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(54) **DETERGENT COMPOSITION COMPRISING HYDRATE-FORMING SALT PARTICLES COATED WITH BETAINE**

BETAINE BEZOGENEN PARTIKEL AUS HYDRATEBILDENDEM SALZ FÜR ASCHMITTELZUSAMMENSETZUNG

PARTICULE COMPRENANT UN SEL HYDRATABLE, ENROBEE DE BETAINE, POUR COMPOSITION DÉTERGENTE

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**US-A1- 2010 261 633**

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**Description****Field of the invention**

5 [0001] The present invention relates to solid detergent compositions having improved solubility. More particularly, it relates to solid laundry detergent compositions having hydrate-forming salts with improved cold-water solubility.

**Background of the invention**

10 [0002] The problem of providing improved dispensing, dispersing and easily dissolving laundry detergent powders is well-known and has been addressed many times in the past. It is undesirable, for example, to have a solid laundry composition with slow dispensing and low solubility, which forms a residue in the drawer of many automatic washing machines.

15 [0003] Furthermore, in some regions during winter, cold water at around 10°C or lower is usually used for washing fabrics. Under such washing conditions noticeable lumps form in the dispensing drawer of a washing machine that may appear as visible solid white masses in the drawer as well as on the washed fabrics. The problem further worsens when the order of addition to the washing machine is laundry detergent product first, clothes second, and water last.

20 [0004] During the release of cold water, into the washing machine, water gradually penetrates into the mass of the solid detergent composition, without substantial physical or mechanical power applied thereto, and hydrates the surfactant and also the hydrate-forming salts present in the composition. Surfactants especially the anionic surfactants present in the detergent compositions form a highly viscous pasty phase. The hydrated water-soluble salts precipitate out in the form of crystals, which further harden the viscous pasty phase of the surfactant. The formed crystals also connect with one another to convert the phase per se into a firm, hydrated solid phase, which is difficult to disperse or dissolve by a mechanical force applied thereafter. The detergent composition under such conditions remains in an undissolved state

25 during ordinary washing time. Such a phenomenon is undesirable for the users.

[0005] Some prior art documents have tried to address the problem of low dispersion and dissolution of detergent compositions in cold water caused primarily by the presence of the hydrate-forming crystalline salts.

30 [0006] In US 2010/0261633, detergent builder granules are described, which contain a water-soluble inorganic salt containing an inorganic salt having and/or being capable of forming a hydrate crystal, and a clay mineral. The detergent builder granules have excellent dispersibility in cold water. In EP229671A2 (Kao, 1987) disclosing a high-density granular detergent composition in which high dispersibility and solubility in cold water is achieved by including particles of water-soluble crystalline, inorganic salt carrying thereon an organic substance having a melting point of 40°C or lower which inhibits hydration of the coated particles. The organic substance is defined to be a non-ionic surfactant. The non-ionic surfactants include polyoxyethylene, polyhydric alcohols and alkanolamides surfactants.

35 [0007] Hydrated salts can easily form during low temperatures in the powder-dispensing drawer of washing machines when appropriate conditions are present. Low water content, low temperatures are conditions particularly favourable for the formation of hydrates. The formation of hydrates often results in loss of surfactant available for washing and these may deposit on fabric, which resemble white stain-like spots on the washed fabric.

40 [0008] The present inventors have investigated ways of providing solid detergent composition that has improved solubility in cold-water conditions specifically at temperatures lower than 10°C, more preferably lower than 5°C, and towards providing a coated filler agent for use in detergent composition, which coated filler agent considerably inhibits formation of crystalline masses, which are difficult to disperse and disperse.

**Summary of the invention**

45 [0009] After intensive investigations made for the purposes of solving the problem, the inventors have found that a coated filler agent having high dispersibility and solubility in cold water at temperatures lower than 10°C, more preferably lower than 5°C can be obtained by at least partially coating the hydrate-forming salt with a betaine class of zwitterionic compound.

50 [0010] Accordingly, in a first aspect the invention provides a coated filler agent for use in a solid detergent composition comprising a hydrate-forming salt and a betaine class of zwitterionic compound wherein the hydrate-forming salt is at least partially coated with the zwitterionic compound.

[0011] In a second aspect, the invention provides a filler composition according to the invention having the coated filler agent and a water-soluble salt.

55 [0012] In a third aspect, the invention provides use of a coated filler agent or a filler composition according to the invention in a detergent composition for providing improved solubility.

[0013] These and other aspects, features and advantages will become apparent to those of ordinary skill in the art from a reading of the following detailed description and the appended claims. For the avoidance of doubt, any feature

of one aspect of the present invention may be utilized in any other aspect of the invention. The word "comprising" is intended to mean "including" but not necessarily "consisting of" or "composed of." In other words, the listed steps or options need not be exhaustive. It is noted that the examples given in the description below are intended to clarify the invention and are not intended to limit the invention to those examples per se. Similarly, all percentages are weight/weight percentages unless otherwise indicated. Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts of material or conditions of reaction, physical properties of materials and/or use are to be understood as modified by the word "about". Numerical ranges expressed in the format "from x to y" are understood to include x and y. When for a specific feature multiple preferred ranges are described in the format "from x to y", it is understood that all ranges combining the different endpoints are also contemplated.

### Detailed description of the invention

**[0014]** In a first aspect, the invention relates to a coated filler agent for use in a solid detergent composition comprising a hydrate-forming salt and a betaine class of zwitterionic compound wherein the hydrate-forming salt is at least partially coated with the zwitterionic compound.

