(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 2 392 638 B1

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
18.10.2017 Bulletin 2017/42

(51) Int Cl.:
C11D 3/33 *(2006.01)* ***C11D 3/08*** *(2006.01)*
C11D 11/02 *(2006.01)*

(21) Application number: **10164975.4**

(22) Date of filing: **04.06.2010**

(54) **Low-hygroscopic particulate composition comprising one or more aminopolycarboxylate chelating compounds**

Partikelzusammensetzung mit geringer Hygroskopie und mit einem oder mehr aminopolycarboxylat-chelatbildenden Verbindungen

Composition particulaire faiblement hygroscopique comprenant un ou plusieurs composés chélateurs d'aminopolycarboxylate

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

(43) Date of publication of application:
07.12.2011 Bulletin 2011/49

(73) Proprietor: **Dalli-Werke GmbH & Co. KG**
52224 Stolberg (DE)

(72) Inventors:
- **De Boer, Robbert**
  8167 PH Oene (NL)
- **Mol, René**
  7913 TL Hollandscheveld (NL)
- **Van Ommen, Janco**
  8031 EZ Zwolle (NL)

(74) Representative: **f & e patent**
Fleischer, Engels & Partner mbB, Patentanwälte
Braunsberger Feld 29
51429 Bergisch Gladbach (DE)

(56) References cited:
WO-A1-2009/103822
WO-A1-2010/076291
BE-A1- 779 030
US-A- 3 697 453
US-A- 3 717 589

- **DATABASE WPI Week 199727 Thomson Scientific, London, GB; AN 1997-294927 XP002610219, -& JP 9 110813 A (NIPPON SHOKUBAI CO LTD) 28 April 1997 (1997-04-28)**

Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

EP 2 392 638 B1

Description

[0001] The present invention relates to a particulate composition, wherein at least a part of the particles forming said composition comprises both (i) an aminopolycarboxylate chelating compound A and (ii) (a) a further aminopolycarboxylate chelating compound B, which has a chemical structure different from the chelating compound A, or (b) a mixture of said aminopolycarboxylate chelating compound B and a silicate having a defined structure.

[0002] For many years phosphates like for example sodium tripolyphosphate and tetrasodium pyrophoshate have been used for reducing staining and/or scale deposition resulting from hard water. Due to environmental reasons, in particular concerns of eutrophication, however, these sequestrants/builders are no longer permitted in many countries. Accordingly, attempts have been made to replace phosphates by other sequestrants and/or builders, such as for example ethylenediaminetetraacetic acid (EDTA) and nitrilotriacetic acid (NTA). EDTA, however, is not biodegradable, while NTA is suspected of causing cancer. For this reason, efforts have been made to provide further chelating agents having a chelating power comparable to EDTA or NTA without having their toxicological or environmental drawbacks.

[0003] Glutamic acid-*N,N*-diacetic acid (GLDA), for example, is a strong chelating agent, its chelating power being comparable to EDTA and NTA, while - in contrast to the latter - not being considered carcinogenic. In addition, GLDA is biodegradable and can be produced from a natural sustainable source. A drawback of GLDA on the other hand is the fact that GLDA as well as its alkali metal and ammonium salts are considerably hygroscopic, which complicates their use particularly in solid cleaning and detergent compositions, such as for example compositions for laundry or automatic dishwashing.

[0004] Especially particulate hygroscopic compositions may absorb a large amount of water from the atmosphere, thus leading to caking of said particles during manufacturing, packing, storage and/or dosing by the user. In addition, handling hygroscopic substances it is rather difficult to ensure a constant water level in the final product.

[0005] In the past, several ways for solving the problem of incorporating hygroscopic substance into particulate solid compositions have been proposed. For example, a powder or granulate, mainly consisting of the hygroscopic substance, may be coated with a non-hygroscopic substance, thus impeding the uptake of water by the (now coated) hygroscopic substance. This approach solves the problem associated with hygroscopicity, however, coating is an expensive process, and thus production costs significantly rise.

[0006] In a further approach, such as in WO2009103822, solutions of the hygroscopic substance have been sprayed on carrier particles. Even though this is a comfortable way of incorporating non-solid and/or highly hygroscopic substances into a formulation, the problem associated with hygroscopicity usually is increased by doing so, as the hygroscopic substance is presented in a finely divided manner on a rather large surface. In any case, the percentage of the hygroscopic substance which can be incorporated into a solid composition by this way is limited and usually does not exceed an amount of about five weight percent, based on the formulation.

[0007] For these reasons, a solid particulate composition comprising a rather high amount of one or more aminopolycarboxylate chelating compounds would be desirable i.e comprising a high amount of ingredients having a complexing ability for divalent cations, wherein the aminopolycarboxylate chelating compound(s) is/are formulated in such a way, that the particulate composition has a relative slow moisture uptake or a relative low maximum moisture uptake or both during packing, storage and/or dosing in order to avoid caking of the particulate substance and ensure the flowability of the particulate substance over a long time. Preferably, the maximum moisture uptake of a particulate composition of the present invention should not exceed 80 wt.-% at 37 $\pm$ 1 °C and 75 $\pm$ 2 % relative humidity, based on a particulate composition having an initial water content of composition preferably should be below 12 % (w/w) per hour at 37 $\pm$ 1 °C and 75 $\pm$ 2 % relative humidity.

[0008] Accordingly, it was an object of the present invention to provide a particulate composition comprising at least 5 % (w/w) of a hygroscopic aminopolycarboxylate chelating compound A, wherein the particulate composition as whole has a relative slow moisture uptake or a relative low maximum moisture uptake or both of water from the atmosphere during packing, storage, and/or further processing of the particulate composition, including handling and dosing by the user. In terms of the present invention a relative slow moisture uptake is defined as being below 12 % (w/w) per 1 hour at 37 +/- 1 °C and 75 +/- 2 % relative humidity. A relative low maximum moisture uptake is defined as being a maximum uptake 80% (w/w) at 37 +/- 1 °C and 75 +/- 2 % relative humidity.

[0009] This problem was solved by the composition of the present invention. The present invention provides a particulate composition wherein at least a part of the particles present in said composition comprises

(i) at least 5% (w/w) of a hygroscopic aminopolycarboxylate chelating compound A and
(ii)

(a) a further aminopolycarboxylate chelating compound B, which has a chemical structure different from the chelating agent A, compounds A and B independently are selected from the group comprising 2-(hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), diethylenetriaminepentacetic acid (DTPA), methylglycine-*N,N*-

diacetic acid (MGDA), glutamic acid-*N,N*-diacetic acid (GLDA), iminodisuccinic acid (IDS), hydroxyiminodisuccinic acid (HIDS), ethylenediaminedisuccinic acid (EDDS), aspartic acid-*N,N*-diacetic acid (ASDA), salts thereof, or mixtures thereof with the proviso that if the aminopolycarboxylate chelating compound A is GLDA and no silicate is present in the composition, the aminopolycarboxylate chelating compound B is not MGDA or

(b) a mixture of said aminopolycarboxylate chelating compound B and a silicate, wherein the silicate is an alkali metal silicate of the general formula (III)

$$M_2O:nSiO_2 \qquad (III)$$

wherein M represents an alkali metal and n is in the range of from 1 to 3.5, and

wherein the combined amount of aminopolycarboxylate chelating compound(s) in said particles particles is equal to or above 40 % (w/w), based on the weight of the particles, and the amount of silicate ranges from 0 to 40 % (w/w), based on the weight of said particles,

and wherein the ratio of the amount of silicate to the amount of combined aminopolycarboxylate chelating compounds, both based on the weight of the particles, is less than 0.75 and

wherein the hygroscopicity of compound B is less than the hygroscopicity of compound A.

