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(54) **Detergent particles**

(57) The present invention relates to spray-dried particles suitable for incorporation into detergent compositions comprising from 10 to 54 wt % (anhydrous basis) zeolite, from 8 to 75 wt % polycarboxylate polymer, and optional additional ingredients to 100 % by weight. The particles have been found to reduce caking of detergent product whilst providing improved cleaning com-

pared with the addition of the polycarboxylate polymer as a separate granule. Preferred particles also include soap. The invention also includes detergent compositions comprising such particles. The particles may be incorporated into a finished detergent composition as part of an agglomerate or extrudate.

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Description**Technical field**

5 **[0001]** The present invention relates to the field of detergent compositions and in particular to particles which comprise polymer and which are suitable for use in detergent compositions.

Background of the Invention and Prior Art

10 **[0002]** It is often desirable to incorporate polycarboxylate polymers into detergent compositions as part of an effective builder system. They are available from suppliers as powder, in solution or in granular form. EP-A-421664 states that these types of polymers are generally added to detergent formulations either in the form of a dried powder that is formed by spray-drying a solution, dispersion, slurry or emulsion of polymer in a liquid ("wet polymer"), or directly as wet polymer to a detergent formulation in slurry form before drying. It is then stated that in both cases the product has

15 a number of undesirable characteristics: the powder formed by spray drying wet polymer is said to be hygroscopic and therefore tends to become sticky upon storage or in the final formulation. EP-A-421664 solves the problem by providing an agglomerate made by agglomerating a dry polymer and an inorganic component.

[0003] It is well known to incorporate these polymers into spray dried detergent base granules. They are usually incorporated at low levels. DE-A-3316513 describes use of wet polymer to prepare a zeolite particle. The problem addressed is that of effectively dispersing zeolite in aqueous wash liquor in view of the water insoluble, finely particulate nature of zeolite. The particles disclosed contain 70 to 95 wt% zeolite and 5 to 30 wt% polycarboxylic acid polymer.

[0004] Trade literature from suppliers of these polycarboxylate polymers and patent publications such as EP-A-658189 and EP-A-759463 teach that the suppliers granules consisting of these polymers (generally containing at least 90 wt % polymer) give the best building benefits, more specifically anti-incrustation benefits.

25 **[0005]** However, the present inventors have found that polycarboxylate polymers incorporated into detergent formulations in the form of such polymer granules still result in caking of the detergent compositions. In addition, agglomeration of the dry powder polymer is expensive. It is therefore an object of the present invention to provide a polycarboxylate polymer in particulate form for addition to detergent compositions in a cost-effective manner whilst overcoming the caking problems associated with these materials and without losing their anti-incrustation benefits.

Definition of the Invention

[0006] In accordance with the present invention there is provided a spray dried detergent particle comprising from 10 to 54 wt % (anhydrous basis) zeolite, from 8 to 75 wt % polycarboxylate polymer, and optional additional ingredients to 100 % by weight. In a preferred embodiment, the level of polycarboxylate polymer is from 12 to 50 wt % and of zeolite is 10 to 50 wt %. A preferred additional ingredient comprises sodium carbonate. Detergent compositions comprising said particles is a further embodiment of the invention. A spray drying process for preparing these particles is a still further aspect of the invention. The inventors have found that the particles of the present invention also provide improved cleaning under cold water conditions. This is surprising in view of the suppliers trade literature and teachings

40 such as EP-A-658189 and EP-A-759463 which teach the use of granules of these polymeric materials in contrast to incorporating them via spray-drying processes.

Detailed Description of the Invention

45 **[0007]** The polycarboxylate polymers include homopolymers or copolymers. Suitable polymers include homopolymers or copolymers of dicarboxylic acids such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, citraconic acid and the anhydrides of dicarboxylic acids, such as maleic anhydride; monocarboxylic acids such as acrylic acid, methacrylic acid, vinyl acetic acid, crotonic acid and acryloxypropionic acid.

[0008] Polymers can be in acid or neutralized or partially neutralized form with Na, K, or other counterions. Preferred polycarboxylate polymers are homopolymers of acrylic acid and copolymers of acrylic and maleic acids. Especially preferred are the acrylic/maleic copolymers available from BASF as Sokalan (tradename) CP5 and CP7 (salt form) and CP45 (acid form).