### Coated filler agent

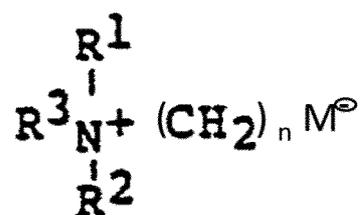
#### Zwitterionic compound

**[0015]** The coated filler agent of the present invention includes a betaine class of zwitterionic compound.

**[0016]** The betaine class of zwitterionic compound is preferably a zwitterionic surfactant or a zwitterionic polymer, preferably a zwitterionic surfactant. Suitable zwitterionic surfactants are known in the art, and include, for example, those surfactants broadly described as derivatives of aliphatic quaternary ammonium, phosphonium and sulfonium compounds, in which the aliphatic radicals can be straight or branched chain, and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic group such as carboxyl, sulfonate, sulfate, phosphate or phosphonate.

**[0017]** Specific examples of suitable zwitterionic surfactants include alkyl betaines, such as cocodimethyl carboxymethyl betaine, lauryl dimethyl carboxymethyl betaine, lauryl dimethyl alphacarboxy-ethyl betaine, cetyl dimethyl carboxymethyl betaine, lauryl bis-(2-hydroxy-ethyl)carboxy methyl betaine, stearyl bis-(2-hydroxypropyl)carboxymethyl betaine, oleyl dimethyl gamma-carboxypropyl betaine, and lauryl bis-(2-hydroxypropyl)alpha-carboxyethyl betaine, alkyl arnido-propyl betaines, and alkyl sultaines, such as cocodimethyl sulfopropyl betaine, stearyldimethyl sulfopropyl betaine, lauryl dimethyl sulfoethyl betaine, lauryl bis-(2-hydroxy-ethyl)sulfopropyl betaine, cocamidopropyl hydroxysultaine and alkylamidopropylhydroxy sultaines. Preferred examples of phospho-betaine includes but are not limited to propyl Monosodium Phosphobetaine, Cocamido Disodium Ethyl Phosphobetaine, Coeamido Disodium 3-l-Hydroxypropyl Phospho-betaine, Laurie myriatic Amido 3-Hydroxypropyl Phospho-betaine.

**[0018]** Useful betaines have the structure as given in formula 1



FORMULA 1

wherein

$M^\ominus$  : is selected from a sulphonate ( $SO_3^-$ ), phosphonate ( $PO_4^-$ ), carbonate ( $CO_3^-$ )

$R_1$  : is an alkyl group having from 1 to 14 carbon atoms, preferably 1 to 2 carbon atoms and is preferably a straight chain or branched chain.

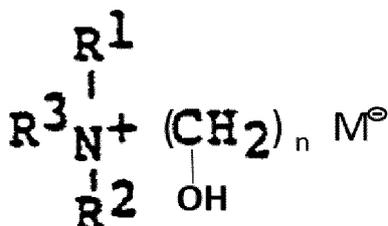
$R_2$  : is an alkyl group having from 1 to 14 carbon atoms, preferably 1 to 2 carbon atoms and is preferably a straight chain or branched chain.

$R_3$  : is an alkyl group having from 6 to 14 carbon atoms, preferably 10 to 14 carbon atoms and is preferably a straight chain or branched chain or



where R is an alkyl group having from 6 to 14 carbon atoms, preferably 10 to 14 carbon atoms and is preferably a straight chain or branched chain. n is an integer from 1 to 6, preferably 2 to 3. Specific betaines useful in the products of the invention are for example alpha-(tetradecyldimethylammonio)acetate, beta-(hexadecyldiethylammonio)propionate, and gamma-(dodecyldimethylammonio)butyrate.

**[0019]** Also useful betaines have the structure as given in formula 2



FORMULA 2

wherein

$\text{M}^\ominus$  : is selected from a sulphonate ( $\text{SO}_3^-$ ), phosphonate ( $\text{PO}_4^-$ ), carbonate ( $\text{CO}_3^-$ )

$\text{R}_1$  : is an alkyl group having from 1 to 14 carbon atoms, preferably 1 to 2 carbon atoms and is preferably a straight chain or branched chain.

$\text{R}_2$  : is an alkyl group having from 1 to 14 carbon atoms, preferably 1 to 2 carbon atoms and is preferably a straight chain or branched chain.

$\text{R}_3$  : is an alkyl group having from 6 to 14 carbon atoms, preferably 10 to 14 carbon atoms and is preferably a straight chain or branched chain or

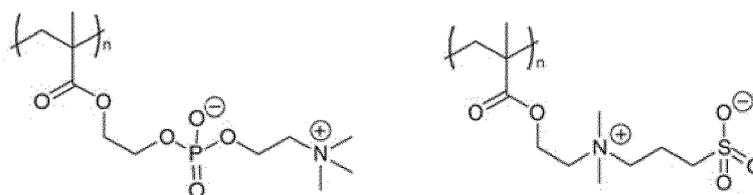


where R is an alkyl group having from 6 to 14 carbon atoms, preferably 10 to 14 carbon atoms and is preferably a straight chain or branched chain. n is an integer from 1 to 6, preferably 2 to 3. Specific betaines useful in the products of the invention are for example alpha-(tetradecyldimethylammonio)acetate, beta-(hexadecyldiethylammonio)propionate, and gamma-(dodecyldimethylammonio)butyrate.

**[0020]** Specific useful sultaines are for example 3-(dodecyldimethylammonio)propane-1-sulfonate and 3-(tetradecyldimethylammonio)ethane-1-sulfonate.

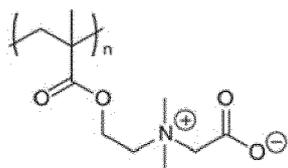
**[0021]** The zwitterionic surfactant is most preferably a carbo-betaine, sulpho-betaine, phospho-betaine, carboxy-betaine or mixtures thereof.

**[0022]** The zwitterionic compound is preferably a zwitterionic polymer. Preferred zwitterionic polymers include but are not limited to poly(phosphobetaine methacrylate), poly(sulfobetaine methacrylate), poly(carboxybetaine methacrylate), poly(serine methacrylate).