[0010] It has surprisingly been found, that a high amount of even a hygroscopic aminopolycarboxylate A can be incorporated into a stable, i.e. essentially low-hygroscopic, particulate composition, if said aminopolycarboxylate A is provided as a co-granulate comprising an appropriate amount of a aminopolycarboxylate B or a mixture of said aminopolycarboxylate B and silicate. The percentage of aminopolycarboxylate A in the co-granulate is at least 5% (w/w). The percentage of said co-granulate in the particulate composition is at least 20%, based on the whole particulate composition.

[0011] The amounts of aminopolycarboxylate B or the mixture of silicate and aminopolycarboxylate B in the co granulate containing aminopolycarboxylate A do affect the maximum moisture uptake and/or the speed of moisture uptake of the co-granulate. Optimizing the composition of the co granulate results in a co-granulate with a rather low hygroscopic character that can be blended in a stable, particulate composition without facing significant problems due to the hygroscopic properties of aminopolycarboxylate A.

[0012] The composition of the present invention has slow moisture uptake and/or a rather low maximum moisture uptake. In addition, not only the hygroscopic aminopolycarboxylate chelating compound A, but also compound B and the silicate which are added for controlling the moisture uptake of the composition, possess chelating ability.

[0013] In terms of the present invention a particulate composition is a composition comprising a plurality of rather small solid "objects" (particles) which are in principle free to move against each other, such as in particular a powder or a granulate. The mean particle size preferably may be in the range of from 0.4 mm to 2.0 mm. Preferably, not more than 10 % (w/w) of the particles in the particulate composition and in particular the particles, comprising at least compound A and at least one of compound B and a silicate, have a particle size below 0.4 mm. Preferably, not more than 10 % of the particles have a particle size above 2.0 mm, when determined by a sieve analysis on a Retsch Sieve Shaker AS200 during 5 minutes at an amplitude of 1 mm.

[0014] At least a part of the particles present in the particulate composition comprises both an (i) aminopolycarboxylate chelating compound A and at least one additional compound, selected from the group consisting of (ii) (a) a further aminopolycarboxylate chelating compound B, which has a chemical structure different from the chelating agent A as defined in the claims, or (b) a mixture of said aminopolycarboxylate chelating compound B as defined in the claims and a silicate. Said particles, comprising at least compound A and at least one of compound B or a mixture of compound B with a silicate, preferably comprise an intimate mixture of said compounds A and B or of said compounds A, B and said silicate.

[0015] Preferably, said compounds may be homogenously dispersed within the particles comprising these compounds. Particles comprising these compounds preferably may be non-coated particles.

[0016] Most preferably, at least 99.9 % (w/w) of the particles present in the particulate composition of the present invention comprise both compound A and compound B or a mixture of compounds A, B and the silicate in an amount as defined above. The ratio of the amount of silicate, given in weight percent based on the whole composition, to the amount of (combined) aminopolycarboxylate chelating compound(s), both based on the weight of the composition, should be less than 0.75, more preferably less than 0.5, even more preferably less than 0.3 and most preferably less than 0.25, being for example 0.2.

[0017] If two different aminopolycarboxylate chelating compounds A and B are present in at least a part of the particles present in said composition, the presence of a silicate is not necessary. If the particles in the particulate composition of the present invention comprises a silicate, said particles should comprise said silicate in an amount of from above 5 to 95 % (w/w, based on the weight of said particles).

[0018] It is, however, preferred that silicate is present in said particles in an amount of 5% up to 40% (w/w), if said particles comprise a mixture of at least two different aminopolycarboxylate chelating compounds A and B.

[0019] Preferably, said particles, comprising compound A and compound B and/or a silicate, do not comprise EDTA, NTA and/or a phosphate. Preferably, said particles do not comprise any of the aforementioned substances. More preferably, the whole particulate composition according to the present invention does not comprise any of these substances, even if the amount of particles, which comprise at least compound A and at least one of compound B or a mixture of compound B and a silicate, in said particulate composition is less than 100% (w/w).

[0020] Preferably, the (combined) amount of aminopolycarboxylate chelating compound(s) in said particles may be equal to or above 40 % (w/w), more preferably above 50 % (w/w), even more preferably above 60 % (w/w), even more preferably above 65 % (w/w) and most preferably in the range of from 65 to 95 %, based on the weight of the particles.

[0021] According to the invention, compounds A and B independently are selected from the group comprising 2-(hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), diethylenetriaminepentacetic acid (DTPA), methylglycine-*N,N*-diacetic acid (MGDA), glutamic acid-*N,N*-diacetic acid (GLDA), iminodisuccinic acid (IDS), hydroxyiminodisuccinic acid (HIDS), ethylenediaminedisuccinic acid (EDDS), aspartic acid-*N,N*-diacetic acid (ASDA), salts thereof, or mixtures thereof with the proviso that if the aminopolycarboxylate chelating compound A is GLDA and no silicate is present in the composition, the aminopolycarboxylate chelating compound B is not MGDA.

[0022] It may particularly be preferred that the hygroscopicity of compound B may be less than the hygroscopicity of compound A and that the ratio of the amount of compound B, given in weight percent based on the particulate composition, to the amount of compound A (B/A) may be equal to or less than 20, preferably equal to or less than 16, more preferably equal to or less than 12 and most preferably may be in the range of from 9 to 3, including 8, 7, 6, 5 and 4.

[0023] Preferably compound A may represent GLDA or a salt thereof, in particular the sodium salt, and compound B may preferably represent IDS or a salt thereof, in particular the sodium salt thereof.

[0024] The particles preferably may comprise of from 5 to 50 % (w/w) of compound A and of from 40 to 85% (w/w) of compound B, both based on the weight of said particles. The total amount of compound A and B in the particles probably may sum up to from 45 to 90 % (w/w). WO2011/079940 discloses a particulate composition comprising a core of glutamic acid-N,N-diacetic acid (GLDA) its salt and/or derivative and a shell of a further aminopolycarboxylate such as methylglycine-N,N-diacetic acid (MGDA) or its alkali metal, ammonium or substituted amino-salt. If the particles in the particulate composition of the present invention comprise a silicate, said silicate is an alkali metal silicate of the general formula (III)

$$M_2O{:}nSiO_2 \qquad (III)$$

wherein n is in the range of from 1 to 3.5, preferably of from 1.6 to 2.6, more preferably of from 1.8 to 2.2 and the alkali metal preferably is selected from the group consisting of lithium, sodium, potassium, and mixtures thereof. Preferably, composition of the present invention preferably an aqueous solution of said silicate is used.

[0025] The amount of water in the particles of the particulate composition of the present invention preferably is in the range of from 5 to 15 % (w/w), based on the weight of the particles. As one example for the particulate composition of the present invention said composition may consists of particles containing an aminopolycarboxylate chelating compound A in an amount of from 5 to 20 % and an aminopolycarboxylate chelating compound B in an amount of from 45 to 70 % (w/w), based on the weight of the composition. Herein, compounds A and B preferably represent the compounds already described above, being present in a combined amount of about 60 to 90 % (w/w), based on the weight of the particles. A disilicate may be present in an amount of from 10 to 25 wt.-%, the combined amount of both aminopolycarboxylate chelating compounds A and B and the disilicate summing up to about 90 to 100 %, wherein the remainder is water plus some additional components in minor amounts.

[0026] On the other hand, the particles or the composition may as well comprise at least one further component selected from the group consisting of surfactants and organic polymers, preferably selected from the group consisting of non-ionic surfactants and organic polymers, most preferably selected from the group consisting of polyvinylalcohol, polyvinylpyrrolidones, polyvinylacetate, polyalkyleneglycols or a mixture thereof or co polymers thereof, like for example a polyvinylalcohol-polyethylene graft copolymer. As an alternative or in addition the particles may as well comprise at least one alkalinity source, preferably selected from the group consisting of carbonate salts, hydroxide salts, phosphonate salts, more preferably alkali and/or ammonium salts thereof.

