity” is a measure of the buffering capacity of the detergent composition (g/NaOH/100g detergent composition) determined by titrating a 1% (w/v) solution of detergent composition with hydrochloric acid to pH 7.5 i.e. in order to calculate Reserve Alkalinity as defined herein:

$$\text{Reserve Alkalinity (to pH 7.5) as \% alkali in g NaOH/100 g product} = \frac{T \times M \times 40 \times \text{Vol}}{10 \times \text{Wt} \times \text{Aliquot}}$$

T = titre (ml) to pH 7.5  
 M = Molarity of HCl = 0.2  
 40 = Molecular weight of NaOH  
 Vol = Total volume (ie. 1000 ml)  
 W = Weight of product (10 g)  
 Aliquot = (100 ml)

Obtain a 10g sample accurately weighed to two decimal places, of fully formulated detergent composition. The sample should be obtained using a Pascall sampler in a dust cabinet. Add the 10g sample to a plastic beaker and add 200 ml of carbon dioxide-free de-ionised water. Agitate using a magnetic stirrer on a stirring plate at 150 rpm until fully dissolved and for at least 15 minutes. Transfer the contents of the beaker to a 1 litre volumetric flask and make up to 1 litre with deionised water. Mix well and take a 100 mls  $\pm$  1 ml aliquot using a 100 mls pipette immediately. Measure and record the pH and temperature of the sample using a pH meter capable of reading to  $\pm 0.01$  pH units, with stirring, ensuring temperature is 20°C  $\pm$  2°C. Titrate whilst stirring with 0.2M hydrochloric acid until pH measures exactly 7.5. Note the millilitres of hydrochloric acid used. Take the average titre of three identical repeats. Carry out the calculation described above to calculate the reserve alkalinity to pH 7.5.

**Calculation of the degree of orientation of lamellar phase in the extrudates:** The process of extruding the surfactant powder through the orifice results in the the lamellar phase structures of the surfactant becoming aligned in the direction of material flow due to the nature of the shear the material has been subjected to.

The degree of the alignment of the lamellar sheets is related to the shear rate the material is subjected to as it passes through the orifice and the amount of time this shear is applied for. The greater the applied shear rate the greater the degree of surfactant lamellar alignment. The degree of alignment and structure that is in the extruded material and hence any resultant extrudate has an impact on the rate of dissolution of the extrudates. This is because the surfactant bilayers which actually form the lamellar surfactant phases dissolve by the action of the bilayers becoming detached from each other due to the solvation from the water. Hence the nature of the processing conditions of the extrudates can be expected to have some impact on the rate of dissolution of the extrudates.

The degree of surfactant lamellar alignment can be determined and quantified by the use of Small Angle X-Ray Scattering techniques (SAXS). SAXS is widely used to determine the molecular-level structures of materials.

**SAXS analysis was carried out on the extrudates as follows:** A Xenocs XeuSS 2.0 machine was used equipped with a Pilatus 200k detector and a Cu Genix source. The sample to detector distance was 363mm as per manufacturers recommendations for surfactant type materials.

The data was recorded using a 2D detector so as to allow measurement of the degree of orientation of the lamellar phase.

The extruded material from each shear rate was cut using a very sharp knife edge to form extrudate discs or “salami slices” of approx 1mm thickness. Each extrudate being tested was mounted in a grooved sample holder and measured in two perpendicular orientations as shown below. All measurements were carried out at ambient (~ 20 °C).

**Data analysis:** From the 2D SAXS pattern, the azimuthal profile of the first harmonic of the lamellar peak is plotted. Due to the thickness of the sample when the discs are being measured and the very high degree of attenuation this causes, such samples were measured over a 4 hour period so as to obtain a strong signal. Samples analysed were measured for periods between 15 minutes and 1 hour.

The tangent of the minima at  $\pi/2$  and  $3\pi/2$  is defined as the constant baseline. Integration of the full curve is then carried out.

The degree of orientation is then defined by the following expression:

$$\frac{\text{Integral of signal above baseline}}{\text{Integral of total signal}} = \text{degree of orientation}$$