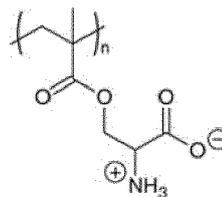


PPBMA: poly(phosphobetaine methacrylate)

PSBMA: poly(sulfobetaine methacrylate)



PCBMA: (poly(carboxybetaine methacrylate))



PSrMA: poly(serine methacrylate)

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10 **[0023]** The betaine class of zwitterionic compound is present in an amount ranging from 0.5 to 20wt% based on the coated filler agent.

**[0024]** Preferably the amount of zwitterionic compound in the coated filler agent is at least 1wt%, still preferably at least 2.5wt%, further preferably at least 3wt% and most preferably at least 5wt%, but typically not more than 10wt%, still preferably not more than 15wt% and most preferably not more than 20wt% based on the coated filler agent.

15 **[0025]** The betaine class of zwitterionic compound according to the present invention is at least partially coating the hydrate-forming salt. More preferably, the zwitterionic compound completely covers the hydrate-forming salt.

#### Hydrate-forming salt

20 **[0026]** The coated filler agent of the present invention comprises a hydrate-forming salt. The hydrate-forming salt of the present invention is at least partially coated with a betaine class of zwitterionic compound.

**[0027]** Non-limiting examples of the hydrate-forming salt are sulphate, carbonate or bicarbonate salt of alkaline earth metal or alkali metal and mixtures thereof.

25 **[0028]** Preferred examples of the hydrate-forming salt according to the present invention are selected from the group consisting of sodium carbonate, sodium sulphate, sodium bicarbonate, sodium tripolyphosphate, sodium pyrophosphate, sodium orthophosphate, magnesium sulphate or mixtures thereof. Preferably the hydrate-forming salt is sodium carbonate, sodium sulphate or mixtures thereof.

30 **[0029]** Present invention is particularly suitable for any salt that exhibits significant change in solubility with decrease in temperature. This is specifically suitable for salts which easily crystallises from the aqueous solution at a relatively low temperature to form crystals which easily coalesce together to form a firm structure. Without being bound by theory it is believed that this crystal formation inhibits the dispersibility and solubility of the detergent composition. Among the known salts used in the detergent composition, sodium carbonate generates a large amount of heat upon hydration and dissolves even in cold water to form a solution having a high concentration with the generation of heat. The heat is later taken up by the low-temperature surrounding and with the lowered temperature the solubility of the hydrated salt reduces and the salt precipitates as crystals. At a temperature of 32°C or below, sodium carbonate is in the form of its decahydrate and it has a large amount of water of crystallization. The hydration increases the volume of the undissolved crystals and such hydrated crystals coalesce together easily to form a firmer structure. Therefore, the problem of residue formation is particularly higher in compositions with sodium carbonate.

35 **[0030]** The hydrate-forming salt is present in an amount ranging from 80 to 99wt% based on the coated filler agent. Preferably the amount of hydrate-forming salt in the coated filler agent is at least 80wt%, still preferably at least 85wt%, further preferably at least 87wt% and most preferably at least 90wt%, but typically not more than 99wt%, still preferably not more than 97.5wt% and most preferably not more than 95wt% based on the coated filler agent.

#### Method of preparing the coated filler agent

45

**[0031]** Any of the methods known in the art for coating may be used for the present invention.

**[0032]** Preferably the method for preparing the coated filler agent comprises the steps of:

- 50 a. providing a hydrate-forming salt;  
 b. applying a coating onto the hydrate-forming salt by either spraying or pouring betaine class of zwitterionic compound or intimately mixing the hydrate forming salt and the zwitterionic compound or preparing a slurry of the hydrate-forming salt and the zwitterionic compound and thereafter drying the slurry.

55 **[0033]** A preferred method of coating includes spraying or pouring the zwitterionic compound onto the hydrate-forming salt. The hydrate-forming salt is constantly mixed during the process of coating. Preferably the hydrate-forming salt is taken in a plough shear mixer or other high speed mixers, which can keep the hydrate-forming salt at high speed rotation during the coating process. The coated filler agent obtained from the process are according to the present invention and the zwitterionic compound at least partially covers or coats the hydrate-forming salt. In another preferred method, the

hydratable salt is taken in a sigma or a z-blender during the coating process in which the zwitterionic compound is constantly sprayed or poured onto the hydrate-forming salt.

5 **[0034]** Another method of preparing the coated filler agent of the present invention involves a first step of preparing an aqueous slurry comprising the hydrate-forming salt and the zwitterionic compound, the slurry is thereafter dried preferably by spray drying route, the coated filler agent obtained is according to the present invention in which the zwitterionic compound at least partially covers the hydrate-forming salt.

#### Filler composition

10 **[0035]** According to a second aspect of the present invention, disclosed is a filler composition having the coated filler agent of the first aspect and a water-soluble salt.

#### Water-soluble salt

15 **[0036]** The filler composition according to the second aspect of the present invention comprises a water-soluble salt that has a solubility of more than 30 grams/100mL in distilled water when measured at a temperature of 10°C, the water-soluble salt being present separate from the hydrate-forming salt. Preferably the water-soluble salt has a solubility of at least 35grams/100mL, still preferably at least 40 grams/100mL, further preferably at least 45grams/100mL.

20 **[0037]** Non-limiting examples of the water-soluble salt includes potassium carbonate, potassium chloride, potassium formate, potassium acetate, sodium chloride, sodium acetate, sodium formate, calcium acetate, magnesium chloridesodium di-hydrogen phosphate, potassium bi-sulphate, magnesium acetate, sodium fumarate. More preferably the water-soluble salt is selected from sodium chloride, sodium acetate or sodium formate.