[0027] The present invention also relates to a method for preparing the particulate composition of the present invention which comprises the steps of

(I) mixing the ingredients, comprising at least compound A, and at least one of compound B or a mixture of compound B and a silicate,
(II) spray-drying the mixture, and
(III) (optionally) granulating the obtained mixture,

wherein preferably at least compounds A and B are present at least temporarily in a liquid form during the mixing procedure, more preferably in the form of an aqueous solution.

[0028] In order to ensure an intimate mixture of the compounds present in the particles of the particulate composition, at least compound A and compound B should be present at least temporarily in a liquid form during the mixing procedure. For example, the inventive particulate composition may be prepared by mixing its ingredients in the solid form, for example in the form of powder, and then adding a suitable solvent, preferably water, in an amount sufficient to dissolve or at least to roughly suspend at least compound A and compound B.

[0029] At least compound A and compound B may as well be provided in a liquid form, preferably in the form of an aqueous solution, before combining it/them with one another and/or any further ingredients which may be present in a solid or in a liquid form. If present, the silicate preferably may be provided in a liquid form, preferably in the form of an aqueous solution as well.

[0030] In a particular preferred embodiment of the method of the present invention solutions of compound A, compound B and the silicate are mixed. A particularly preferred particular composition of the present invention may, for instance, be prepared by mixing about 10 to 25 % (w/w), based on the whole mixture, of an aqueous solution of compound A, comprising about 30 to 50 % (w/w), based on said aqueous solution, of compound A, about 40 to 80 % (w/w), based on the whole mixture, of an aqueous solution of compound B, comprising about 25 to 45 % (w/w), based on said aqueous solution, of compound B, and about 5 to 25 % (w/w), based on the whole mixture, of an aqueous solution of the silicate, comprising about 20 to 50% (w/w), based on said aqueous solution, of the silicate.

[0031] After mixing the ingredients, the mixture is dried, preferably spray-dried. Optionally, the dried mixture may then be granulated in a granulation and/or melt granulation step.

[0032] The inventive particulate composition(s) may be used as ingredient in detergent and/or cleaning formulations as well as for the manufacture of said detergent and/or cleaning formulations and for cleaning and/or washing articles, preferably in automatic laundering of textiles or dishwashing.

[0033] Accordingly, the present invention also relates to a cleaning and/or detergent formulation comprising the particulate composition of the present invention. Preferably, the cleaning and/or detergent formulation comprises of from 0.5 to 80 % (w/w), based on the weight of the formulation, of the inventive particulate composition.

[0034] The particulate composition preferably may be used for the manufacture of and/or may be comprised in any formulation commonly comprising a chelating agent, preferably any cleaning and/or detergent formulation, more preferably a detergent formulation for laundering or dishwashing, more preferably for automatic laundering or dishwashing. Ingredients of such preferred automatic laundering/dishwashing formulations are shown in detail below. In principle, the cleaning and/or detergent formulations may be of any form known in the state of the art, including solids, melts, liquids, gels and pastes. It may, however, be preferred to provide the formulation in form of a solid composition, including a powder, granulates, a tablet, a bar, a block, pellets, balls, pearls or any other suitable solid form or combinations thereof. Preferably, the formulation is provided in the form of tablets and granules.

[0035] Detergent tablets of the present invention may be monophase, as well as multiphase tablets. The different phases in a multiphase tablet may comprise partly or completely different ingredients. As well they may have different colors. The different phases in such a multiphase tablet represent discrete regions of the tablet and may have the form of layers, one or more cores or inserts, stripes, dot(s), strand(s), extruded line(s), or a pattern in or on another portion of the detergent formulation or a coating on or around at least a part of the surface of the tablet.

FURTHER INGREDIENTS

[0036] The following further ingredients may be included in the particulate chelating composition of the present invention, or may be ingredients of the cleaning and/or detergent formulation combined with or manufactured using the particulate composition of the present invention. The ingredients below can be combined in any suitably manner, e.g. some of them might be included in the blend of the particulate composition, while some of them might be part of the cleaning and/or detergent formulation or several of them might be included in a cleaning and/or detergent formulation, while the particulate chelating composition does not comprise any further ingredient.

[0037] Preferably, the particulate chelating composition of the present invention is provided in combination with and/or as a part of a cleaning and/or detergent composition. If the particulate chelating composition is combined with a cleaning, washing and/or detergent composition, the amount of particulate chelating composition in a cleaning and/or detergent composition preferably is in the range of from 10% to 50%.

[0038] The cleaning and/or detergent formulation(s) of the present invention may comprise any of the ingredients known in the art as common ingredients in cleaning and/or detergent compositions, preferably in automatic laundering and/or dishwashing compositions. Said ingredients are e.g. (further) surfactants, builders, chelants, complexing agents, bleaching agents, bleach activators, bleach catalysts, optical brighteners, rinse aid additives, corrosion inhibitors, anti-redeposition agents, enzymes, dispersing agents, pH modifiers, colorants, dyes, perfume, without being limited to these. Preferably the cleaning and /or detergent formulation(s) of the present invention is/are provided as a tablet, powder, or a granulate.

[0039] Furthermore all of the optional ingredients known in the state of the art to be effective or usable in detergent

compositions might be included.

[0040] The following ingredients are not limiting the present invention.

BUILDERS

[0041] The composition of the present invention may optionally comprise one or more additional builder(s).

[0042] The main functions of the builders are to soften the washing water, to provide alkalinity and a buffering capacity to the washing liquid and to have an anti redeposition or dispersing function in the detergent composition. The physical properties of the detergent composition are also depending on the builders that are used.

[0043] Inorganic non-phosphate builders include, but are not limited to, phosphonates, silicates, carbonates, sulphates, citrate, and aluminosilicates.

[0044] Organic builders include, but are not limited to, a wide variety of (poly)carboxylated compounds having one or more carboxylate groups.

[0045] Phosphoric builders include, but are not limited to, various alkali metal phosphates such as tripolyphosphate, pyrophosphate, orthophosphate, etc. However, as already mentioned above, preferably the cleaning and/or detergent formulation of the present invention preferably is free of any phosphates.

[0046] Complexing agents are commonly used as co-builders to support the performance of the builders.

[0047] Builders and co-builders can generally be added to the composition in acid form, neutralized or in a partly neutralized form. When used in a partly or completely neutralized form alkali metal salts are preferred, like sodium, potassium and lithium or alkyl ammonium salts.

SURFACTANTS

[0048] The composition of the present invention may optionally comprise one or more surfactants.

[0049] The main functions of surfactants are changing the surface tension, dispersing, foam controlling and surface modification. A special type of surfactants used in automatic dishwasher detergent compositions is a 'carry-over' surfactant. In a 'carry-over' surfactant some amount of the surfactant used remains in the machine after the rinsing cycles to give a performance during the final rinsing cycle and the (optional) drying phase of the whole washing cycle of the dishwashing machine. This type of surfactant is described in EP 1 524 313 in more detail.

[0050] For automatic dishwasher detergent compositions alkoxylated nonionic surfactants and Gemini surfactants are commonly used. The alkoxy groups mostly consist of ethyleneoxide, propyleneoxide and butyleneoxide or combinations thereof. Also amphoteric surfactants are known to be used in automatic dishwasher detergent compositions.

[0051] Alkyl poly glucoside (APG) surfactants can also be used in automatic dishwasher detergent compositions, preferably in a low foaming form.

[0052] Further all surfactants commonly known to be used in detergent compositions can be part of the composition. This includes all anionic, non-ionic, cationic and amphoteric surfactants known in the art. The present invention is not limited by any of the surfactants commonly used in automatic dishwashing compositions.

ENZYMES

[0053] The composition of the present invention may optionally comprise one or more enzymes.

[0054] Enzymes are often used to aid the removal of stains. In most cases enzymes react with the soiling and break it down into particles that have an increased water solubility or are better dispersible in the washing liquid.