[0009] The average molar mass M_w of the polymers is typically from 500 to 5,000,000. Preferably the molecular weight will be above 10 000, more preferably above 20 000. The molecular weight may be below 1 000 000, but is usually below 500 000 or even 100 000.

55 **[0010]** The zeolite (alkali metal aluminosilicate) is present in an amount of from 10 to 54 wt % (based on anhydrous material). Preferably there will be at least 12 wt % and more preferably at least 15 wt % or even at least 20 wt % zeolite. The particle may comprise no greater than 50 wt % or even no greater than 45 wt % zeolite (anhydrous basis). The

alkali metal aluminosilicate may be either crystalline or amorphous or mixtures thereof, having the general formula: $0.8-1.5 \text{ Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 0.8-6 \text{ SiO}_2$. These materials contain some bound water.

[0011] Suitable zeolites are described for example, in GB 1 473 201 (Henkel) and GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A, X and P, and mixtures thereof. The zeolite may be the the commercially available zeolite 4A now widely used in laundry detergent powders. The zeolite builder incorporated in the compositions of the invention may be maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070B (Unilever). Zeolite MAP is defined as an alkali metal aluminosilicate of the zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably not exceeding 1.15.

[0012] In a further advantageous embodiment of the invention, preferably the spray dried particle also comprises from 0.05 to 50 wt % of a shear thinning component. At higher levels, the viscosity is too low for effective spray drying processes. This may be any shear-thinning detergent additive. It may be a hydrotrope or wax but is preferably a soap or surfactant or mixtures thereof. Soap and/or anionic and/or non-ionic surfactants are preferred, with soap being most preferred. Any conventional soap may be used. It is preferably added to the spray drying process by incorporation of solid particles which may be flakes or noodles or other solid particles, of soap into the slurry for spray drying. Alternatively, where the slurry is sufficiently alkaline to form the soap salt in situ, a fatty acid may be added to the slurry to generate soap in situ. The acid precursors of the anionic surfactants may also be formed in situ in this way.

[0013] Suitable surfactants may be any used in detergent compositions as described below. Preferred are the anionic surfactants described below. Where present the shear-thinning component is preferably present at levels of from 1 to 30 wt %, more preferably from 2 to 15 or 10 wt % of the particle. The inventors have found an additional surprising benefit associated with the use of shear-thinning components comprising soap; soap is a useful suds suppressor in laundry detergents for automatic washing machines. However, it is often associated with leaving residues on laundered clothes particularly noticeable on dark-coloured fabrics. The inventors have found that incorporation of the soap into the particles of the present invention produces a significant benefit in reducing these residues.

[0014] In a further embodiment of the invention, chelants or mixtures of chelants are present in the particles of the invention, generally at levels from 0 to 45 wt %, preferably at levels from 1 to 20 wt % or even 2 to 15 wt %. Suitable chelants can be selected from the group consisting of carboxylates, phosphonates, polyfunctionally-substituted aromatic chelants and mixtures thereof. The chelant is preferably a phosphonic acid or succinic acid, or salt thereof.

[0015] Useful carboxylates include ethylenediaminetetracetates ("EDTA"), N-hydroxyethylethylene diaminetriacetates, nitrilotriacetates, ethylene diamine tetrapropionates, triethylene tetraaminehexacetates, diethylenetriaminepentaacetates, and ethanoldiglycines, alkali metal, ammonium, and substituted ammonium salts thereof, and mixtures thereof.

[0016] Useful phosphonates include ethylenediaminetetrakis (methylenephosphonates), sold as DEQUEST®. Preferably these amino phosphonates do not contain alkyl or alkenyl groups with more than about 6 carbon atoms. Particularly preferred chelants are diethylene triamine penta (methylene phosphonic acid) ("DTPMP") and ethylene diamine tetra (methylene phosphonic acid) (EDTMP) and hydroxyethylenediphosphonate (HEDP).

[0017] Polyfunctionally-substituted aromatic chelants are also useful in the components herein. See US-A- 3 812 044, issued May 21, 1974, to Connor et al. Preferred compounds of this type in acid form are dihydroxydisulfobenzenes such as 1,2-dihydroxy-3,5-disulfobenzene.

[0018] A preferred biodegradable chelant for use herein is ethylene diamine-N, N-disuccinate ("EDDS"), especially the [S,S] isomer as described in US-A-4 704 233, issued on November 3, 1987, to Hartman and Perkins.