25 **[0038]** Preferably the amount of water-soluble salt in the filler composition is at least 5wt%, still preferably at least 10wt%, further preferably at least 15wt% and most preferably at least 20wt%, but typically not more than 40wt%, still preferably not more than 50wt% and most preferably not more than 60wt% based on the filler composition.

#### Detergent composition

30 **[0039]** According to a third aspect of the present invention disclosed is a solid detergent composition comprising a coated filler agent according to the first aspect or a filler composition according to the second aspect of the present invention.

35 **[0040]** A solid detergent composition according to the present disclosure encompasses powders as well as a variety of cast and extruded forms including, for example, pellets, blocks, particles and tablets. It should be understood that the term "solid" refers to the state of the detergent composition under the expected conditions of storage and use of the solid detergent composition. In general, it is expected that the detergent composition will remain a solid when provided at a temperature of up to about 100°F. Preferably the solid detergent composition is a powder, tablet, granular or a particulate composition.

40 **[0041]** In certain embodiments, the detergent composition is provided in the form of a unit dose. A unit dose refers to a detergent composition in unit size so that the entire unit is used during a single washing cycle. When the solid detergent composition is provided as a unit dose, it is preferably provided as a cast solid, an extruded particle or pellet, or a tablet having a size of between about 1 gram and about 50 grams. In other embodiments, a cast solid, an extruded pellet, or a tablet having a size of between 50 grams up through 250 grams, or an extruded solid with a weight of about 100 grams or greater.

45 **[0042]** Furthermore, it should be appreciated that the solid detergent composition can be provided as a cast solid, an extruded pellet, or a tablet so that a plurality of the solids will be available in a package having a size of between about 40 grams and about 11,000 grams.

50 **[0043]** In other embodiments, the solid detergent composition is provided in the form of a multiple-use solid, such as, a block or a plurality of pellets, and can be repeatedly used to generate aqueous detergent composition for multiple washing cycles. In certain embodiments, the solid detergent composition is provided as a powder, cast solid, an extruded block, or a tablet having a mass of between about 5 grams and 10 kilograms. In certain embodiments, a multiple-use form of the solid detergent composition has a mass between about 1 and 10 kilograms, more preferably 1 kg to 5 kg.

55 **[0044]** Preferably the solid detergent composition includes 2wt% to 40wt% of the coated filler agent. Preferably the amount of coated filler agent in the solid detergent composition is at least 2wt%, still preferably at least 5wt%, further preferably at least 10wt% and most preferably at least 15wt%, but typically not more than 30wt%, still preferably not more than 35wt% and most preferably not more than 40wt% based on the detergent composition.

**[0045]** In a further embodiment of the present invention, the detergent composition includes a filler composition having a coated filler agent and a water-soluble salt.

**[0046]** The detergent composition preferably includes a filler composition in an amount which is at least 5wt%, still

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preferably at least 8wt%, further preferably at least 10wt% and most preferably at least 15wt% based on the weight of the detergent composition, but typically not more than 30wt%, still preferably not more than 40wt% and most preferably not more than 50wt% based on the weight of the detergent composition.

5 Surfactant:

**[0047]** Preferably, the detergent composition includes a surfactant selected from anionic, non-ionic, zwitterionic, cationic or amphoteric surfactant.

10 Anionic Surfactant

**[0048]** Suitable anionic detergent compounds which may be used are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals.

15 **[0049]** Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher C<sub>8</sub> to C<sub>18</sub> alcohols, produced for example from tallow or coconut oil, sodium and potassium alkyl C<sub>9</sub> to C<sub>20</sub> benzene sulphonates, particularly sodium linear secondary alkyl C<sub>10</sub> to C<sub>15</sub> benzene sulphonates; and sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum.

20 **[0050]** The anionic surfactant is preferably selected from: linear alkyl benzene sulphonate; alkyl sulphates; alkyl ether sulphates; soaps; alkyl (preferably methyl) ester sulphonates, and mixtures thereof.

**[0051]** The most preferred anionic surfactants are selected from: linear alkyl benzene sulphonate; alkyl sulphates; alkyl ether sulphates and mixtures thereof. Preferably the alkyl ether sulphate is a C<sub>12</sub> to C<sub>14</sub> n-alkyl ether sulphate with an average of 1 to 3EO (ethoxylate) units. Sodium lauryl ether sulphate is particularly preferred (SLES). Preferably the linear alkyl benzene sulphonate is a sodium C<sub>11</sub> to C<sub>15</sub> alkyl benzene sulphonates. Preferably the alkyl sulphates is a linear or branched sodium C<sub>12</sub> to C<sub>18</sub> alkyl sulphates. Sodium dodecyl sulphate is particularly preferred, (SDS, also known as primary alkyl sulphate). Preferably the solid detergent composition includes 2wt% to 80wt% of the anionic surfactant.

30 Non-ionic surfactant

**[0052]** The nonionic surfactant component preferably comprises alcohol ethoxylate. The alcohol ethoxylates are formed from the reaction of primary or secondary alcohols with ethylene oxide. Typically, an aliphatic C<sub>8</sub> to C<sub>18</sub> primary or secondary linear or branched alcohol is reacted with ethylene oxide in the required molar amount to produce the alcohol ethoxylate. Preferred alcohol ethoxylates have from 2 to 40, preferably from 3 to 30, more preferably from 5 to 20 ethylene oxide units attached to the aliphatic chain.

35 **[0053]** The surfactants may be chosen from the surfactants described in "Surface Active Agents" Vol. 1, by Schwartz & Perry, Interscience 1949, Vol. 2 by Schwartz, Perry & Berch, Interscience 1958, in the current edition of "McCutcheon's Emulsifiers and Detergents" published by Manufacturing Confectioners Company or in "Tenside-Taschenbuch", H. Stache, 2nd Edn., Carl Hauser Verlag, 1981. Preferably the surfactants used are saturated.