[0055] The enzymes that can be used in cleaning and/or detergent formulations include, but are not limited to, proteases, amylases, lipases, cellulases, mannanase, peroxidase, oxidase, xylanase, pullulanase, glucanase, pectinase, cutinase, hemicellulases, glucoamylases, phospholipases, esterases, keratanases, reductases, phenoloxidases, lipoxygenases, ligninases, tannases, pentosanases, malanases, arabinosidases, hyaluronidase, chondroitinase, laccase or mixtures thereof. These enzymes are known to the skilled artisans and can be used as a granulate and a liquid in common amounts.

ANTI CORROSION AGENTS

[0056] The cleaning and/or detergent composition of the present invention may optionally comprise one or more anticorrosion agents.

[0057] The main function of anticorrosion agents is to minimize the amount of material damage caused on glass and metal during automatic dishwashing.

[0058] Glass corrosion occurs because metal ions are dissolved out of the glass surface. This occurs more intensively when soft tap water is used for the cleaning. In this case the builders and complexing agents can only bind a limited amount of ions responsible for the water hardness from the tap water. In turn, they extract (alkaline earth) metals from

the glass surface. Also of influence for glass corrosion are the washing temperature, the quality of the glassware and the duration of the cleaning program.

[0059] Glass corrosion becomes visible in white lines or white clouds on the glass surface. The glass corrosion damage can be repaired by replacing the extracted metal ions, however preferably the glassware can be protected against glass corrosion.

[0060] Metal corrosion occurs in many cases when oxide, sulphide and/or chlorides are present in the washing liquid, which normally is a mixture of tap water, soil and a detergent composition. The anions react with the metal or metal alloy surface of articles that are contained in the dishwashing machine. In the case of silver the silver salts which are formed give a discoloration of the silver metal surface which becomes visible after one or more cleaning cycles in an automatic dishwashing machine.

[0061] The occurrence of metal corrosion can be slowed down or inhibited by use of detergent ingredients that provides the metal with a protective film or ingredients forming compounds with the oxide, sulfide and/or chlorides to prevent them from reacting with the metal surface.

[0062] The protective film can be formed because the inhibitor ingredient may become insoluble on the metal or metal alloy surface, or because of adsorption to the surface by aid of free electron pairs of donor atoms (like N, S, O, P). The metals can be silver, copper, stainless steel, iron, etc.

[0063] Anti corrosion agents which often are used in detergent compositions or which are described in literature include, but are not limited to, triazole-based compounds (like tolyltriazole and 1,2,3-benzotriazole), polymers with an affinity to attach to glass surfaces, strong oxidizers (like permanganate), cystine (as silver-protector), silicates, organic or inorganic metal salts, or metal salts of biopolymers. The metal of these metal salts can be selected from the group aluminum, strontium, barium, titanium, zirconium, manganese, lanthanum, bismuth, zinc, wherein the latter two are most commonly applied for the prevention of glass corrosion. Further compounds to be added e.g. are manganese compounds as described e.g. in WO 2005/095570.

POLYMERS

[0064] The composition of the present invention may optionally comprise one or more polymers.

[0065] The main function of polymers is to act as a (co-)builder or dispersing agent. Dispersing agents are used to inhibit crystal growth and/or to disperse insoluble materials in the washing liquor, such as (fatty) soil, inorganic or organic salts, etc. Dispersing agents often have a polymeric character and are at least partly hydrophilic. Dispersing agents are e.g. described in particular in DE 199 34 704 A1.

[0066] The polymers that often are used in cleaning and/or detergent compositions include, but are not limited to, homo-, co- or terpolymers of or based on oleic monomer, acrylic acid, methacrylic acid or maleic acid, or "salts" thereof which are obtained by neutralizing the acidic moieties present in these polymers, either completely or in part. Such polymers can be combined with or can include monomers that give the polymer a special function like improved dispersing properties, improved water solubility, etc..

These polymers often also contain monomers with various properties, like e.g. sulphonated styrene, styrene, 2-acrylamido-2-methyl propane sulphonic acid (AMPS), methallyl sulphonic acid, acryl amide, etc. Such polymers are commonly known and are described e.g. in EP-A 1 363 986, EP-A 1 268 729, EP-A1 299 513 and EP-A 0 877 002.

COMPLEXING AGENTS

[0067] The composition of the present invention may optionally comprise one or more additional complexing agent(s).

[0068] A function of complexing agents is to capture trace metal ions like Cu(II), Fe(II), Fe(III), Mn(II), Cd(II), Co(II), Cr(III), Hg(II), Ni(II), Pb(II), Pd(II), Zn(II). These ions can interfere with or disturb certain processes of the detergent in the washing machine, like e.g. the bleach performance. Complexing agents can also be used as co-builder or builder.

[0069] The complexing agent(s) that are known to be used in cleaning and/or detergent compositions include, but are not limited to *S,S*-ethylenediamine-*N,N'*-disuccinic acid (*S,S*-EDDS), ethylenediaminetetraacetic acid (EDTA), diethylenetriamine penta(methylenephosphonate) (DETPMP), nitrilotriacetic acid (NTA), ethanol diglycine (EDG), iminodisuccinic acid (IDS), methylglycine-*N,N*-diacetic acid (MGDA), diethylenetriaminepentaacetic acid (DTPA), ethylenediaminedihydroxyphenyl acetic acid (EDDHA), N-(hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), hydroxyethylidene-1,1-diphosphonic acid (HEDP), phytic acid, diethylene triamine (DETA), triethylene tetramine (TETA), tetraethylene pentamine (TEPA), aminoethyl ethanolamine (AEEA), glutamic acid-*N,N*-diacetic acid (GLDA), 1,3-propylenediamine tetraacetic acid (PDTA), glucoheptonic acid, dipicolinic acid, ethylene diamine tetra (methylene phosphonic acid) (EDTMPA), 2-hydroxyethyliminodiacetic acid (HEIDA) or water soluble salts thereof or mixtures thereof. As already mentioned above, the compositions of the present invention preferably do not comprise EDTA or NTA.

ANTI-REDEPOSITION AGENTS

[0070] The composition of the present invention may optionally comprise one or more anti-redeposition agents.

[0071] The main function of anti-redeposition agents is the aid to prevent the soil from redepositioning on the washing substrate when a washing liquor provides insufficient soil anti-redeposition capacity.

[0072] Anti-redeposition agent(s) can provide their effect by becoming adsorbed irreversibly or reversibly to the soil particles or to the substrate. Thereby the soil becomes better dispersed in the washing liquor or the substrate is occupied with anti-redeposition agent(s) on those places the soil could redeposit.

[0073] The anti-redeposition agent(s) that are known to be used in detergent compositions include, but are not limited to, carboxymethyl cellulose, polyester-PEG co-polymer, polyvinyl pyrrolidone based polymers etc.

BLEACHING AGENTS

[0074] The composition of the present invention may optionally comprise one or more bleaching agents.

[0075] Bleaching agents can be used in a detergent composition either alone or in combination with a bleach activator and/or a bleach catalyst. The function of the bleaching agent is the removal of bleachable stains and to achieve an antibacterial effect on the load and inside of the (dish)washing machine.

[0076] Bleaching agents commonly used as a sole bleaching ingredient in detergents react with the soil.

[0077] When an inorganic oxygen based bleaching agent is used in combination with a bleach activator it does react with the bleach activator. One of the reaction products provides the actual performance.

[0078] When an inorganic oxygen based bleaching agent is used in combination with a bleach catalyst the catalyst catalyses the oxidation reaction with the substrate. The oxidized bleach catalyst provides the actual bleach performance. A bleach activator can optionally be present.

[0079] Bleaching agents that can be used in cleaning and/or detergent compositions include, but are not limited to, active chlorine compounds, inorganic peroxygen compounds and organic peracids. Examples are sodium percarbonate, sodium perborate monohydrate, sodium perborate tetrahydrate, hydrogen peroxide, hydrogen peroxide based compounds, persulfates, peroxymonosulphate, peroxodisulphate, ε-phthalimido-perox-caproic acid, benzoyl peroxide, sodium hypochlorite, sodium dichloroisocyanurate, etc. as well as mixtures thereof.

