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(54) Title: DETERGENT COMPOSITION COMPRISING GLUTAMIC-N,N-DIACETATE, WATER AND BLEACHING AGENT

(57) Abstract: One aspect of the invention relates to a detergent composition comprising 20-99.7 wt.% of a continuous phase containing glutamic-N,N-diacetate (GLDA), water and optionally other components; and 0.3-80 wt.% of a dispersed phase comprising at least 0.3% of bleach particles by weight of the composition, said bleach particles having a particle size of at least 10 μπι, wherein the continuous phase contains at least 10 wt.% of GLDA and at least 10 wt.% of water and wherein the weight ratio of GLDA to water lies within the range of 5:6 to 5:1. Despite the high water content, the present bleach containing detergent composition is storage stable and retains bleach activity for a prolonged period of time. The invention also provides a process of preparing a liquid base for a detergent composition comprising GLDA and water in a weight ratio of GLDA to water of 5:6 to 5:1, said process comprising combining 100 parts by weight of a liquid mixture of GLDA and water with 10-300 parts by weight of GLDA powder, said liquid mixture containing 20-50 wt.% of GLDA and 50-80 wt.% water and said GLDA powder containing at least 65 wt.% GLDA.

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DETERGENT COMPOSITION COMPRISING GLUTAMIC-N.N-DIACETATE, WATER AND BLEACHING AGENT

TECHNICAL FIELD OF THE INVENTION

The present invention relates to a detergent composition comprising glutamic-N,N-diacetate (GLDA), water and a bleaching agent. More particularly, the present invention relates to a detergent composition comprising 20-99.7 wt.% of a continuous phase containing GLDA, water and optionally other components; and 0.3-80 wt.% of a dispersed phase comprising at least 0.3% of bleach particles by weight of the composition, said bleach particles having a particle size of at least 10 μm, wherein the continuous phase contains at least 10 wt.% of GLDA and at least 10 wt.% water, and wherein the weight ratio of GLDA to water lies within the range of 5:6 to 5:1.

The composition of the present invention offers the advantage that the bleaching agent remains stable despite the fact that the composition contains a substantial amount of water. This detergent composition may suitably be provided in the form of a liquid, a gel or a paste. Examples of detergent compositions according to the present invention include dishwashing compositions, laundry detergents and hard surface cleaning compositions.

BACKGROUND OF THE INVENTION

Detergent formulations typically contain a number of different active components, including builders, surfactants, enzymes and bleaching agents. Liquid and gelled detergent formulations have been developed as these products offer the advantage that they are easy to dose and/or that they can easily be dispersed into aqueous cleaning liquid. However, a major challenge in the development of liquid and gelled detergent formulations lies in the fact that bleaching agents, such as percarbonates and peracids, tend to be unstable in aqueous formulations, especially under alkaline conditions.

Bleaching agents are employed in detergent compositions to remove bleachable stains, e.g. tea stains. A problem associated with the use of bleaching agents in liquid, water-containing detergent formulations lies in the fact that most bleaching agents are not stable in an aqueous (alkaline) environment. Not only does this instability mean that bleaching activity is lost over time, but in addition this bleach instability also adversely affects the functionality of other detergent components, notably enzymes.

Calcium and magnesium ions have a negative effect on the removal of soils by detergent compositions. In order to negate these negative effects so called 'builders' (complexing agents) are commonly applied in detergent compositions.

Phosphorous based builders, such as phosphates, have been used for many years in a wide variety of detergent compositions. However, as part of an increasing trend towards environmentally friendly detergent compositions, alternative building agents have been developed and these alternative builders have found their way into commercial detergent products. Glutamic-N,N-diacetate (GLDA), methyl-glycine diacetate (MGDA) and citrate are examples of environmentally friendly builders that are used in commercial detergent products.

WO 2007/141527 describes a detergent composition comprising an amino acid based builder, enzymes that are destabilized by the builder and a stabilization system for the enzymes that comprises one or more divalent metal compounds or salts and one or more non-ionic surfactants. The example of this patent application describes detergent pouches made of PVA that contain a bleach-free liquid dishwashing formulation comprising 58.7 wt.% water and 31.0 wt.% GLDA.

WO 2009/123322 describes an automatic dishwasher composition comprising a builder and a malic acid based water-soluble sulphonated copolymer. Example 3 of this international patent application describes a bleach-free formulation comprising 53.4 wt.% water and 30.0 wt.% GLDA.

SUMMARY OF THE INVENTION

The present inventors have unexpectedly discovered that storage stable, bleach- and water-containing, liquid detergent compositions can be produced if these detergent compositions additionally contain a substantial amount of GLDA and if the balance between water and GLDA is properly tuned.

One aspect of the invention relates to a detergent composition comprising 20-99.7 wt.% of a continuous phase containing GLDA, water and optionally other components; and 0.3-80 wt.% of a dispersed phase comprising at least 0.3% of bleach particles by weight of the composition, said bleach particles having a particle size of at least 10 μm, wherein the continuous phase contains at least 10 wt.% of GLDA and at least 10 wt.% of water and wherein the weight ratio of GLDA to water lies within the range of 5:6 to 5:1.

Another aspect of the invention relates to a composition comprising 20-99.7 wt.% of a continuous phase containing GLDA, water and optionally other components; and 0.3-80 wt.% of a dispersed phase comprising at least 0.3 wt.% of bleach particles by weight of the composition, said bleach particles having a particle size of at least 10 μm, wherein the composition contains at least 10 wt.% water and has a water activity of not more than 0,7.

Although the inventors do not wish to be bound by theory, it is believed that the GLDA in the present composition binds the water contained therein so strongly that the water can no longer exert its destabilising effect on the bleaching agent.