40 **[0054]** Suitable nonionic detergent compounds which may be used include, in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are C<sub>6</sub> to C<sub>22</sub> alkyl phenoethylene oxide condensates, generally 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic C<sub>8</sub> to C<sub>18</sub> primary or secondary linear or branched alcohols with ethylene oxide, generally 5 to 40 EO.

45 **[0055]** The total amount of surfactant present in the composition is preferably at least 5 wt. %, more preferably at least 10 wt. %, More preferably the total amount of surfactant is from 15 to 65 wt. %, preferably from 10 to 50 wt. %

50 **[0056]** Other surfactants such as cationic surfactants and amphoteric/zwitterionic surfactants such as betaines may also be present in addition to the aforementioned nonionic and anionic surfactants.

### Builders and sequestrants

**[0057]** Builder materials may be selected from 1) calcium sequestrant materials, 2) precipitating materials, 3) calcium ion-exchange materials and 4) mixtures thereof.

55 **[0058]** Examples of calcium sequestrant builder materials include alkali metal polyphosphates, such as sodium tripolyphosphate and organic sequestrants, such as ethylene diamine tetra-acetic acid.

**[0059]** Examples of precipitating builder materials include sodium orthophosphate.

**[0060]** Examples of calcium ion-exchange builder materials include various types of water-insoluble crystalline or amorphous aluminosilicates, of which zeolites are well known representatives thereof, e.g. zeolite A, zeolite B (also known as zeolite P), zeolite C, zeolite X, zeolite Y and also the zeolite P-type described in EP-A-0,384,070.

**[0061]** The detergent compositions may also optionally contain relatively low levels of organic detergent builder or sequestrant material. Examples include the alkali metal, citrates, succinates, malonates, carboxymethyl succinates, carboxylates, polycarboxylates and polyacetyl carboxylates. Specific examples include sodium, potassium and lithium salts of oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, ethylene diamine tetra-acetic acid, diethylene-triaminepentaacetic acid, alkyl- or alkenylsuccinic acid, nitrilotriacetic acid, and citric acid. Other examples are DEQUEST™, organic phosphonate type sequestering agents sold by Monsanto and alkanehydroxy phosphonates.

**[0062]** Other suitable organic builders include the higher molecular weight polymers and copolymers known to have builder properties. For example, such materials include appropriate polyacrylic acid, polymaleic acid, and polyacrylic/polymaleic acid copolymers and their salts, such as those sold by BASF under the name SOKALAN™.

**[0063]** If utilized, the builder materials may comprise from about 0.5% to 20 wt%, preferably from 1 wt% to 10 wt%, of the composition. The preferred builder level is less than 10 wt% and preferably less than 5 wt% of the composition. Preferably the laundry detergent formulation is a non-phosphate built laundry detergent formulation, i.e., contains less than 1 wt. % of phosphate.

#### Shading Dye

**[0064]** Shading dyes deposit to fabric during the wash or rinse step of the washing process providing a visible hue to the fabric. Shading of white garments may be done with any colour depending on consumer preference. Blue and Violet are particularly preferred shades and consequently preferred dyes or mixtures of dyes are ones that give a blue or violet shade on white fabrics. The shading dyes used are preferably blue or violet. The shading dye chromophore is preferably selected from the group comprising: mono-azo, bis-azo, triphenylmethane, triphenodioxazine, phthalocyanin, naphtholactam, azine and anthraquinone. Most preferably mono-azo, bis-azo, azine and anthraquinone. Most preferably the dye bears at least one sulfonate group. Preferred shading dyes are selected from direct dyes, acid dyes, hydrophobic dyes, cationic dyes and reactive dyes.

**[0065]** If included, the shading dye is present in the composition in range from 0.0001 to 0.01 wt %.

#### Fluorescent Agent

**[0066]** The composition preferably comprises a fluorescent agent (optical brightener). Fluorescent agents are well known and many such fluorescent agents are available commercially. Usually, these fluorescent agents are supplied and used in the form of their alkali metal salts, for example, the sodium salts. The total amount of the fluorescent agent or agents used in the composition is generally from 0.005 to 2 wt. %, more preferably 0.01 to 0.1 wt. %. Preferred classes of fluorescer are: Di-styryl biphenyl compounds, e.g. Tinopal (Trade Mark) CBS-X, Di-amine stilbene di-sulphonic acid compounds, e.g. Tinopal DMS pure Xtra and Blankophor (Trade Mark) HRH, and Pyrazoline compounds, e.g. Blankophor SN. Preferred fluorescers 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-sulfoslyryl)biphenyl.

#### Perfume

**[0067]** Preferably the composition comprises a perfume. The perfume is preferably in the range from 0.001 to 3 wt. %, most preferably 0.1 to 1 wt. %. Many suitable examples of perfumes are provided in the CTFA (Cosmetic, Toiletry and Fragrance Association) 1992 International Buyers Guide, published by CFTA Publications and OPD 1993 Chemicals Buyers Directory 80th Annual Edition, published by Schnell Publishing Co.

**[0068]** It is commonplace for a plurality of perfume components to be present in a formulation. In the compositions of the present invention it is envisaged that there will be four or more, preferably five or more, more preferably six or more or even seven or more different perfume components.

#### Polymers

**[0069]** The composition may comprise one or more polymers. Polymers can assist in the cleaning process by helping to retail soil in solution or suspension and/or preventing the transfer of dyes. Polymers can also assist in the soil removal process. Dye transfer, anti-redeposition and soil-release polymers are described in further detail below.

**[0070]** The composition may comprise one or more polymers. Examples are carboxymethylcellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, poly(ethylene glycol), poly(vinyl alcohol), ethoxylated polyamines, polycarboxylates

such as polyacrylates, maleic/acrylic acid copolymers and lauryl methacrylate/acrylic acid copolymers.