BLEACH ACTIVATORS

[0080] The composition of the present invention may optionally comprise one or more bleach activators.

[0081] When inorganic peroxygen based bleaching agents are applied, a bleach activator provides the possibility to use a comparatively low temperature to achieve the desired bleaching performance. The bleach activator reacts with the peroxygen to form an organic peracid. Depending on the used bleach activator these peracids can have a hydrophobic or a hydrophilic character.

[0082] Bleaching agents that can be used in detergent compositions include, but are not limited to, tetraacetylethylenediamine (TAED), sodium nonanoyloxybenzene sulfonate (NOBS), acetyl caprolactone, N-methyl morpholinium acetonitrile and salts thereof, sodium 4-(2-decanoyloxyethoxycarbonyloxy)benzenesulfonate (DECOBS) and salts thereof, etc.

BLEACH CATALYSTS

[0083] The composition of the present invention may optionally comprise one or more bleach catalysts.

[0084] A bleach catalyst can be used besides to or instead of a bleach activator. Most activators used are complexes of transition metal ions with organic ligands. Metal ions that may be applied in catalysts are manganese, iron, copper, cobalt and molybdenum. Complexes including these metals can interact with inorganic and organic peroxygen compounds to form reactive intermediates. The use of a bleach catalyst can result in achieving the desired bleaching performance at an even lower temperature than needed for bleach activators.

[0085] Bleach catalysts that can be used in cleaning and/or detergent compositions are intensively described in the state of the art. These include, but are not limited to a complex of manganese(IV) with 1,4,7-trimethyl-1,4,7-triazacyclononane ($MnMe_3TACN$), tris[2-(salicylideneamino)ethyl]amine manganese(III), siderophore-metal complexes (as described e.g in WO 2008/101909), metal complexes containing ligands of 1,4,7-triazacyclononane (TACN), manganese-protein complexes, etc.

DYES

[0086] The composition of the present invention may optionally comprise one or more dyes. The dye is used to colour

the detergent, parts of the detergent or speckles in the detergent. This might render the product more attractive to the consumer. Dyes that can be used in detergent compositions include, but are not limited to, Nylosan yellow N-7GL, Sanolin brilliant flavine 8GZ, Sanolin yellow BG, Vitasyn quinoline yellow 70, Vitasyn tartrazine X90, Puricolor yellow AYE23, Basacid yellow 232, Vibracolor yellow AYE17, Simacid Eosine Y, Puricolor red ARE27, Puricolor red ARE14, Vibracolor red ARE18, Vibracolor red ARE52, Vibracolor red SRE3, Basacid red 316, Ponceau SX, Iragon blue DBL86, Sanolin blue EHRL, Sanolin turquoise blue FBL, Basacid blue 750, Iragon blue ABL80, Vitasyn blue AE90, Basacid blue755, Vitasyn patentblue V 8501, Vibracolor green AGR25. These dyes are available at the firms Clariant or BASF.

PERFUME

[0087] The composition of the present invention may optionally comprise one or more perfumes. The perfume is added to the detergent to improve the sensorial properties of the product or of the machine load after cleaning.

[0088] The perfume can be added to the detergent as a liquid, paste or as a co-granulate with a carrier material for the perfume. To improve the stability of the perfume it can be used in an encapsulated form or as a complex like for example a perfume-cyclodextrine complex.

[0089] Also perfumes that have a deodorizing effect can be applied. Such perfumes or raw materials encapsulate malodours by binding to their sulphur groups.

PROCESS AIDS

[0090] The composition of the present invention may optionally comprise one or more process aids. Process aids are used to improve certain product or production properties.

[0091] The process aids used in cleaning and/or detergent compositions commonly are used for various purposes often depending of the physical form of the final product. Process aids for example can optimize compressibility, friability, toughness, elasticity, disintegration speed, hygroscopicity, density, free flowing properties, stickiness, viscosity, rheology, etc. of a detergent product in a certain physical shape. Such process aids are widely described in the state of the art.

[0092] Further, the invention relates to the use of a disilicate, preferably a disilicate as described above, for the preparation of a particulate composition according to the present invention. Most notably, using said disilicate it is possible to prepare a particulate composition which comprises particles which comprise at least 15 % (w/w), based on the weight of the particles, of at least one hygroscopic aminopolycarboxylate chelating compound in the form of a relative low-hygroscopic granulate according to the present invention.

Figures

[0093]

Figure 1 shows the speed of moisture uptake of two particulate compositions according to the present invention (granulates 2.2 and 2.3) in comparison to a commercially available granulate comprising the same compound A (granulate 2.1) (example 2).

Figure 2 shows the maximum moisture uptake of said granulates (example 2).

Examples

Example 1: Preparation of co-granulates according to the present invention

[0094] Aqueous solutions of sodium silicate and the aminopolycarboxylate(s) were blended homogeneous. This blend was spray dried into a co-granulate. Said spray-dried co granulate was compactated and broken or grinned to the desired particle size.

[0095] In this example the following raw materials were used: The silicate is an about 30% aqueous solution of sodium disilicate, aminopolycarboxylate A is an about 38% aqueous solution of tetrasodium GLDA (Dissolvine GL-47-S from AkzoNobel), and aminopolycarboxylate B is an about 34% aqueous solution of tetrasodium IDS (Baypure CX100/34 from LANXESS).

[0096] Both co-granulates 1.1 and 1.2 were easy to produce, show an acceptable to good hygroscopicity and contain an amount as high as 15 % of the strong chelating agent aminopolycarboxylate A (GLDA).

[0097] With acceptable to good hygroscopicity it is meant that the co granulate can be handled properly without having to face problems caused by the hygroscopicity of the co granulate. A co granulate with a relative good hygroscopicity has a relative slow moisture uptake, of a maximum of 12 % (w/w) per hour at 37 +/- 1 °C and 75 +/- 2 % relative humidity,

and a relative low maximum moisture uptake, of a maximum of 80% (w/w) at 37 +/- 1 °C and 75 +/- 2 % relative humidity.

[0098] Two co-granulates 1.1 and 1.2 according to the present invention were prepared.

Table 1

Particulate co-granulate	1.1	1.2
Silicate	15	-
Aminopolycarboxylate A -	15	15
Aminopolycarboxylate B	55	68
Water and rest materials	15	17

[0099] All mentioned percentages can have a deviation of up to 10%.

Example 2: Speed of moisture uptake and maximum moisture

[0100] The speed of the moisture uptake and the maximum moisture uptake of co-granulates according to the present invention (2.2 and 2.3) is determined. The composition of the co-granulates are listed below;

Table 2

Particulate co-granulate	(2.1)	(2.2)	(2.3)
Silicate	-	15	-
Aminopolycarboxylate A	75	15	15
Aminopolycarboxylate B	-	55	68
Water and rest materials	25	15	17

[0101] All mentioned percentages can have a deviation of up to 10%. Co-granulates 2.2 and 2.3 are prepared as described in example 1. Co-granulate 2.1 is commercially available as Dissolvine GL-PD-S from LANXESS (Leverkusen, Germany).

[0102] The granulates were placed on a clock watch glass and stored in a climatized room under at 37 +/- 1 °C and 75 +/- 2 % relative humidity. The weight increase was measured several times until a stable weight was reached, ie. no further weight increase could be detected.

[0103] Fig. 1 shows the speed of the moisture uptake and Fig. 2 shows the maximum moisture uptake.

[0104] This test shows that the inventive co-granulate (2.2) and (2.3) have a moisture uptake below 12 % (w/w) per 1 hour storage at 37 +/- 1 °C and 75 +/- 2 % relative humidity, and a maximum moisture uptake is below 80% (w/w) at 37 +/- 1 °C and 75 +/- 2 % relative humidity.