The present invention also provides a process of preparing a liquid base for a detergent composition comprising GLDA and water in a weight ratio of GLDA to water of 5:6 to 5:1, said process comprising combining 100 parts by weight of a liquid mixture of GLDA and water with 10-300 parts by weight of GLDA powder, said liquid mixture containing 20-50 wt.% of GLDA and 50-80 wt.% water and said GLDA powder containing at least 65 wt.% GLDA.

DEFINITIONS

The term "glutamic-N,N-diacetate" or "GLDA" as used herein refers to glutamic-N,N-diacetic acid and salts thereof.

The term "bleach particles" as used herein refers to particles comprising a bleaching agent that is capable of releasing peroxygen, chlorine- or bromine under conditions typically encountered during the cleansing process, especially if the present composition is diluted 10-1 000 times with water having a temperature of 5-80°C.

The term "particles" as used herein, unless indicated otherwise, refers to a solid material in the form of, for instance, particles, grains, granules or tablets.

The term "water soluble" as used herein means a solubility in distilled water of 20°C of at least 0.01 mol/L, preferably of at least 0.1 mol/L.

The term "dispersed phase" as used herein refers to any particulate matter that is contained in the present composition. This particulate matter may be solid, semi-solid or liquid. Particulate matter having a particle size of less than 100 nm is deemed to be part of the continuous phase.

Whenever reference is made herein to a water content, unless indicated otherwise, said water content includes unbound (free) as well as bound water.

Whenever a parameter, such as a concentration or a ratio, is said to be less than a certain upper limit it should be understood that in the absence of a specified lower limit the lower limit for said parameter is 0.

Whenever an amount or concentration of a component is quantified herein, unless indicated otherwise, the quantified amount or quantified concentration relates to said component pe*r* se, even though it may be common practice to add such a component in the form of a solution or of a blend with one or more other ingredients..

The term "comprising" is used herein in its ordinary meaning and means including, made up of, composed of, consisting and/or consisting essentially of. In other words, the term is defined as not being exhaustive of the steps, components, ingredients, or features to which it refers.

DETAILED DESCRIPTION OF THE INVENTION

Accordingly, one aspect of the present invention relates to a composition comprising 20-99.7 wt.% of a continuous phase containing GLDA, water and optionally other components; and 0.3-80 wt.% of a dispersed phase comprising at least 0.3% of bleach particles by weight of the composition, said bleach particles having a particle size of at least 10 μιτι, wherein the continuous phase contains at least 10 wt.% of GLDA and at least 10 wt.% water and wherein the weight ratio of GLDA to water lies within the range of 5:6 to 5:1.

The water activity of the composition typically does not exceed 0.7. More preferably, the water activity of the composition lies within the range of 0.1-0.6, even more preferably 0.2-0.55, most preferably of 0.3-0.5 The water activity of the composition may suitably be determined by a Novasina labmaster conditioned Aw measuring device that is set at 25 °C and measured until stable.

Another aspect of the invention relates to a composition comprising 20-99.7 wt.% of a continuous phase containing GLDA, water and optionally other components; and 0.3-80 wt.% of a dispersed phase comprising at least 0.3 wt.% of bleach particles by weight of the composition, said bleach particles having a particle size of at least 10 μιτι, wherein the composition contains at least 10 wt.% water and has a water activity of not more than 0.7. Preferably, the water activity of the composition lies within the range of 0.1-0.6, even more preferably of 0.2-0.55 and most preferably of 0.3-0.5

The composition according to the latter embodiment preferably contains GLDA and water in weight ratio of GLDA to water that lies within the range of 5:6 to 5:1.

The continuous phase containing GLDA, water and optional other components and the dispersed phase comprising the bleach particles are present as distinct (separate) phases in the detergent composition of the present invention. Besides the bleach particles the dispersed phase may contain additional solid or liquid component that are not soluble in the continuous phase.

This detergent composition may suitably be provided in the form of a liquid, a gel or a paste. According to a particularly preferred embodiment, the composition is a liquid or a gel.

In accordance with a preferred embodiment, the present composition contains 5-80 wt.%, more preferably 10-60 wt.% and most preferably 15-50 wt.% of GLDA. Expressed differently, the present composition preferably contains 30-70% of GLDA by weight of the continuous phase. Even more preferably, the composition contains 35-65% and most preferably 40-60% of GLDA by weight of the continuous phase.

The water content of the present composition preferably lies in the range of 10-50 wt.%, more preferably of 15-45 wt.% and most preferably of 20-35 wt.%.

The water content of the present composition expressed by weight of the continuous phase preferably lies in the range of 6-55 wt.%, more preferably 15-50 wt.% and most preferably 25-48 wt.%.

Together, GLDA and water typically represent at least 60 wt.%, more preferably at least 70 wt.% and most preferably at least 80 wt.% of the continuous phase of the present composition.

As explained herein before, the stability of the bleaching agent in the present composition is dependent on the water/GLDA balance of the product. Advantageously, the present composition contains GLDA and water in a weight ratio of GLDA to water that lies within the range of 9:10 to 3:1; more preferably of 1:1 to 5:2; and most preferably of 5:4 to 5:3.

The continuous phase of the present detergent composition preferably is a liquid, a paste or a gel. Even more preferably, the continuous phase of the detergent composition is a liquid or a gel.

The one or more other component that are optionally present in the continuous phase include, for instance polyols (e.g. glycerol), acids (e.g. citric acid), viscosifiers, surfactants, perfume, preservatives and colorant. Preferably the one or more other components represent not more than 40 wt.% , more preferably not more than 30 wt.%, even more preferably not more than 20 wt.% and most preferably not more than 10 wt.% of the continuous phase.