## EXAMPLES

## Illustrative Example

<b>Ingredient</b>	<b>Amount (in wt%)</b>
<b>Anionic detergent surfactant</b> (such as alkyl benzene sulphonate, alkyl ethoxylated sulphate, alkyl ether carboxylic acid, and mixtures thereof)	from 15wt% to 35wt%
<b>Non-ionic detergent surfactant</b> (such as alkyl ethoxylated alcohol)	from 0.1wt% to 5wt%
<b>Carboxylate polymer</b> (such as co-polymers of maleic acid and acrylic acid and/or carboxylate polymers comprising ether moieties and sulfonate moieties)	from 0wt% to 4wt%
<b>Polyethylene glycol polymer</b> (such as a polyethylene glycol polymer comprising polyvinyl acetate side chains)	from 0wt% to 4wt%
<b>Polyester soil release polymer</b> (such as Repel-o-tex and/or Texcare polymers)	from 0wt% to 2wt%
<b>Cellulosic polymer</b> (such as carboxymethyl cellulose, methyl cellulose and combinations thereof)	from 0wt% to 2wt%
<b>Other polymer</b> (such as care polymers)	from 0wt% to 4wt%
<b>Citric acid and/or sodium citrate</b>	from 10wt% to 40wt%
<b>Inorganic salts</b> (such as sodium carbonate, sodium sesquicarbonate, sodium bicarbonate and combinations thereof)	from 10wt% to 40wt%
<b>Amino acid derivative complexing agent</b> (such as MGDA, GLDA, ASDA and combination thereof)	from 0.1wt% to 40wt%
<b>Amylase</b>	from 0.001wt% to 0.1wt%
<b>Metalloprotease</b> (such as a M4 metalloprotease)	from 0.001wt% to 0.1wt%
<b>Lipase</b>	from 0.001wt% to 0.1wt%
<b>Cellulase</b>	from 0wt% to 0.1wt%
<b>Pectate Lyase</b>	from 0wt% to 0.1wt%
<b>Mannanase</b>	from 0wt% to 0.1wt%
<b>Subtilisin-type protease</b>	from 0wt% to 0.1wt%

<b>Fluorescent brightener</b>	from 0wt% to 1.0wt%
<b>Alkoxylated polyaryl/polyalkyl phenol</b>	from 0.5wt% to 20wt%
<b>Hueing dye</b>	from 0.001wt%to 0.5wt%
<b>Organic pigment and/or inorganic pigment</b>	from 0.001wt% to 0.5wt%
<b>Other ingredients &amp; miscellaneous</b>	balance to 100wt%

### **Preparation of surfactant-containing extrudates at different applied shear rates**

Surfactant powder was prepared as follows

5

1000g of C12-14 linear alkylbenzene sulphonic acid (HLAS) (sourced from Tensachem SA) was placed in the bowl of a kitchen Kenwood K-Blade mixer. 245g of a 50% active sodium hydroxide solution were gradually added to the HLAS over a 5 minute period with vigorous stirring so as to avoid excessive generation of heat. After the sodium hydroxide solution had been added, the stirring was continued at a medium speed for a further 20 minutes to allow the reaction to complete. The stirring was then stopped and the paste allowed to cool to ambient. This formed a high active paste of sodium linear alkylbenzene sulphonate of 82% active (by wt) with the balance being water and miscellaneous (such as unsulphonated material).

10

15 The LAS paste described above was then converted to a surfactant powder containing LAS, AE7 nonionic surfactant (Neodol 45-7) and Polyethylene Glycol 4000 as follows.

20

91.3g of the LAS paste described above were vigorously mixed by hand with 7.3g of warm (hence liquid) Neodol 45-7 and 1.5g of molten PEG 4000. The nonionic surfactant and the PEG4000 were easily blended into the LAS paste to make a visually homogenous paste. This paste mixture was then dried in an oven at 90 °C for 48 hours to make a hard and dry material. This material was then ground in a domestic coffee grinder to make a fine powder which was passed through a 425 micron sieve. The equilibrium relative humidity of the powder was measured to be 6% at 23 °C. The composition of the surfactant powder was calculated (based on measurement of weight loss) to be 88.4% LAS, 8.6% AE7, 1.8% PEG 4000 with the balance being water and miscellaneous.

25

This powder was then divided into three batches and used to make surfactant extrudates at different shear rates as follows. All tests were done at ambient temperature (~ 20 °C) with no sources of external heating or cooling on the die or piston.

5 ~ 30g of surfactant powder was loaded into a die consisting of a circular steel cylinder of length 100mm and internal diameter 26mm. One end of the die was open to allow entry of a suitable piston or punch and in the other end there was a 5mm diameter circular orifice located axisymmetrically. The length of the orifice through the steel of the die was 3mm.

10 The loaded die was suitably mounted on an Instron 3369 Compression Tester and a suitable punch placed in the die such that it closely fitted the internal diameter but could still be moved without excessive force. The punch loosely compressed the surfactant powder. The crosshead of the Instron was moved such that the load cell of the Instron (model 2525) forced the punch down at a controlled speed. This first compressed the surfactant powder and then forced the surfactant  
15 powder to be extruded through the orifice at a controlled constant speed. This was continued until all the surfactant powder had been extruded. The test was carried out at three different crosshead speeds (0.1mm/minute, 0.5mm/minute and 5mm/minute) using ~ 30g portions of the powder made above. This gave three separate examples of extrudates made at different crosshead speeds and thus different shear rates in the orifice.