[0019] Magnesium salts, particularly magnesium sulphate, have been found to be useful in ensuring efficient processing in the spray drying process. In particular when chelant is present in the particles of the invention, preferably a magnesium salt is also present. Generally the magnesium salt will be present at levels of from 0-40 wt % of the particle, preferably 0.1 to 10 wt %, more preferably from 0.5 to 5 wt %. Magnesium sulphate is particularly preferred. The chelating agent may be precomplexed with a metal salt such as magnesium in order to provide some protection from degradation in the presence of bleach. Preferably this is done by dissolving a salt of the metal ion into a solution of the chelating agent in the required ratios. The molar ratio of metal ion to chelating agent is preferably at least 1:1, the present invention allows molar ratios of greater than 3:1 to be prepared, most preferred is a molar ratio of about 5:1. Again, whilst any metal salt may be used, magnesium sulphate is most preferred.

[0020] The particles of the present invention generally also contain an additional inorganic component such as sodium carbonate, phosphate or silicate. Sodium carbonate and/or silicate are preferred, sodium carbonate being most preferred. Such an inorganic component is preferably added in amounts from 0 up to 30 wt%, generally in amounts from 1 to 20 wt% or from 2 to 15 wt%.

[0021] In a further embodiment of the invention, the particles additionally contain other ingredients which are incorporated in detergent compositions in minor amounts, such as at levels below 10 wt%, more usually below 5 wt%, or even below 2 wt% or even lower in the finished detergent product. Examples of such materials include polymers useful in detergent compositions such as soil release polymers, optical brighteners, dye-transfer inhibitors (such as PVP, PVNO, PVPVI and combinations thereof),

anti-redeposition agents such as CMC, etc. In particular the present invention is useful for incorporating into a detergent composition components which are used at low levels in finished product and which are available from suppliers in an aqueous-based form such as a solution of suspension or dispersion in water. These materials can be added directly to the slurry for spray-drying.

[0022] The particles of the present invention are made by a spray-drying process. The polymer, zeolite and optional additional ingredients are prepared into a slurry which is then spray-dried by conventional means. Generally this will be in a spray-drying tower using a high pressure (e.g. 6000-7000kPa) spray nozzle. Spinning disc atomisers may also be used. Generally raw materials which are provided by suppliers in solution or dispersion in water are pre-mixed and the solids including zeolite subsequently added.

[0023] The particles produced will generally have a bulk density at least 300g/l or at least 400 g/l (as measured by the method now described). The final density of the particles and compositions herein can be measured by a simple technique which involves dispensing a quantity of the granular material into a container of known volume, measuring the weight of material and reporting the density as grams/liter. The method used herein allows the material to flow into the measuring container under gravity, and without pressure or other compaction in the measuring container. The density measurements should be run at room temperature. The granular material whose density is being measured should be at least 24 hours old and should be held at room temperature for 24 hours prior to testing. A relative humidity of 50 % or less is convenient. Of course, any clumps in the material should be gently broken up prior to running the test. The sample of material is allowed to flow through a funnel mounted on a filling hopper and stand (#150; Seedburo Equipment Company, Chicago, Illinois) into an Ohaus cup of known volume and weight (#104; Seedburo). The top of the cup is positioned about 50 mm from the bottom of the funnel, and the cup is filled to overflowing. A spatula or other straight edge is then scraped over the top of the cup, without vibration or tapping, to level the material, thereby exactly and entirely filling the cup. The weight of material in the cup is then measured. Density can be reported as g/l. Two repeat runs are made and the bulk density is reported as an average of the three measurements. Relative error is about 0.4 %.

[0024] In a further embodiment of the invention the particles are further processed to incorporate them into conventional granules such as agglomerates or extrudates. In this embodiment of the invention, any solid particulate in a conventional granulation process is wholly or partially replaced by the spray-dried particles of the present invention. Suitable conventional and known granulation processes include using a pan-granulator, fluidized bed, Schugi mixer, Lödige ploughshare mixer, rotating drum or other low energy mixers, marumeriser or spheroniser; by compaction, including extrusion and tableting; when melt binding agents are used by prilling and pastilling using a Sandvik Roto Former; and by high shear processes in which the mixers have a high speed stirring and cutting action. Suitable mixers will be well known to those skilled in the art.