Examples of detergent compositions encompassed by the present invention include dishwashing compositions, laundry detergents, rim blocks and hard surface cleaning compositions. According to a particularly preferred embodiment, the detergent composition is a dishwashing composition, especially a machine dishwashing composition.

Preferably the composition of the invention is a machine dishwashing composition which, when added to demineralised water having a temperature of 20°C in a concentration of 1 g per 100 ml of water, yields an aqueous solution having a pH of 7-13, preferably of 8-12, most preferably of 9-11.4.

Machine dishwash detergent compositions according to the invention may suitably be dosed at levels of 5 to 40 grams per wash, more preferably at 10 to 30 grams per wash.

The inventors have unexpectedly discovered that the stability of the bleaching agent in the present composition is further improved if the composition contains 0.1-10%, preferably 0.5-4% by weight of the continuous phase of dissolved acid. According to a preferred embodiment, the acid is selected from sulphuric acid, citric acid and combinations thereof. The incorporation of dissolved acid in the specified amounts enables the preparation of stable bleach-containing detergent compositions having a water activity in the range of, for instance, 0.4-0.7, especially of 0.45-0.65.

Bleach

Typically, the bleach particles in the detergent composition contain at least 20 wt.%, more preferably at least 40 wt.% and most preferably at least 70 wt.% of bleaching agent.

The bleach particles contained in the present composition may be contained therein in suspended and/or sedimented form. Preferably, the particles are suspended in the continuous phase.

The bleach particles contained in the present composition preferably have a volume weighted mean particle size of 10-3000 μιτι, more preferably of 100-1500 μιτι and most preferably of 200-1000 μιτι. The particle size of particles may suitably be determined by a Malvern particle size analyzer based on laser differentiation.

The amount of bleach particles contained in the present composition typically lies in the range of 0.5-25 wt.%. More preferably the bleach particles content is in the range of 1-20 wt.%, most preferably of 3-18 wt.%.

The bleach particles of the present composition may suitably comprise a chlorine-, or bromine-releasing agent or a peroxygen compound. The bleach particles of the present composition may comprise a bleaching agent selected from peroxides, organic peracids, salts of organic peracids and combinations thereof.

Examples of peroxides are acids and corresponding salts of monopersulphate, perborate monohydrate, perborate tetrahydrate, and percarbonate.

Organic peracids useful herein include alkyl peroxy acids and aryl peroxyacids such as peroxybenzoic acid and ring-substituted peroxybenzoic acids (e.g. peroxy-alpha-naphthoic acid), aliphatic and substituted aliphatic monoperoxy acids (e.g. peroxylauric acid and peroxystearic acid), and phthaloyl amido peroxy caproic acid (PAP).

Typical diperoxy acids useful herein include alkyl diperoxy acids and aryldiperoxy acids, such as 1,12-di-peroxy-dodecanedioic acid (DPDA), 1,9-diperoxyazelaic acid, diperoxybrassylic acid, diperoxysebacic acid and diperoxy-isophthalic acid, and 2-decyldiperoxybutane-1 ,4-dioic acid.

Preferably, the bleach particles of the present composition comprise a bleaching agent selected from peroxides (including peroxide salts such as sodium percarbonate), organic peracids, salts of organic peracids and combinations thereof. More preferably, the bleaching agent is a peroxide. Most preferably, the bleaching agent is a percarbonate.

According to a preferred embodiment, the present composition comprises coated bleach particles. In a further preferred embodiment, substantially all bleach particles are coated bleach particles. According to a particularly preferred embodiment, the coated bleach particles comprise a water-soluble coating. The water-soluble coating advantageously comprises a coating agent selected from alkali sulphate, alkali carbonate or alkali chloride and combinations thereof.

The coating of the bleaching agent can be done by, for example, crystallisation or by spray granulation. Suitable coated bleaching agents are described in, for example, EP-A 0 891 417, EP-A 0 136 580 and EP-A 0 863 842. The use of spray granulated coated percarbonate is most preferred.

The present detergent composition offers the advantage that despite the high water content, the bleach particles remain very stable. Typically, bleach activity of a freshly prepared composition according to the present invention decreases by not more than 70%, more preferably by not more than 50% and most preferably by not more than 20% when the composition is stored in a closed container for 8 weeks at 20 °C. Bleach activity may suitably be determined by iodometric titration. In this titration a suitable amount of detergent sample is dissolved in acidified water containing a molybdate catalyst. Subsequently potassium iodide is added to an aliquot of the solution. The iodine liberated by the bleach is titrated with sodium thiosulphate solution

The detergent composition may contain one or more bleach activators such as peroxyacid bleach precursors. Peroxyacid bleach precursors are well known in the art. As non-limiting examples can be named N,N,N',N'-tetraacetyl ethylene diamine (TAED), sodium nonanoyloxybenzene sulphonate (SNOBS), sodium benzoyloxybenzene sulphonate (SBOBS) and the cationic peroxyacid precursor (SPCC) as described in US-A-4,751 ,01 5.

If desirable, a bleach catalyst, such as the manganese complex, e.g. Mn-Me TACN, as described in EP-A-0458397, or the sulphonimines of US-A-5,041 ,232 and US-A-5,047, 163, can be incorporated. This bleach catalyst may suitably be present in the composition in the form of a encapsulate, notably an encapsulate that is separate from the bleach particles (to avoid premature bleach activation). Cobalt or iron catalysts can also be used.