Claims

1. Particulate composition wherein at least a part of the particles present in said composition comprises

(i) at least 5% (w/w) of a hygroscopic aminopolycarboxylate chelating compound A and
(ii)

(a) a further aminopolycarboxylate chelating compound B, which has a chemical structure different from the chelating agent A, compounds A and B independently are selected from the group comprising 2-(hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), diethylenetriaminepentacetic acid (DTPA), methylglycine-*N,N*-diacetic acid (MGDA), glutamic acid-*N,N*-diacetic acid (GLDA), iminodisuccinic acid (IDS), hydroxyiminodisuccinic acid (HIDS), ethylenediaminedisuccinic acid (EDDS), aspartic acid-*N,N*-diacetic acid (ASDA), salts thereof, or mixtures thereof with the proviso that if the aminopolycarboxylate chelating compound A is GLDA and no silicate is present in the composition, the aminopolycarboxylate chelating compound B is not MGDA,
or
(b) a mixture of said aminopolycarboxylate chelating compound B and a silicate, wherein the silicate is an alkali metal silicate of the general formula (III)

$$M_2O:nSiO_2 \qquad (III)$$

wherein M represents an alkali metal and n is in the range of from 1 to 3.5, and
wherein the combined amount of aminopolycarboxylate chelating compound(s) in said particles is equal to or above 40 % (w/w), based on the weight of the particles, and the amount of silicate ranges from 0 to 40 % (w/w), based on the weight of said particles,
and wherein the ratio of the amount of silicate to the amount of combined aminopolycarboxylate chelating compounds, both based on the weight of the particles, is less than 0.75 and
wherein the hygroscopicity of compound B is less than the hygroscopicity of compound A.

2. A composition according to claim 1, wherein compound A represents GLDA or a salt thereof and compound B preferably represents IDS or a salt thereof.

3. A composition according to any of claims 1 to 2, wherein the particles comprise of from 5 to 50% (w/w) of compound A and of from 40 to 85% (w/w) of compound B, both based on the weight of the particles, and the total amount of compound A and B in the particles sums up to 45 to 90 % (w/w).

4. A composition according to any of claims 1 to 3, wherein the alkali metal silicate of the general formula (III) is further **characterized by** n being in the range of from 1.6 to 2.6, preferably of from 1.8 to 2.2 and the alkali metal preferably is selected from the group consisting of lithium, sodium, potassium, and mixtures thereof.

5. A composition according to claim 1 to 4, wherein the composition is a free-flowing composition, preferably a free-flowing powder or granulate and most preferably a free-flowing granulate.

6. A composition according to any of claims 1 to 5, wherein the mean particle size is in the range of from 0.4 to 2.0 mm.

7. A composition according to any of claims 1 to 6, wherein the particles comprise at least one further component selected from the group consisting of surfactants and organic polymers, preferably selected from the group consisting of non-ionic surfactants and organic polymers, most preferably selected from the group consisting of polyvinylalcohol, polyvinylpyrrolidones, polyvinylacetate, polyalkyleneglycols or a mixture thereof or copolymers thereof.

8. A method for preparing a particulate composition according to any of claims 1 to 7 comprising the steps of

(i) mixing the ingredients, comprising at least compound A and compound B or compound B and a silicate,
(ii) spray-drying the mixture, and
(iii) (optionally) granulating the obtained mixture,

wherein preferably at least compounds A and B are present at least temporarily in a liquid form during the mixing procedure, more preferably in the form of an aqueous solution.

9. Use of a particulate composition according to any of claims 1 to 7 or a particulate composition prepared according to claim 8 as an ingredient in detergent and/or cleaning formulations, for the manufacture of said detergent and/or cleaning formulations, and for cleaning and/or washing articles.

10. A cleaning and/or detergent formulation comprising of from 0.5 to 80% (w/w), based on the weight of the formulation, of a particulate composition according to any of claims 1 to 7.

11. A cleaning and/or detergent formulation according to claim 10, wherein the composition is a dishwashing formulation, preferably for automated dishwashers.

12. A cleaning and/or detergent formulation according to claim 11, further comprising at least one further ingredient selected from the group consisting of further surfactants, builders, chelants, complexing agents, bleaching agents, bleach activators, bleach catalysts, optical brighteners, rinse aid additives, corrosion inhibitors, anti-redeposition agents, enzymes, dispersing agents, pH modifiers, colorants, dyes, perfume.

13. Use of a disilicate, preferably a disilicate according to claim 4, for the preparation of a particulate composition according to any of claims 1 to 7.

Patentansprüche

1. Partikelförmige Zusammensetzung, wobei wenigstens ein Teil der Partikel, die in der Zusammensetzung vorliegen, enthält:

   (i) wenigstens 5% (Gew./Gew.) eines hygroskopischen Aminopolycarboxylat-Chelatbildners A und
   (ii)

   (a) ein anderer Aminopolycarboxylat-Chelatbildner B, der eine chemische Struktur aufweist, die sich von dem Chelatbildner A unterscheidet, wobei die Verbindungen A und B unabhängig voneinander ausgewählt sind aus der Gruppe, umfassend 2-(Hydroxyethyl)-ethylendiamintriessigsäure (HEDTA), Diethylentriaminpentaessigsäure (DTPA), Methylglycin-*N,N*-diessigsäure (MGDA), Glutaminsäure-*N,N*-diessigsäure (GLDA), Iminodibernsteinsäure (IDS), Hydroxyiminodibernsteinsäure (HIDS), Ethylendiamindibernsteinsäure (EDDS), Asparaginsäure-*N,N*-diessigsäure (ASDA), Salze davon oder Mischungen davon, unter der Voraussetzung, dass, wenn der Aminopolycarboxylat-Chelatbildner A GLDA ist und kein Silicat in der Zusammensetzung vorliegt, der Aminopolycarboxylat-Chelatbildner B nicht MGDA ist,
   oder
   (b) eine Mischung des Aminopolycarboxylat-Chelatbildners B und eines Silicats, wobei das Silicat ein Alkametallsilicat der allgemeinen Formel (III)

   $$M_2O:nSiO_2 \qquad (III)$$

   ist, wobei M ein Alkalimetall darstellt und n in dem Bereich von 1 bis 3,5 liegt, und
   wobei die kombinierte Menge des/der Aminopolycarboxylat-Chelatbildner(s) in diesen Partikeln gleich oder oberhalb 40% (Gew./Gew.), bezogen auf das Gewicht der Partikel, liegt, und die Menge an Silicat von 0 bis 40% (Gew./Gew.), bezogen auf das Gewicht dieser Partikel, reicht,
   und wobei das Verhältnis der Menge an Silicat zu der kombinierten Menge der Aminopolycarboxylat-Chelatbildner, beides bezogen auf das Gewicht der Partikel, unterhalb von 0,75 liegt und
   wobei die Hygroskopie der Verbindung B kleiner ist als die Hygroskopie der Verbindung A.

2. Eine Zusammensetzung gemäß Anspruch 1, wobei die Verbindung A GLDA oder ein Salz davon darstellt und die Verbindung B bevorzugt IDS oder ein Salz davon darstellt.

3. Eine Zusammensetzung gemäß einem der Ansprüche 1 bis 2, wobei die Partikel 5 bis 50% (Gew./Gew.) der Verbindung A und 40 bis 85% (Gew./Gew.) der Verbindung B enthalten, beides bezogen auf das Gewicht der Partikel, und die Gesamtmenge der Verbindung A und B in den Partikeln sich auf 45 bis 90% (Gew./Gew.) aufsummiert.

4. Eine Zusammensetzung gemäß einem der Ansprüche 1 bis 3, wobei das Alkalimetallsilicat der allgemeinen Formel (III) außerdem **dadurch gekennzeichnet ist, dass** n im Bereich von 1,6 bis 2,6, bevorzugt von 1,8 bis 2,2 liegt und das Alkalimetall bevorzugt ausgewählt ist aus der Gruppe, bestehend aus Lithium, Natrium, Kalium und Mischungen davon.