[0025] Suitable processes are described in the patent literature: an example of an agglomeration process is described in US 5133924 (Appel). An example of a suitable fluidised bed agglomeration process is described for example in WO97/22685 (Dhanuka). Suitable extrusion processes are described for example in WO97/03181 (EP-A-840780) (Henkel) or in EP-A-518888 (Henkel).

[0026] In accordance with a further embodiment of the invention, there is provided a detergent composition comprising a particle as described above. Suitable detergent compositions may be for any cleaning purpose, but the invention is particularly directed to laundry washing applications. The detergent composition will generally be in the form of a solid composition. Solid compositions include powders, granules, noodles, flakes, bars, tablets, and combinations thereof. The detergent composition may be in the form of a liquid composition. The detergent composition may also be in the form of a paste, gel, liqui-gel, suspension, or any combination thereof. The detergent composition may be at least partially enclosed, preferably completely enclosed, by a film or laminate such as a water-soluble and/or water-dispersible material. Preferred water-soluble and/or water-dispersible materials are polyvinyl alcohols and/or carboxymethyl celluloses.

[0027] The detergent compositions of the invention are preferably granular detergents having an overall bulk density of from 550 to 1000 g/l, more preferably 650 to 1000g/l or even 700 to 900g/l. Generally the particles of the invention will be mixed with other detergent particles including combinations of agglomerates, spray-dried powders and/or dry added materials such as bleaching agents, enzymes etc, to provide a level of polycarboxylate polymer in the finished product from 0.1 or from 1 wt% based on finished product up to 10 wt%, or up to 7 wt% or even up to 5 wt%. Generally this means that the particles of the invention may be added into a detergent composition in amounts generally from 1 to 30 wt% based on finished product, or from 1 to 20 wt% or from 1 to 10 wt% based on finished product.

[0028] Preferably, the detergent particles or the composition has a size average particle size of from 200µm to 2000µm, preferably from 350µm to 600µm.

Detergent compositions incorporating the Particles.

[0029] As described above, detergent compositions comprising the particles of the invention will comprise at least

some of the usual detergent adjunct materials, such as agglomerates, extrudates, other spray dried particles having different composition to those of the invention, or dry added materials. Conventionally, surfactants are incorporated into agglomerates, extrudates or spray dried particles along with solid materials, usually builders, and these may be admixed with the spray dried particles of the invention. However, as described above some or all of the solid material may be replaced with the particles of the invention.

[0030] The detergent adjunct materials are typically selected from the group consisting of deterative surfactants, builders, polymeric co-builders, bleach, chelants, enzymes, anti-redeposition polymers, soil-release polymers, polymeric soil-dispersing and/or soil-suspending agents, dye-transfer inhibitors, fabric-integrity agents, suds suppressors, fabric-softeners, flocculants, perfumes, whitening agents, photobleach and combinations thereof.

[0031] The precise nature of these additional components, and levels of incorporation thereof will depend on the physical form of the composition or component, and the precise nature of the washing operation for which it is to be used.

[0032] A highly preferred adjunct component is a surfactant. Preferably, the detergent composition comprises one or more surfactants. Typically, the detergent composition comprises (by weight of the composition) from 0% to 50%, preferably from 5% and more preferably from 10 or even 15 wt% to 40%, or to 30%, or to 20% one or more surfactants.

Preferred surfactants are anionic surfactants, non-ionic surfactants, cationic surfactants, zwitterionic surfactants, amphoteric surfactants, cationic surfactants and mixtures thereof.

[0033] Preferred anionic surfactants comprise one or more moieties selected from the group consisting of carbonate, phosphate, sulphate, sulphonate and mixtures thereof. Preferred anionic surfactants are C₈₋₁₈ alkyl sulphates and C₈₋₁₈ alkyl sulphonates. Suitable anionic surfactants incorporated alone or in mixtures in the compositions of the invention are also the C₈₋₁₈ alkyl sulphates and/or C₈₋₁₈ alkyl sulphonates optionally condensed with from 1 to 9 moles of C₁₋₄ alkylene oxide per mole of C₈₋₁₈ alkyl sulphate and/or C₈₋₁₈ alkyl sulphonate. The alkyl chain of the C₈₋₁₈ alkyl sulphates and/or C₈₋₁₈ alkyl sulphonates may be linear or branched, preferred branched alkyl chains comprise one or more branched moieties that are C₁₋₆ alkyl groups. Other preferred anionic surfactants are C₈₋₁₈ alkyl benzene sulphates and/or C₈₋₁₈ alkyl benzene sulphonates. The alkyl chain of the C₈₋₁₈ alkyl benzene sulphates and/or C₈₋₁₈ alkyl benzene sulphonates may be linear or branched, preferred branched alkyl chains comprise one or more branched moieties that are C₁₋₆ alkyl groups.