The high stability of the bleaching agent in the present composition makes it possible to include enzymes in the formulation as the enzymes are not immediately deactivated by the bleaching agent. Examples of enzymes suitable for use in the cleaning compositions of this invention include lipases, cellulases, peroxidases, proteases (proteolytic enzymes), amylases (amylolytic enzymes) and others which degrade, alter or facilitate the degradation or alteration of biochemical soils and stains encountered in cleansing situations so as to remove more easily the soil or stain from the object being washed to make the soil or stain more removable in a subsequent cleansing step. Both degradation and alteration can improve soil removal.

Well-known and preferred examples of these enzymes are proteases, amylases, cellulases, peroxidases, mannanases, pectate lyases and lipases and combinations thereof. The enzymes most commonly used in detergent compositions are proteolytic and amylolytic enzymes.
Enzymes may be added in liquid or in encapsulated form. In a preferred embodiment of this invention the enzymes are present in encapsulated form. Well know enzyme stabilizers such as polyalcohols/borax, calcium, formate or protease inhibitors like 4-formyl phenyl boronic acid may also be present in the composition The proteolytic enzymes in this invention include metalloproteases and serine proteases, including neutral or alkaline microbial serine protease, such as subtilisins (EC 3.4.2 1.62). The proteolytic enzymes for use in the present invention can be those derived from bacteria of fungi. Chemically or genetically modified mutants (variants) are included. Preferred proteolytic enzymes are those derived from *Bacillus,* such as *B. lentus, B. gibsonii, B. subtilis, B. licheniformis, B. alkalophilus, B. amyloliquefaciens* and *Bacillus pumilus,* of which *B. lentus* and *B. gibsonii* are most preferred. Examples of such proteolytic enzymes are Excellase™ , Properase™ , Purafect™ , Purafect™ Prime, Purafect™ Ox by Genencor; and those sold under the trade names Blaze ™,Ovozyme™ , Savinase™ , Alcalase™ , Everlase™ , Esperase™ , Relase™ , Polarzyme™ , Liquinase™ and Coronase™ by Novozymes.

Preferred levels of protease in the present composition are from 0.1 to 10, more preferably from 0.2 to 5, most preferably 0.4 to about 4 mg active protease per gram of composition.

The amylolytic enzymes for use in the present invention can be those derived from bacteria or fungi. Chemically or genetically modified mutants (variants) are included. Preferred amylolytic enzyme is an alpha-amylase derived from a strain of *Bacillus,* such as *B. subtilis, B. licheniformis, B.. amyloliquefaciens* or *B. stearothermophilus.* Examples of such amylolytic enzymes are produced and distributed under the trade name of Stainzyme™ , Stainzyme™ Plus, Termamyl™ , Natalase™ and Duramyl™ by Novozymes; as well as Powerase™ , Purastar™ , Purastar™ Oxam by Genencor. Stainzyme™ , Stainzyme™ Plus and Powerase™ are the preferred amylases.

Preferred levels of amylase in the present composition are from 0.01 to 5, more preferably from 0.02 to 2, most preferably from 0.05 to about 1 mg active amylase per gram of composition.

The enzymes may suitably be incorporated in the detergent composition in liquid or in encapsulated form. In case the composition has a pH of 9.0 and more it is preferred to employ enzymes in encapsulated form.

Examples of encapsulated forms are enzyme granule types D, E and HS by Genencor and granule types , T, GT, TXT and Evity™ of Novozymes.

In case the pH is less than 9.0 it can be advantageous to employ non-encapsulated enzymes.

In accordance with a particularly preferred embodiment of the invention, the composition contains active protease and the protease activity of the freshly prepared composition decreases by not more than 70%, more preferably by not more than 50% and most preferably by not more than 20% when the composition is stored in a closed container for 8 weeks at 20 °C.

Surfactants

The present detergent composition preferably contains one or more surfactants. Surfactants, within the invention, are components within the classification as described in "Surfactant Science Series", Vol.82, Handbook of detergents, part A: *Properties, chapter 2 (Surfactants, classification),* G. Broze (ed.).

According to a particularly preferred embodiment, the composition contains 0.1-15 wt.%, more preferably 0.5-10 wt.% and most preferably 1-5 wt.% of a nonionic surfactant or a mixture of two or more non-ionic surfactants.

Examples of nonionic surfactants that may be employed in the present composition include the condensation products of hydrophobic alkyl, alkenyl, or alkyl aromatic compounds bearing functional groups having free reactive hydrogen available for condensation with hydrophilic alkylene oxide, such as ethylene oxide, propylene oxide, butylene oxide, polyethylene oxide or polyethylene glycol to form nonionic surfactants. Examples of such functional groups include hydroxy, carboxy, mercapto, amino or amido groups.

Examples of useful hydrophobes of commercial nonionic surfactants include C8-C18 alkyl fatty alcohols, **C_8-Ci4** alkyl phenols, C_8-Ci$_8$ alkyl fatty acids, C_8-Ci$_8$ alkyl mercaptans, C_8-Ci$_8$ alkyl fatty amines, C_8-Ci$_8$ alkyl amides and C_8-Ci$_8$ alkyl fatty alkanolamides. Accordingly, suitable ethoxylated fatty alcohols may be chosen from ethoxylated cetyl alcohol, ethoxylated ketostearyl alcohol, ethoxylated isotridecyl alcohol, ethoxylated lauric alcohol, ethoxylated oleyl alcohol and mixtures thereof.