#### Dye transfer inhibitors

5 **[0071]** Modern detergent compositions typically employ polymers as so-called 'dye-transfer inhibitors'. These prevent migration of dyes, especially during long soak times. Generally, such dye-transfer inhibiting agents include polyvinyl pyrrolidone polymers, polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, manganese pthalocyanine, peroxidases, and mixtures thereof, and are usually present at a level of from 0.01 to 10 wt. % based on total amount in the laundry composition.

#### Anti-redeposition polymers

15 **[0072]** Anti-redeposition polymers are designed to suspend or disperse soil. Typically 25 antiredeposition polymers are ethoxylated and or propoxylated polyethylene imine or polycarboxylate materials, for example, Acrylic acid based homo or copolymers available under the trade mark ACUSOL from Dow Chemical, Alcosperse from Akzonobel or Sokolan from BASF.

#### Soil Release Polymers

20 **[0073]** Examples of suitable soil release polymers include graft copolymers of poly(vinyl ester), e.g., C<sub>1</sub> - C<sub>6</sub> vinyl esters, preferably poly(vinyl acetate) grafted onto polyalkylene oxide backbones. Commercially available soil release agents of this kind include the SOKALAN type of material, e.g., SOKALAN HP-22, available from BASF (West Germany). Further suitable soil release polymers of a different type include the commercially available material ZELCON 5126 (from DuPont) and MILEASE T (from ICI). If present, the soil release polymer may be included at a level of from 0.01 to 10 wt. % based on total amount in the laundry composition. Further examples of soil release polymers are terephthalic acid / glycol copolymers sold under the tradenames Texcare, Repel-o-tex, Gerol, Marloquest, Cirrasol.

#### Enzyme

30 **[0074]** Enzymes can also be present in the formulation. Preferred enzymes include protease, lipase, pectate lyase, amylase, cutinase, cellulase, mannanase.

**[0075]** According to another aspect of the present invention disclosed is use of a coated filler agent of the first aspect or a filler composition of the second aspect in a detergent composition for improving cold water solubility and/or dispersibility.

35 **[0076]** The invention will now be explained in greater details with non-limiting examples of compositions according to the present invention.

#### **Examples**

40 Example 1: Evaluation of the solubility and dispersibility.

**[0077]** To determine the solubility of the hydrate-forming salts in the coated filler agent different compositions were prepared and tested in the following manner. The compositions tested are given in table 1.

45 **[0078]** 10 grams of the composition having the hydrate-forming salt as given in Table 1 was taken in a beaker. To this 100mL of water maintained at a temperature of 5°C is added and left undisturbed for 5 minutes. After 5 minutes the solution was gently mixed 5 times in clockwise direction and another 5 times in anticlockwise direction using a glass rod. The solution was then slowly decanted to separate out the solid portion. The collected residue was dried at a temperature of 60°C and the weight of the residue was weighed and recorded in table 1.

50 **[0079]** In Example 1, 10 grams of sodium sulphate (hydrate-forming salt) was taken in a plough shear mixer and rotated at high speed, during the mixing 0.05 grams of sulphobetaine was sprayed onto the salt and after 5 minutes of mixing the coated filler agent was obtained and tested for solubility.

**[0080]** In Example 2, the coated filler agent was prepared similar to example 1 and thereafter the sodium chloride was added to obtain the filler composition. Similarly, in Example 3 a different filler composition having sodium chloride and potassium carbonate was obtained.

55 **[0081]** In example B, the preparation of the filler agent was similar to that of Example 1 except that instead of the sulphobetaine, given amount of Neodol EO7 (ethoxylated non-ionic surfactant) was used.

Table 1

Filler agent	Ex A (grams)	Ex 1 (grams)	Ex 2 (grams)	Ex 3 (grams)	Ex B (grams)
Na <sub>2</sub> SO <sub>4</sub>	10	10	10	10	10
sulphobetaine	-	0.05	0.05	0.05	
Non-ionic surfactant	-	-	-	-	0.05
Sodium chloride	-	-	2	1	-
Potassium carbonate	-	-	-	1	-
Residue obtained	9.5	7.3	3.2	2.2	7.3
Nature of residue	Hard	Soft	Very soft and powder like	Very soft and powder like	Hard and similar to example A

**[0082]** The table above shows that best results for solubility when dissolved at 5°C are obtained when the sodium sulphate (hydrate-forming salt) is at least partially coated with the sulphobetaine (Ex 2) according to the present invention. The table also indicates that the solubility of the filler agent is lower and the residue formed is hard in Ex A, and Ex B which has either no coating or coated with a compound outside the scope of the present invention. Further filler composition of Ex 2 and 3 having a coated filler agent and a water-soluble salt shows improved solubility and the nature of residue is also soft.

Example 2: Evaluation of the solubility and dispersibility of solid detergent composition

**[0083]** 3 different solid detergent compositions as shown in Table 2 were evaluated. 100 grams of each of the solid detergent composition was packed in pouches and kept at a temperature of 5°C for 24 hours. Thereafter 100grams of each of the solid detergent composition was poured in the powder-dispensing drawer of Samsung automatic top loading machine. Water maintained at a temperature of 5°C was allowed to contact the powder at the required flow rate as given in Table 2 and then followed by the fuzzy cycle. The residue remaining in powder dispensing drawer at the end of the cycle was taken and dried at a temperature of 60°C for 24 hours. After drying, the residue was weight and the amount of residue was determined as provided in Table 2.