[0034] Other preferred anionic surfactants are selected from the group consisting of: C₈₋₁₈ alkenyl sulphates, C₈₋₁₈ alkenyl sulphonates, C₈₋₁₈ alkenyl benzene sulphates, C₈₋₁₈ alkenyl benzene sulphonates, C₈₋₁₈ alkyl di-methyl benzene sulphate, C₈₋₁₈ alkyl di-methyl benzene sulphonate, fatty acid ester sulphonates, di-alkyl sulphosuccinates, and combinations thereof. The anionic surfactants may be present in the salt form. For example, the anionic surfactant may be an alkali metal salt of one or more of the compounds selected from the group consisting of: C₈₋₁₈ alkyl sulphate, C₈₋₁₈ alkyl sulphonate, C₈₋₁₈ alkyl benzene sulphate, C₈₋₁₈ alkyl benzene sulphonate, and combinations thereof. Preferred alkali metals are sodium, potassium and mixtures thereof. Typically, the detergent composition comprises from 10% to 30wt% anionic surfactant.

[0035] Preferred non-ionic surfactants are selected from the group consisting of: C₈₋₁₈ alcohols condensed with from 1 to 9 of C₁₋₄ alkylene oxide per mole of C₈₋₁₈ alcohol, C₈₋₁₈ alkyl N-C₁₋₄ alkyl glucamides, C₈₋₁₈ amido C₁₋₄ dimethyl amines, C₈₋₁₈ alkyl polyglycosides, glycerol monoethers, polyhydroxyamides, and combinations thereof. Typically the detergent compositions of the invention comprises from 0 to 15, preferably from 2 to 10 wt% non-ionic surfactant.

[0036] Preferred cationic surfactants are quaternary ammonium compounds. Preferred quaternary ammonium compounds comprise a mixture of long and short hydrocarbon chains, typically alkyl and/or hydroxyalkyl and/or alkoxyalkyl chains. Typically, long hydrocarbon chains are C₈₋₁₈ alkyl chains and/or C₈₋₁₈ hydroxyalkyl chains and/or C₈₋₁₈ alkoxyalkyl chains. Typically, short hydrocarbon chains are C₁₋₄ alkyl chains and/or C₁₋₄ hydroxyalkyl chains and/or C₁₋₄ alkoxyalkyl chains. Typically, the detergent composition comprises (by weight of the composition) from 0% to 20% cationic surfactant.

[0037] Preferred zwitterionic surfactants comprise one or more quaternized nitrogen atoms and one or more moieties selected from the group consisting of: carbonate, phosphate, sulphate, sulphonate, and combinations thereof. Preferred zwitterionic surfactants are alkyl betaines. Other preferred zwitterionic surfactants are alkyl amine oxides. Cationic surfactants which are complexes comprising a cationic surfactant and an anionic surfactant may also be included. Typically, the molar ratio of the cationic surfactant to anionic surfactant in the complex is greater than 1:1, so that the complex has a net positive charge.

[0038] A further preferred adjunct component is a builder. Preferably, the detergent composition comprises (by weight of the composition and on an anhydrous basis) from 5% to 50% builder. Preferred builders are selected from the group consisting of: inorganic phosphates and salts thereof, preferably orthophosphate, pyrophosphate, tri-poly-phosphate, alkali metal salts thereof, and combinations thereof; polycarboxylic acids and salts thereof, preferably citric acid, alkali metal salts thereof, and combinations thereof; aluminosilicates, salts thereof, and combinations thereof, preferably amorphous aluminosilicates, crystalline aluminosilicates, mixed amorphous/crystalline aluminosilicates, alkali metal salts thereof, and combinations thereof, most preferably zeolite A, zeolite P, zeolite MAP, salts thereof, and combinations thereof; silicates such as layered silicates, salts thereof, and combinations thereof, preferably sodium layered

silicate; and combinations thereof.