Examples of suitable nonionic surfactants for use in the invention are found in the low- to non-foaming ethoxylated/ propoxylated straight-chain alcohols of the Plurafac™ LF series, supplied by the BASF and the Synperonic™ NCA series supplied by Croda. Also of interest are the end-capped ethoxylated alcohols available as the SLF 18 series from BASF and the alkylpolyethylene glycol ethers made from a linear, saturated C_{16}-C_{18} fatty alcohol of the Lutensol™ AT series, supplied by BASF. Other suitable nonionics to apply in the composition of the invention are modified fatty alcohol polyglycolethers available as Dehypon™ 3697 GRA or Dehypon™ Wet from BASF/Cognis. Also suitable for use herein are nonionics from the Lutensol™ TO series of BASF, which are alkylpolyethylene glycol ethers made from a saturated iso-C_{13} alcohol.

Amineoxide surfactants may also be used in the present invention as anti-redeposition surfactant. Examples of suitable amineoxide surfactants are C_{10}-Ci$_8$ alkyl dimethylamine oxide and C_{10}-Ci$_8$ acylamido alkyl dimethylamine oxide.

The inventors have found that, a detergent composition that is not only chemically but also physically very stable can be produced if the nonionic surfactant employed is solid at ambient temperature. Thus, advantageously, the present composition contains 0.1-15 wt.%, more preferably 0.5-10 wt.% and most preferably 1-5 wt.% of nonionic surfactant that is solid at 25°C.

If an anionic surfactant is used, the total amount present preferably is less than 5 wt.%, and more preferably not more than 2 wt.%. Furthermore, if an anionic surfactant is present, it is preferred that an antifoam agent to suppress foaming is present. Examples of suitable anionic surfactants are methylester sulphonates or sodium lauryl sulphate.

Builders

The GLDA contained in the present composition preferably is an alkali metal salt of glutamic-N,N-diacetic acid. More preferably, the GLDA employed is a sodium salt of glutamic-N,N-diacetic acid. Most preferably, the GLDA employed is a tetra sodium salt of glutamic-N,N-diacetic acid.

Compositions according to the present invention may, next to GLDA, also contain additional water-softening builders. Traditionally phosphorous based builders, such as phosphates have been used as builders, but due to environmental pressures other builders are preferred. These include organic builders such as citrate, MGDA (methyl-glycine diacetate) and inorganic builders such as carbonates, in particular sodium carbonate.

Silicates

Silicates may be added to the formulation. Silicates can act as builder, buffering agent or article care agent. Preferred silicates are sodium silicate such as sodium disillicate, sodium metasilicate and crystalline phyllosilicates and mixtures thereof. Silicates are preferably used in the detergent composition in a concentration of 1 to 20%, more preferably of 2 to 10% by weight of the composition.

Dispersing polymers

In a preferred embodiment of the current invention, the detergent composition furthermore comprises at least one dispersing polymer. Dispersing polymers as referred to in this invention are chosen from the group of anti-spotting agents and/or anti-scaling agents.

Examples of suitable anti-spotting polymeric agents include hydrophobically modified polycarboxylic acids such as Acusol ™ 460 ND (ex Dow) and Alcosperse™ 747 by AkzoNobel, whereas also synthetic clays, and preferably those synthetic clays which have a high surface area are very useful to prevent spots, in particular those formed where soil and dispersed remnants are present at places where the water collects on the glass and spots formed when the water subsequently evaporates.

Examples of suitable anti-scaling agents include organic phosphonates, amino carboxylates, polyfunctionally-substituted compounds, and mixtures thereof. Particularly preferred anti-scaling agents are organic phosphonates such as a-hydroxy-2 phenyl ethyl diphosphonate, ethylene diphosphonate, hydroxy 1,1-hexylidene, vinylidene 1,1-diphosphonate, 1,2-dihydroxyethane 1,1-diphosphonate and hydroxy-ethylene 1,1-diphosphonate. Most preferred is hydroxy-ethylene 1,1-diphosphonate (EDHP) and 2-phosphono-butane, 1,2,4-tricarboxylic acid (Bayhibit ex Bayer). Suitable anti-scaling agents are water soluble dispersing polymers prepared from an allyloxybenzenesulfonic acid monomer, a methallyl sulfonic acid monomer, a copolymerizable nonionic monomer and a copolymerizable olefinically unsaturated carboxylic acid monomer as described in US 5 547 612 or known as acrylic sulphonated polymers as described in EP 851 022. Polymers of this type include polyacrylate with methyl methacrylate, sodium methallyl sulphonate and sulphophenol methallyl ether such as Alcosperse™ 240 supplied (AkzoNobel). Also suitable is a terpolymer containing polyacrylate with 2-acrylamido-2 methylpropane sulphonic acid such as Acumer 3100 supplied by Dow. As an alternative, polymers and co-polymers of acrylic acid having a molecular weight between 500 and 20,000 can also be used, such as homo-polymeric polycarboxylic acid compounds with acrylic acid as the monomeric unit. The average weight of such homo-polymers in the acid form preferably ranges from 1,000 to 100,000 particularly from 3,000 to 10,000 e.g. Sokolan ™ PA 25 from BASF or Acusol™ 425 from Dow.

Also suitable are polycarboxylates co-polymers derived from monomers of acrylic acid and maleic acid, such as CP 5 from BASF. The average molecular weight of these polymers in the acid form preferably ranges from 4,000 to 70,000. Modified polycarboxylates like Sokalan™CP42, Sokalan™ CP50 from BASF or Alcoguard ™ 4160 from AkzoNobel may also be used.

Mixture of anti-scaling agents may also be used. Particularly useful is a mixture of organic phosphonates and polymers of acrylic acid.

It is preferable if the level of dispersing polymers ranges from 0.2 to 10 wt.% of the total composition, preferably from 0.5 to 8 wt.%, and further preferred from 1 to 6 wt.%.