5. Eine Zusammensetzung gemäß Anspruch 1 bis 4, wobei die Zusammensetzung eine rieselfähige Zusammensetzung, bevorzugt ein rieselfähiges Pulver oder Granulat und am meisten bevorzugt ein rieselfähiges Granulat darstellt.

6. Eine Zusammensetzung gemäß einem der Ansprüche 1 bis 5, wobei die durchschnittliche Partikelgröße im Bereich von 0,4 bis 2,0 mm liegt.

7. Eine Zusammensetzung gemäß einem der Ansprüche 1 bis 6, wobei die Partikel wenigstens eine weitere Komponente enthalten, ausgewählt aus der Gruppe, bestehend aus Tensiden und organischen Polymeren, bevorzugt ausgewählt aus der Gruppe, bestehend aus nichtionischen Tensiden und organischen Polymeren, am meisten bevorzugt ausgewählt aus der Gruppe, bestehend aus Polyvinylalkohol, Polyvinylpyrrolidonen, Polyvinylacetat, Polyalkylenglykolen oder einer Mischung daraus oder CoPolymeren davon.

8. Ein Verfahren zum Herstellen einer partikelförmigen Zusammensetzung gemäß einem der Ansprüche 1 bis 7, umfassend die Schritte:

(i) Mischen der Bestandteile, umfassend wenigstens die Verbindung A und die Verbindung B oder die Verbindung B und ein Silicat,
(ii) Sprühtrocknen der Mischung und
(iii) (optional) das Granulieren der erhaltenen Mischung,

wobei bevorzugt wenigstens die Verbindungen A und B zumindest vorübergehend in einer flüssigen Form während des Mischungsprozesses vorliegen, weiter bevorzugt in Form einer wässrigen Lösung.

9. Verwendung einer partikelförmigen Zusammensetzung gemäß einem der Ansprüche 1 bis 7 oder einer partikelförmigen Zusammensetzung, hergestellt gemäß Anspruch 8, als ein Bestandteil in einem Reinigungsmittel und/oder Reinigungsformulierungen, für die Herstellung eines solchen Reinigungsmittels und/oder Reinigungsformulierungen und zum Reinigen und/oder Waschen von Gegenständen.

10. Eine Reinigungs- und/oder Waschmittelformulierung, enthaltend von 0,5 bis 80% (Gew./Gew.), bezogen auf das Gewicht der Formulierung, eine partikelförmige Zusammensetzung gemäß einem der Ansprüche 1 bis 7.

11. Eine Reinigungs- und/oder Waschmittelformulierung gemäß Anspruch 10, wobei die Zusammensetzung eine Geschirrspülmittelzusammensetzung, bevorzugt für Spülmaschinen ist.

12. Eine Reinigungs- und/oder Waschmittelformulierung gemäß Anspruch 11, außerdem enthaltend wenigstens einen weiteren Bestandteil, ausgewählt aus der Gruppe, bestehend aus weiteren Tensiden, Gerüststoffen, Chelatbildnern, Komplexierungsmittel, Bleichmittel, Bleichaktivatoren, Bleichkatalysatoren, optischen Aufhellern, Klarspüladditiven, Korrisionsinhibitoren, Anti-Wiederablagerungsmitteln, Enzymen, Dispergiermitteln, pH-Modifikatoren, Farbstoffen, Färbemitteln, Parfüm.

13. Verwendung eines Disilicats, bevorzugt eines Disilicats gemäß Anspruch 4, für die Herstellung einer partikelförmigen Zusammensetzung gemäß einem der Ansprüche 1 bis 7.

Revendications

1. Composition en particules, dans laquelle au moins une partie des particules présentes dans ladite composition comporte :

i) au moins 5 % (pds/pds) d'un composé hygroscopique et chélateur de type amino-polycarboxylate, nommé A,
ii)

a) et un autre composé chélateur de type amino-polycarboxylate, nommé B, qui possède une structure chimique différente de celle de l'agent chélateur A,
ces composés A et B étant choisis indépendamment dans l'ensemble comprenant les suivants : acide 2-hydroxy-éthyl)-éthylènediamine-triacétique (HEDTA), acide diéthylènetriamine-pentaacétique (DTPA), acide méthyl-glycine-N,N-diacétique (MGDA), acide glutamique-N,N-diacétique (GLDA), acide imino-disuccinique (IDS), acide hydroxy-imino-disuccinique (HIDS), acide éthylènediamine-disuccinique (EDDS), et acide aspartique-N,N-diacétique (ASDA), et leurs sels, ainsi que leurs mélanges, sous réserve que si le composé chélateur de type amino-polycarboxylate A est du GLDA et s'il n'y a pas de silicate dans la composition, le composé chélateur de type amino-polycarboxylate B n'est pas du MGDA,
b) ou un mélange dudit composé chélateur de type amino-polycarboxylate B et d'un silicate, lequel silicate est un silicate de métal alcalin de formule générale (III) :

$$M_2O : n\ SiO_2 \qquad (III)$$

dans laquelle M représente un métal alcalin et n vaut de 1 à 3,5 ; et dans laquelle

- la proportion combinée des composés chélateurs de type amino-polycarboxylate dans lesdites particules est égale ou supérieure à 40 %, en poids rapporté au poids des particules, et la proportion de silicate vaut de 0 à 40 %, en poids rapporté au poids desdites particules,
- le rapport de la proportion de silicate à la proportion combinée des composés chélateurs de type amino-

polycarboxylate, toutes deux rapportées au poids des particules, vaut moins de 0,75,
- et l'hygroscopicité du composé B est inférieure à l'hygroscopicité du composé A.

2. Composition conforme à la revendication 1, dans laquelle le composé A est du GLDA ou de l'un de ses sels, et le composé B est de préférence de l'IDS ou de l'un de ses sels.

3. Composition conforme à l'une des revendications 1 et 2, dans laquelle les particules comportent de 5 à 50 % (pds/pds) de composé A et de 40 à 85 % (pds/pds) de composé B, par rapport au poids des particules dans les deux cas, et le total des proportions des composés A et B dans les particules vaut de 45 à 90 % (pds/pds).

4. Composition conforme à l'une des revendications 1 à 3, dans laquelle le silicate de métal alcalin de formule générale (III) est plus précisément **caractérisé en ce que** le coefficient n vaut de 1,6 à 2,6 et de préférence de 1,8 à 2,2, et **en ce que** le métal alcalin est de préférence choisi dans l'ensemble constitué par les lithium, sodium et potassium et leurs mélanges.

5. Composition conforme à l'une des revendications 1 à 4, laquelle composition est une composition qui peut s'écouler librement, de préférence une poudre ou un granulat s'écoulant librement, et surtout un granulat s'écoulant librement.

6. Composition conforme à l'une des revendications 1 à 5, dans laquelle la taille moyenne des particules se situe dans l'intervalle allant de 0,4 à 2,0 mm.

7. Composition conforme à l'une des revendications 1 à 6, dans laquelle les particules comprennent au moins un composant supplémentaire choisi dans l'ensemble formé par les tensioactifs et les polymères organiques, de préférence choisi dans l'ensemble formé par les tensioactifs non-ioniques et les polymères organiques, et surtout choisi dans l'ensemble formé par les poly(alcool vinylique), poly(vinyl-pyrrolidone), poly(acétate de vinyle) et polyalkylèneglycols, leurs mélanges et leurs copolymères.

8. Procédé de préparation d'une composition en particules, conforme à l'une des revendications 1 à 7, lequel procédé comporte les étapes suivantes :

i) mélanger les ingrédients, qui comprennent au moins du composé A, ainsi que du composé B ou du composé B et un silicate,
ii) et faire sécher ce mélange par pulvérisation,
iii) et en option, granuler le mélange obtenu,

et dans lequel, de préférence, au moins les composés A et B se trouvent, au moins temporairement, sous forme liquide au cours de l'opération de mélange, et mieux encore, sous la forme de solution aqueuse.