Table 2

Solid detergent composition	Ex C	Ex 4	Ex 5
<b>Spray dried base powder</b>			
Anionic surfactant	12	12	12
Na silicate	6	6	6
Na caboxymethyl cellulose	0.14	0.14	0.14
Na carbonate	11.52	11.52	11.52
Na sulphate	27.67	27.67	27.67
Dyes, polymers, moisture etc.	2.67	2.67	2.67
<b>Post dosed ingredients</b>			
Zeolite	3.64	3.64	3.64
Sodium sulphate	23.82	22.82	18.82
Sodium carbonate	9.48	9.48	9.48
Enzymes, SRPs etc	3.06	3.06	3.06
Sulphobetaine (49% purity)	0	1.0	1.0

(continued)

<b>Post dosed ingredients</b>			
Potassium carbonate	0	0	2.0
Sodium acetate	0	0	2.0
Residue when the flow rate of water at 5°C was maintained at 2.5 litres per minute (grams)	68.88	60.15	Not evaluated at this flow rate
Residue when the flow rate of water at 5°C was maintained at 5 litres per minute (grams)	17.27	Not evaluated at this flow rate	5.76

**[0084]** The table above shows that best results for solubility when dissolved at 5°C are obtained when the sodium sulphate (hydrate-forming salt) is at least partially coated with the sulphobetaine (Ex 4) according to the present invention. Further filler composition of Ex 5 having a coated filler agent and a water-soluble salt shows improved solubility and dispersibility as compared to detergent composition of comparative example C having only the hydrate-forming salt.

**[0085]** It will be appreciated that the illustrated examples provide a coated filler agent having a hydrate-forming salt at least partially coated with the zwitterionic compound provides improved cold water solubility and dispersibility.

**[0086]** It should be understood that the specific forms of the invention herein illustrated and described are intended to be representative only as certain changes may be made therein without departing from the clear teachings of the disclosure.

**[0087]** Although the invention has been described with reference to specific embodiments, it will be appreciated by those skilled in the art that the invention may be embodied in many other forms.

## Claims

1. A coated filler agent for use in a solid detergent composition comprising a hydrate-forming salt and a betaine class of zwitterionic compound wherein the hydrate-forming salt is at least partially coated with the zwitterionic compound.
2. A coated filler agent according to claim 1 wherein the hydrate-forming salt is selected from the group consisting of sulphate, carbonate or bicarbonate salt of alkaline earth metal or alkali metal and mixtures thereof.
3. A coated filler agent as claimed in claim 2 wherein the hydrate-forming salt is selected from sodium carbonate, sodium sulphate or mixtures thereof.
4. A coated filler agent according to any one of the preceding claims wherein zwitterionic compound is a zwitterionic surfactant.
5. A coated filler agent according to claim 4 wherein the zwitterionic surfactant is selected from a carbo-betaine/carboxy-betaine, sulpho-betaine, phospho-betaine, or mixtures thereof.
6. A coated filler agent according to any one of the preceding claims wherein the zwitterionic surfactant is present an amount from 0.5 to 20wt% based on the coated filler agent.
7. A coated filler agent according to any one of the preceding claims wherein the hydrate-forming salt is present in an amount from 80 to 99 wt% based on the coated filler agent.
8. A filler composition comprising a coated filler agent according to any one of the preceding claims and a water-soluble salt with a solubility of more than 30grams/100mL in distilled water when measured at a temperature of 10°C, the water-soluble salt being present separate from the hydrate-forming salt.
9. A filler composition according to claim 8 wherein the water-soluble salt is selected from sodium chloride, sodium formate or sodium acetate.
10. A solid detergent composition comprising a coated filler agent according to any one of the preceding claims 1 to 7 or a filler composition according to claim 8 or 9.

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11. A solid detergent composition according to claim 10 wherein the coated filler agent is in an amount ranging from 2wt% to 40wt% based on the solid detergent composition.
- 5 12. A solid detergent composition according to claim 10 wherein the filler composition is in an amount ranging from 5wt% to 50wt% based on the solid detergent composition.
13. A solid detergent composition according to claim 10 or 11 wherein the composition is a powder, tablet, granular or a particulate composition.
- 10 14. A solid detergent composition according to any one of the claims 10 to 12 wherein the composition comprises 2 wt% to 80wt% anionic surfactant.
- 15 15. Use of a coated filler agent according to any one of the claims 1 to 6 or a filler composition according to claims 8 or 9 in a solid detergent composition for improving cold water solubility and/or dispersibility.