Other ingredients

Glass corrosion inhibitors can prevent the irreversible corrosion and iridescence of glass surfaces in machine dishwash detergents. The claimed composition may suitably contain glass corrosion inhibitors. Suitable glass corrosion agents can be selected from the group the group consisting of salts of zinc, bismuth, aluminum, tin, magnesium, calcium, strontium, titanium, zirconium, manganese, lanthanum, mixtures thereof and precursors thereof. Most preferred are salts of bismuth, magnesium or zinc or combinations thereof. Preferred levels of glass corrosion inhibitors in the present composition are 0.01-2 wt.%, more preferably 10.01 - 0.5 wt.%.

Anti-tarnishing agents may prevent or reduce the tarnishing, corrosion or oxidation of metals such as silver, copper, aluminium and stainless steel. Anti-tarnishing agents such as benzotriazole or bis-benzotriazole and substituted or substituted derivatives thereof and those described in EP 723 577 (Unilever) may also be included in the composition. Other anti-tarnishing agents that may be included in the detergent composition are mentioned in WO 94/26860 and WO 94/26859. Suitable redox active agents are for example complexes chosen from the group of cerium, cobalt, hafnium, gallium, manganese, titanium, vanadium, zinc or zirconium, in which the metal are in the oxidation state of I, II, IV V or VI.

The present composition may suitably contain a non-surfactant, water-soluble, liquid binder, e.g. in a concentration of 0-50% by weight of the continuous phase. Examples of such liquid binders include polyethylene glycols, polypropylene glycols, glycerol, glycerol carbonate, ethylene glycol, propylene gylcol and propylene carbonate.

The continuous phase of the detergent composition preferably contains a viscosifier. By increasing the viscosity of the continuous phase through the addition of a viscosifier a stable suspension of the particles in the continuous phase can be produced. Preferably, the continuous phase contains 0.5-10 wt.% of a viscosifier.

Optionally other components may be added to the formulation such as perfume, colorant or preservatives.

The desired viscosity profile of the detergent composition depends on the end use of the product. It may be a liquid, gel or a paste depending on the application.

Another aspect of the present invention relates to a water-soluble sachet that is filled with a composition as defined herein before.

The inventors have found that the detergent composition of the present invention, despite the fact that it contains a substantial amount of water, can be packaged in a water-soluble sachet.

According to a preferred embodiment, the sachet comprises 5-40 ml, more preferably 10-30 ml and most preferably 15-25 ml of the detergent composition.

The water-soluble sachet is advantageously made of a polymer selected from polyvinyl alcohol, cellulose ethers, polyethylene oxide, starch, polyvinylpyrrolidone, polyacrylamide, polyvinyl methyl ether-maleic anhydride, polymaleic anhydride, styrene maleic anhydride, hydroxyethylcellulose, methylcellulose, polyethylene glycols, carboxymethylcelulose, polyacrylic acid salts, alginates, acrylamide copolymers, guar gum, casein, ethylene-maleic anhydride resin series, polyethylene imine, ethyl hydroxyethylcellulose, ethyl methylcellulose, hydroxyethyl methylcellulose and combinations thereof. Preferably, the water-soluble sachet is made of polyvinyl alcohol, polyethelene oxide, polyvinylpyrrolidone and combinations thereof.

Most preferably, the water-soluble sachet is made of polyvinyl alcohol, a copolymer of polyvinyl alcohol and combinations thereof. Polyvinyl alcohols preferred have a weight average molecular weight between 1,000 and 300,000, more preferably, between 2,000 and 150,000, and most preferably, between 3,000 and 100,000.

Yet another aspect of the invention relates to a process of preparing a liquid base for a detergent composition comprising GLDA and water in a weight ratio of GLDA to water of 5:6 to 5:1, said process comprising combining 100 parts by weight of a liquid mixture of GLDA and water withl 0-300 parts by weight, preferably 20-1 00 parts by weight of GLDA powder, said liquid mixture containing 20-50 wt.% of GLDA and 50-80 wt.% water and said GLDA powder containing at least 65 wt.% GLDA. The inventors have found that the detergent composition described herein before can suitably be produced by first producing a liquid base by combining a GLDA/water mixture and GLDA powder, followed by the addition of other detergent components.

Preferably, the liquid base that is obtained in the present method comprises GLDA and water in a weight ratio of GLDA to water that lies within the range of 9:10 to 3:1; more preferably of 1:1 to 5:2; and most preferably of 5:4 to 5:3

The liquid base that is obtained in the present process preferably contains 50-70 wt.%, more preferably 53-68 wt. and most preferably 55-65 wt.% GLDA. The water content of the liquid mixture preferably is in the range of 30-50 wt.%, more preferably 32-47 wt.% and most preferably 35-45wt.%.

The liquid mixture of GLDA and water that is employed in the present process typically contains 30-50 wt.% of GLDA and 50-70 wt.% water. The GLDA powder preferably contains at least 70 wt.% of GLDA.

Together, GLDA and water typically represent at least 90 wt.%, more preferably at least 95 wt.% and most preferably at least 98 wt.% of the liquid mixture. Likewise, GLDA and water together also represent preferably at least 90 wt.%, more preferably at least 95 wt.% and most preferably at least 98 wt.% of the GLDA powder.

Preferably the liquid mixture of GLDA and water has a temperature of at least 5°C, more preferably of 10-80°C and most preferably of 15-70°C when it is combined with the GLDA powder in order to ensure that a fully liquid base can be obtained without difficulty.