20

The shear rates experienced by the material at the different crosshead speeds in the orifice are as below. The material in the orifice is treated as a simple Newtonian fluid flowing through a pipe hence the shear rate in the orifice is defined by the following equation

25 Shear Rate = {4 x Velocity}/Radius

Shear Rate in reciprocal seconds

Velocity = average velocity of material passing through the orifice in metres/second

Radius = radius of the orifice in metres

30

This gives the following shear rates experienced by the material during the different runs

Crosshead speed = 0.1mm/min.      Shear Rate =            0.036 s<sup>-1</sup>

Crosshead speed = 5mm/min.      Shear Rate =            1.8 s<sup>-1</sup>



The samples were then coated with inorganic coating agent and tested for solubility.

### **Analysis**

5

Using the technique described in the description, the degree of orientation was measured for the different extrudates. Results were as below.

<b>Sample</b>	<b>Degree of orientation (in the direction of the shear)</b>
0.036 s <sup>-1</sup> applied shear rate	89%
1.8 s <sup>-1</sup> applied shear rate	95%

10

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

What is claimed is:

1. A coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein the particle comprises:

(d) from 20wt% to 39wt% deterative surfactant selected from anionic deterative surfactant and/or non-ionic deterative surfactant;

(e) from 10wt% to 40wt% inorganic salts selected from sodium carbonate, sodium sesquicarbonate, sodium bicarbonate and any mixtures thereof;

(f) optionally, from 10wt% to 40wt% citric acid and/or salts thereof;

wherein the deterative surfactant comprises from 51wt% to 100wt% alkyl benzene sulphonate,

wherein the coating comprises the inorganic salt (b), and wherein the core comprises the deterative surfactant (a),

5 wherein the alkyl benzene sulphonate has a degree of orientation of at least 92%.

2. A coated detergent particle according to claim 1, wherein the degree of orientation of at least 95%.

3. A coated detergent particle according to any preceding claim, wherein at 1wt% dilution in de-ionised water at 20°C, the composition has a pH in the range of from 7.6 to 10.0.

10 4. A coated detergent particle according to any preceding claim,, wherein at 1wt% dilution in de-ionised water at 20°C, the composition has a reserve alkalinity to pH 7.5 of greater than 3.0.

15 5. A coated detergent particle according to any preceding claim, wherein the coated laundry detergent particle is curved.

6. A process for the preparation of a coated detergent particle according to any preceding claim, wherein the process comprises the following steps:

(i) forming a deterative mixture comprising from 40wt% to 90wt% deterative surfactant;

- (ii) extruding the deterative mixture of step (i) to form an extruded material;
- (iii) coating the extruded material with water-soluble inorganic salt in the form of an aqueous solution to form a wet coated extruded material comprising from 1wt% to 40wt% water soluble inorganic salt: and
- (iv) removing water from the wet coated extruded material to form the coated detergent particle,

wherein, in step (ii) the material in the orifice of the extruder is subjected to a shear rate of at least  $0.033\text{ms}^{-1}$ .

7. A process according to claim 6, wherein in step (ii) the material in the orifice of the extruder is subjected to a shear rate of at least  $0.33\text{ms}^{-1}$ .

**INTERNATIONAL SEARCH REPORT**

International application No  
PCT/US2017/067156

**A. CLASSIFICATION OF SUBJECT MATTER**  
 INV. C11D3/10 C11D3/386 C11D17/00  
 ADD.  
 According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**  
 Minimum documentation searched (classification system followed by classification symbols)  
 C11D  
 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
 EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2013/139702 A1 (UNILEVER PLC [GB]; UNILEVER NV [NL]; CONOPCO INC DBA UNILEVER [US]) 26 September 2013 (2013-09-26) page 18, line 1 - page 21, line 4; claims; examples	1-7
A	WO 2010/122050 A2 (UNILEVER PLC [GB]; UNILEVER NV [NL]; UNILEVER HINDUSTAN [IN]; CHAMBERS) 28 October 2010 (2010-10-28) cited in the application claims; examples	1-7

Further documents are listed in the continuation of Box C.

See patent family annex.

\* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier application or patent but published on or after the international filing date
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- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "&" document member of the same patent family

Date of the actual completion of the international search

6 February 2018

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2017/067156

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