### Patentansprüche

- 20 1. Beschichtetes Füllmittel zur Verwendung in einer festen Reinigungsmittelzusammensetzung, umfassend ein hydratbildendes Salz und eine Betainklasse von zwitterionischer Verbindung, wobei das hydratbildende Salz zumindest teilweise mit der zwitterionischen Verbindung beschichtet ist.
- 25 2. Beschichtetes Füllmittel nach Anspruch 1, wobei das hydratbildende Salz ausgewählt ist aus der Gruppe bestehend aus Sulfat-, Carbonat- oder Bicarbonatsalz von Erdalkalimetall oder Alkalimetall und Mischungen davon.
- 30 3. Beschichtetes Füllmittel nach Anspruch 2, wobei das hydratbildende Salz ausgewählt ist aus Natriumcarbonat, Natriumsulfat oder Mischungen davon.
- 35 4. Beschichtetes Füllmittel nach irgendeinem der vorhergehenden Ansprüche, worin die zwitterionische Verbindung ein zwitterionisches Tensid ist.
- 40 5. Beschichtetes Füllmittel nach Anspruch 4, wobei das zwitterionische Tensid ausgewählt ist aus einem Carbobetain / Carboxybetain, Sulfobetain, Phosphobetain oder Mischungen davon.
- 45 6. Beschichtetes Füllmittel nach irgendeinem der vorhergehenden Ansprüche, wobei das zwitterionische Tensid in einer Menge von 0,5 bis 20 Gew.-%, bezogen auf das beschichtete Füllmittel, vorliegt.
- 50 7. Beschichtetes Füllmittel nach irgendeinem der vorhergehenden Ansprüche, wobei das hydratbildende Salz in einer Menge von 80 bis 99 Gew.-%, bezogen auf das beschichtete Füllmittel, vorliegt.
- 55 8. Füllstoffzusammensetzung, umfassend ein beschichtetes Füllmittel nach irgendeinem der vorhergehenden Ansprüche und ein wasserlösliches Salz mit einer Löslichkeit von mehr als 30 Gramm/100 mL in destilliertem Wasser, gemessen bei einer Temperatur von 10 °C, wobei das wasserlösliche Salz gesondert von dem hydratbildenden Salz vorliegt.
9. Füllstoffzusammensetzung nach Anspruch 8, wobei das wasserlösliche Salz ausgewählt ist aus Natriumchlorid, Natriumformiat oder Natriumacetat.
10. Feste Reinigungsmittelzusammensetzung, umfassend ein beschichtetes Füllmittel nach irgendeinem der vorhergehenden Ansprüche 1 bis 7 oder eine Füllstoffzusammensetzung nach Anspruch 8 oder 9.
11. Feste Reinigungsmittelzusammensetzung nach Anspruch 10, wobei das beschichtete Füllmittel in einer Menge im Bereich von 2 Gew.-% bis 40 Gew.-%, bezogen auf die feste Reinigungsmittelzusammensetzung, vorliegt.
12. Feste Reinigungsmittelzusammensetzung nach Anspruch 10, wobei die Füllstoffzusammensetzung in einer Menge im Bereich von 5 Gew.-% bis 50 Gew.-%, bezogen auf die feste Reinigungsmittelzusammensetzung, vorliegt.
13. Feste Reinigungsmittelzusammensetzung nach Anspruch 10 oder 11, wobei die Zusammensetzung eine Pulver-,

Tabletten-, Granulat- oder eine teilchenförmige Zusammensetzung ist.

14. Feste Reinigungsmittelzusammensetzung nach irgendeinem der Ansprüche 10 bis 12, wobei die Zusammensetzung 2 Gew.-% bis 80 Gew.-% anionisches Tensid umfasst.

15. Verwendung eines beschichteten Füllmittels nach irgendeinem der Ansprüche 1 bis 6 oder einer Füllstoffzusammensetzung nach Anspruch 8 oder 9 in einer festen Reinigungsmittelzusammensetzung zur Verbesserung der Kaltwasserlöslichkeit und/oder -dispergierbarkeit.

## Revendications

1. Agent de charge revêtu pour une utilisation dans une composition de détergent solide comprenant un sel formant un hydrate et une classe de bétaïne de composé zwitterionique dans lequel le sel formant un hydrate est au moins partiellement revêtu avec le composé zwitterionique.

2. Agent de charge revêtu selon la revendication 1, dans lequel le sel formant un hydrate est choisi dans le groupe consistant en sel de sulfate, carbonate ou bicarbonate de métal alcalino-terreux ou métal alcalin et mélanges de ceux-ci.

3. Agent de charge revêtu selon la revendication 2, dans lequel le sel formant un hydrate est choisi parmi le carbonate de sodium, sulfate de sodium et mélanges de ceux-ci.

4. Agent de charge revêtu selon l'une quelconque des revendications précédentes, dans lequel le composé zwitterionique est un tensioactif zwitterionique.

5. Agent de charge revêtu selon la revendication 4, dans lequel le tensioactif zwitterionique est choisi parmi une carbo-bétaïne/carboxy-bétaïne, sulfo-bétaïne, phospho-bétaïne, ou des mélanges de celles-ci.

6. Agent de charge revêtu selon l'une quelconque des revendications précédentes, dans lequel le composé zwitterionique est présent dans une quantité de 0,5 à 20 % en masse sur la base de l'agent de charge revêtu.

7. Agent de charge revêtu selon l'une quelconque des revendications précédentes, dans lequel le sel formant un hydrate est présent dans une quantité de 80 à 99 % en masse rapporté à l'agent de charge revêtu.

8. Composition de charge comprenant un agent de charge revêtu selon l'une quelconque des revendications précédentes et un sel soluble dans l'eau avec une solubilité supérieure à 30 grammes/100 ml dans de l'eau distillée lorsque mesurée à une température de 10°C, le sel soluble dans l'eau étant présent séparé du sel formant un hydrate.

9. Composition de charge selon la revendication 8, dans laquelle le sel soluble dans l'eau est choisi parmi le chlorure de sodium, formate de sodium ou acétate de sodium.

10. Composition de détergent solide comprenant un agent de charge revêtu selon l'une quelconque des revendications 1 à 7 précédentes ou une composition de charge selon la revendication 8 ou 9.

11. Composition de détergent solide selon la revendication 10, dans laquelle l'agent de charge revêtu se trouve dans une quantité de 2 % en masse à 40 % en masse sur la base de la composition de détergent solide.

12. Composition de détergent solide selon la revendication 10, dans laquelle la composition de charge se trouve dans une quantité de 5 % en masse à 50 % en masse sur la base de la composition de détergent solide.

13. Composition de détergent solide selon la revendication 10 ou 11, dans laquelle la composition est une poudre, un comprimé, granulaire ou une composition particulière.

14. Composition de détergent solide selon l'une quelconque des revendications 10 à 12, dans laquelle la composition comprend de 2 % en masse à 80 % en masse de tensioactif anionique.

15. Utilisation d'un agent de charge revêtu selon l'une quelconque des revendications 1 à 6 ou d'une composition de

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charge selon la revendication 8 ou 9 dans une composition de détergent solide pour améliorer la solubilité et/ou dispersibilité dans l'eau froide.

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**REFERENCES CITED IN THE DESCRIPTION**

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