Advantageously, an acid component as defined herein before is present in the liquid base, most preferably said acid component is added in protonated, i.e. acid form. Typically, the acid component is added in an amount of 0.1-10%, more preferably of 1-7% and most preferably 3-5% by weight of the total amount of GLDA in the liquid base. The acid component may suitably be added to the continuous phase before addition of the bleach particles.

According to a particularly preferred embodiment the aforementioned process for preparing a liquid base is followed by further manufacturing steps to create a detergent composition from that liquid base, preferably a storage stable bleach-containing detergent composition as described herein before. In accordance with a particularly preferred embodiment, these further manufacturing steps include the addition to the liquid base of the bleach particles as defined herein before. Typically, these bleach particles are added in an amount of at least 0.3%, more preferably of 0.3-50%, even more preferably of 0.5-30% by weight of the liquid base.

Advantageously, before addition of bleach particles, a viscosifier as defined herein before is incorporated in the liquid base, typically in an amount of 0.5-10% by weight of the liquid base..

The invention is further illustrated by the following non-limiting examples.

EXAMPLES

Example 1

A detergent composition according to the present invention was prepared on the basis of the formulation presented in table 1.

Table 1

Component	Weight%
GLDA (Dissolvine™ GL PD-S)[1]	22.28
GLDA (Dissolvine™ GL 47-S)[2]	46.83
Citric acid (50%)	2.97
Poly acrylic acid polymer, anti-scalant	5.88
Bismuth citrate	0.35
Protease granule	0.88
Amylase granule	0.88
Nonionic [3]	3.6
Coated, spray-dried percarbonate	15.67
Mn catalyst granule	0.66

[1] Dissolvine™ GL PD-S, solid powder of GLDA tetrasodium salt ex Akzo Nobel.

[2] Dissolvine™ GL 47-S, a 47 wt% solution of GLDA tetrasodium salt ex Akzo Nobel.

[3] polyoxyethylene alkyl ether, melting point > 40°C

A liquid premix base was prepared by adding Dissolvine™ PD-S to Dissolvine™ GL 47-S at ambient temperature. Subsequently citric acid was admixed to the Dissolvine™ solution under stirring. The water content of the liquid premix so obtained was approximately 40 wt.%. Next, the remaining ingredients were added under stirring to produce a homogeneous suspension. The water content of the total composition was approximately 31%. The measured A_w (water activity) of the composition was 0.48.

The detergent composition was stored in a closed container for 2 weeks at 37°C. After this storage period the residual bleach activity was measured to be 80%.

20 g of the detergent composition was used in a machine dishwasher. The composition showed excellent cleaning performance.

Example 2

The detergent composition of Example 1 was filled into transparent pouches made of polyvinyl alcohol. Each pouch contained about 20 ml of the detergent composition. The appearance of these pouches did not change when the pouches were stored for several weeks under ambient conditions.

Example 3

A detergent composition according to the present invention was prepared on the basis of the formulation presented in table 2.

Table 2

Component	Weight%
GLDA (Dissolvine™ GL PD-S)[1]	22.96
GLDA (Dissolvine™ GL 47-S)[2]	48.27
Poly acrylic acid polymer, anti-scalant	6.06
Bismuth citrate	0.36
Protease granule	0.91
Amylase granule	0.91
Nonionic [3]	3.71
Coated, spray-dried percarbonate	16.15
Mn catalyst granule	0.68

[1] Dissolvine™ GL PD-S, solid powder of GLDA tetrasodium salt ex Akzo Nobel.

[2] Dissolvine™ GL 47-S, a 47 wt% solution of GLDA tetrasodium salt ex Akzo Nobel.

[3] polyoxyethylene alkyl ether, melting point > 40°C

A liquid premix base was prepared by adding Dissolvine™ PD-S to Dissolvine™ GL 47-S at ambient temperature under stirring. The water content of the liquid premix so obtained was approximately 42% wt.%. Next, the remaining ingredients were added under stirring to produce a homogeneous suspension. The water content of the total composition was approximately 29%.The measured A_w (water activity) of the composition was 0.46.

The detergent composition was stored in a closed container for 2 weeks at 37°C. After this storage period the residual bleach activity was measured to be 76%.

20 g of the detergent composition was used in a machine dishwasher. The composition showed excellent cleaning performance.

Example 4

A detergent composition according to the present invention was prepared on the basis of the formulation presented in table 3.

Table 3

Component	Weight%
GLDA (Dissolvine™ GL PD-S)[1]	30.53
GLDA (Dissolvine™ GL 47-S)[2]	35.15
Coated, spray-dried percarbonate	11.01
Citric acid (50%)	3.85
Foaming agent (35%)	5.51
Active surfactant[3]	13.94

[1] Dissolvine™ GL PD-S, solid powder of GLDA tetrasodium salt ex Akzo Nobel.

[2] Dissolvine™ GL 47-S, a 47 wt% solution of GLDA tetrasodium salt ex Akzo Nobel.

[3] Sodium alkyl ether sulphate

The pH of the formulation was 6.5. The water content of the total composition was approximately 39%. The A_w (water activity) of the composition was <0.5.

The detergent formulation was found to retain its stability for over 7 months at ambient temperature.

Comparative Example A

A detergent composition according to the present invention was prepared on the basis of the formulation presented in table 4.