9. Utilisation d'une composition en particules conforme à l'une des revendications 1 à 7, ou d'une composition en particules préparée conformément à la revendication 8, en tant qu'ingrédient dans des formulations de détergent et/ou de nettoyage, pour la fabrication desdites formulations de détergent et/ou de nettoyage et pour des articles de nettoyage et/ou de lavage.

10. Formulation de nettoyage et/ou de détergent, comprenant de 0,5 à 80 %, en poids rapporté au poids de la formulation, d'une composition en particules conforme à l'une des revendications 1 à 7.

11. Formulation de nettoyage et/ou de détergent, conforme à la revendication 10, laquelle composition est une formulation pour lavage de vaisselle, et de préférence pour lave-vaisselle automatique.

12. Formulation de nettoyage et/ou de détergent, conforme à la revendication 11, qui comprend en outre au moins un ingrédient supplémentaire choisi dans l'ensemble constitué par les tensioactifs, adjuvants actifs, chélateurs, agents complexants, agents de blanchiment, activateurs de blanchiment, catalyseurs de blanchiment, azurants optiques, adjuvants auxiliaires de rinçage, inhibiteurs de corrosion, agents anti-redéposition, enzymes, agents dispersants, agents de modification du pH, colorants, pigments et parfums.

13. Utilisation d'un disilicate, de préférence un disilicate tel que défini dans la revendication 4, pour la préparation d'une composition en particules conforme à l'une des revendications 1 à 7.

Figure 1

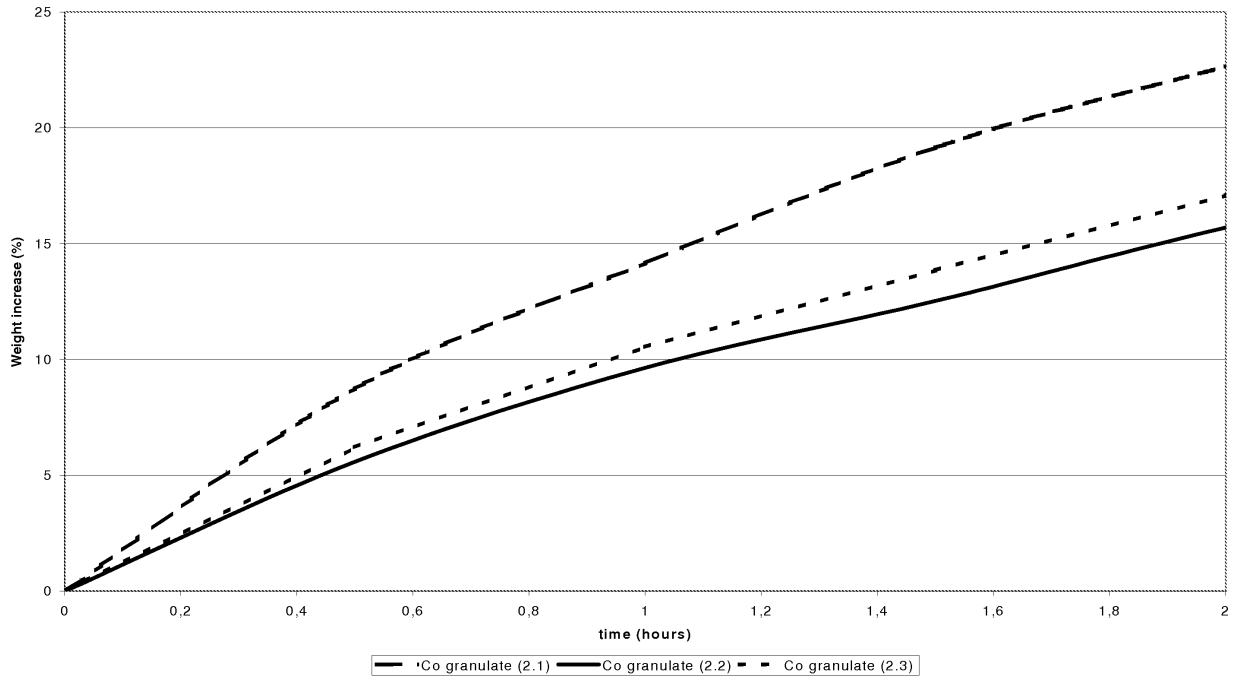
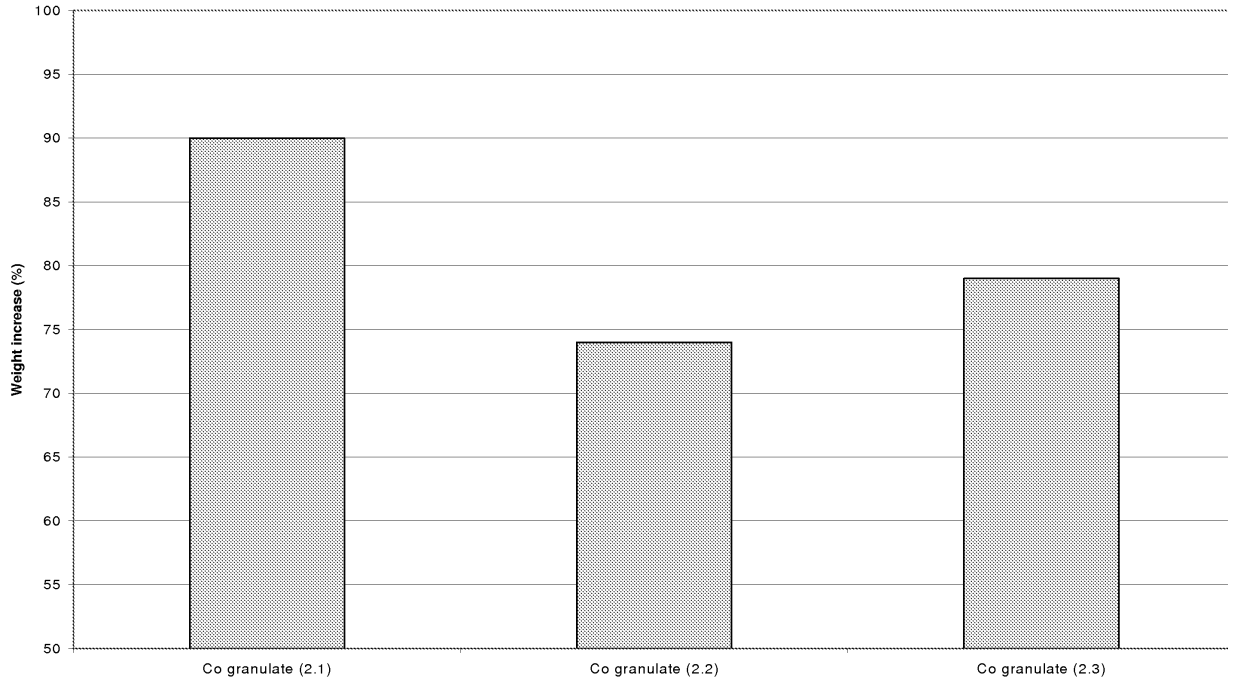

25
20
15
10
5
0
Weight increase (%)
0
0,2
0,4
0,6
0,8
1
1,2
1,4
1,6
1,8
2
time (hours)
Co granulate (2.1)
Co granulate (2.2)
Co granulate (2.3)

Figure 2


100
95
90
85
80
75
70
65
60
55
50
Weight increase (%)
Co granulate (2.1)
Co granulate (2.2)
Co granulate (2.3)

REFERENCES CITED IN THE DESCRIPTION

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

Patent documents cited in the description

- WO 2009103822 A **[0006]**
- WO 2011079940 A **[0024]**
- EP 1524313 A **[0049]**
- WO 2005095570 A **[0063]**
- DE 19934704 A1 **[0065]**
- EP 1363986 A **[0066]**
- EP 1268729 A **[0066]**
- EP 299513 A1 **[0066]**
- EP 0877002 A **[0066]**
- WO 2008101909 A **[0085]**