Table 4

Component	Weight%
MGDA, Trilon™ M Liquid (40%)	40.37
MGDA, Trilon™ M granule (78%)	28.74
Citric acid (50%)	2.97
Poly acrylic acid polymer, anti-scalant	5.88
Bismuth citrate	0.35
Protease granule	0.88
Amylase granule	0.88
Nonionic [1]	3.6
Coated, spray-dried percarbonate	15.67
Mn catalyst granule	0.66

[1] polyoxyethylene alkyl ether, melting point > 40°C

A liquid premix base was prepared by adding Trilon™ M granule to Trilon™ M liquid at ambient temperature. Subsequently citric acid was admixed to the Trilon ™ M solution under stirring. The water content of the liquid premix so obtained was approximately 44 wt.%. Next, the remaining ingredients were added under stirring to produce a homogeneous suspension. The water content of the total composition was approximately 32%. The measured A_w (water activity) of the composition was 0.64.

The detergent composition was stored in a closed container for 2 weeks at 37°C. After this storage period the residual bleach activity was measured to be 2%.

Comparative Example B

A detergent composition was prepared on the basis of the formulation presented in table 5.

Table 5

Component	Weight%
GLDA, Dissolvine™ GL 47-S [1]	55.50
Citric Acid (50%)	6.26
DI water	13.85
Poly acrylic acid polymer, anti-scalant	3.48
Coated, spray-dried sodium percarbonate	13.07
Perfume	0.09
Dye	0.003
Phosphonate	0.004
Amylase	0.17
Protease	0.87
Nonionic [2]	1.73
Quaternary amine, ethoxylated	1.73
Zinc sulphate	0.52
Sodium sillicate	1.74
$CaCl_2.2H_2O$	0.29
Sodium benzoate	0.17
Xanthan Gum	0.52

[1] Dissolvine™ GL 47-S, a 47 wt% solution of GLDA tetrasodium salt ex Akzo Nobel.

[2] polyoxyethylene alkyl ether (melting point < 25°C)

The water content of the total composition was approximately 47%. The measured A_w (water activity) of the composition was 0.72.

The detergent composition was stored in a closed container for 2 weeks at 37°C. After this storage period the residual bleach activity was measured to be 2%.

CLAIMS

1. A composition comprising 20-99.7 wt.% of a continuous phase containing glutamic-N,N-diacetate (GLDA), water and optionally other components; and 0.3-80 wt.% of a dispersed phase comprising at least 0.3% of bleach particles by weight of the composition, said bleach particles having a particle size of at least 10 μιτι, wherein the continuous phase contains at least 10 wt.% of GLDA and at least 10wt. % water and wherein the weight ratio of GLDA to water lies within the range of 5:6 to 5:1, preferably of 9:10 to 3:1; more preferably of 1:1 to 5:2.

2. Composition according to claim 1, wherein the continuous phase contains 0.1-10 wt.% of dissolved acid.

3. Composition according to claim 2, wherein the dissolved acid is selected from sulphuric acid, citric acid and combinations thereof.

4. Composition according to any one of the preceding claims, wherein the composition has a water activity of not more than 0.7, preferably of not more than 0.6.

5. Composition according to any one of the preceding claims, wherein the composition has a water activity of at least 0.2, preferably of at least 0.3.

6. Composition according to claim any one of the preceding claims, wherein GLDA and water together represent at least 60% by weight of the continuous phase.

7. Composition according to any one of the preceding claims, wherein the composition comprises coated bleach particles.

8. Composition according to any one of the preceding claims, wherein the bleach particles comprise peroxide, preferably percarbonate.

9. Composition according to any one of the preceding claims, wherein the bleach particles comprise a water-soluble coating.

10. Composition according to clam 8, wherein the water-soluble coating comprises a coating agent selected from alkali sulphate, alkali carbonate, alkali chloride and combinations thereof.

11. Composition according to any one of the preceding claims, wherein the composition contains enzyme.

12. Composition according to any one of the preceding claims, wherein the composition is a liquid, a gel or a paste.

13. Composition according to any one of the preceding claims, wherein the composition, when added to demineralised water having a temperature of 20°C in a concentration of 1g per 100 ml of water, yields an aqueous solution having a pH of 7-13, preferably of 8-12, most preferably from 9-11.4.

14. A water-soluble sachet filled with a detergent composition according to any one of the preceding claims.

15. A process of preparing a liquid base for a detergent composition comprising GLDA and water in a weight ratio of GLDA to water of 5:6 to 5:1, said process comprising combining 100 parts by weight of a liquid mixture of GLDA and water with 10-300 parts by weight of GLDA powder, said liquid mixture containing 20-50 wt.% of GLDA and 50-80 wt.% water and said GLDA powder containing at least 65 wt.% GLDA.

16. Process according to claim 14, wherein bleach particles are added to the liquid base in an amount of at least 0.3% by weight of said liquid base.

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2012/074947

A. CLASSIFICATION OF SUBJECT MATTER

INV. C11D3/33 C11D3/39 C11D17/04

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Week 201180 Thomson Scientific, London, GB; AN 2011-P45328 XP002675668, & WO 2011/145420 Al (LION CORP) 24 November 2011 (2011-11-24) abstract -----	1-15
A	DE 197 02 734 Al (BASF AG [DE]) 30 July 1998 (1998-07-30) page 3, lines 1-8; claims page 8, line 65 -----	1-15
	-/- ·	

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" documentwhich may throw doubts on priority claim(s) orwhich is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search 9 April 2013	Date of mailing of the international search report 16/04/2013
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Pfannenstein, Heide

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International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Week 200003 Thomson Scientific, London, GB; AN 2000-033795 XP002675669 , & JP 11 302694 A (KAO CORP) 2 November 1999 (1999-11-02) abstract -----	1
A	WO 2007/141527 A1 (RECKITT BENCKISER NV [NL] ; RECKITT BENCKISER UK LTD [GB]) 13 December 2007 (2007-12-13) cited in the application page 14 - page 15; claims ; examples -----	1-15

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2012/074947

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