[0039] A preferred adjunct component is a bleaching agent. Preferably, the detergent composition comprises one or more bleaching agents. Typically, the composition comprises (by weight of the composition) from 1% to 50% of one or more bleaching agent. Preferred bleaching agents are selected from the group consisting of sources of peroxide, sources of peracid, bleach boosters, bleach catalysts, photo-bleaches, and combinations thereof. Preferred sources of peroxide are selected from the group consisting of: perborate monohydrate, perborate tetra-hydrate, percarbonate, salts thereof, and combinations thereof. Preferred sources of peracid are selected from the group consisting of: bleach activator typically with a peroxide source such as perborate or percarbonate, preformed peracids, and combinations thereof. Preferred bleach activators are selected from the group consisting of: oxy-benzene-sulphonate bleach activators, lactam bleach activators, imide bleach activators, and combinations thereof. A preferred source of peracid is tetra-acetyl ethylene diamine (TAED) and peroxide source such as percarbonate. Preferred oxy-benzene-sulphonate bleach activators are selected from the group consisting of: nonanoyl-oxy-benzene-sulphonate, 6-nonamido-caproyl-oxy-benzene-sulphonate, salts thereof, and combinations thereof. Preferred lactam bleach activators are acyl-caprolactams and/or acyl-valerolactams. A preferred imide bleach activator is N-nonanoyl-N-methyl-acetamide.

[0040] Preferred preformed peracids are selected from the group consisting of N,N-phthaloyl-amino-peroxycaproic acid, nonyl-amido-peroxyadipic acid, salts thereof, and combinations thereof. Preferably, the STW-composition comprises one or more sources of peroxide and one or more sources of peracid. Preferred bleach catalysts comprise one or more transition metal ions. Other preferred bleaching agents are di-acyl peroxides. Preferred bleach boosters are selected from the group consisting of: zwitterionic imines, anionic imine polyions, quaternary oxaziridinium salts, and combinations thereof. Highly preferred bleach boosters are selected from the group consisting of: aryliminium zwitterions, aryliminium polyions, and combinations thereof. Suitable bleach boosters are described in US360568, US5360569 and US5370826.

[0041] A preferred adjunct component is an anti-redeposition agent. Preferably, the detergent composition comprises one or more anti-redeposition agents. Preferred anti-redeposition agents are cellulosic polymeric components, most preferably carboxymethyl celluloses.

[0042] A preferred adjunct component is a chelant. Preferably, the detergent composition comprises one or more chelants. Preferably, the detergent composition comprises (by weight of the composition) from 0.01 % to 10% chelant. Preferred chelants are selected from the group consisting of: hydroxyethane-dimethylene-phosphonic acid, ethylene diamine tetra(methylene phosphonic) acid, diethylene triamine pentacetate, ethylene diamine tetraacetate, diethylene triamine penta(methyl phosphonic) acid, ethylene diamine disuccinic acid, and combinations thereof.

[0043] A preferred adjunct component is a dye transfer inhibitor. Preferably, the detergent composition comprises one or more dye transfer inhibitors. Typically, dye transfer inhibitors are polymeric components that trap dye molecules and retain the dye molecules by suspending them in the wash liquor. Preferred dye transfer inhibitors are selected from the group consisting of: polyvinylpyrrolidones, polyvinylpyridine N-oxides, polyvinylpyrrolidone-polyvinylimidazole copolymers, and combinations thereof.

[0044] A preferred adjunct component is an enzyme. Preferably, the detergent composition comprises one or more enzymes. Preferred enzymes are selected from the group consisting of: amylases, arabinosidases, carbohydrases, cellulases, chondroitinases, cutinases, dextranases, esterases, β -glucanases, gluco-amylases, hyaluronidases, keratanases, laccases, ligninases, lipases, lipoxxygenases, malanases, mannanases, oxidases, pectinases, pentosanases, peroxidases, phenoloxidases, phospholipases, proteases, pullulanases, reductases, tannases, transferases, xylanases, xyloglucanases, and combinations thereof. Preferred enzymes are selected from the group consisting of: amylases, carbohydrases, cellulases, lipases, proteases, and combinations thereof.

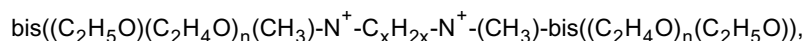
[0045] A preferred adjunct component is a fabric integrity agent. Preferably, the detergent composition comprises one or more fabric integrity agents. Typically, fabric integrity agents are polymeric components that deposit on the fabric surface and prevent fabric damage during the laundering process. Preferred fabric integrity agents are hydrophobically modified celluloses. These hydrophobically modified celluloses reduce fabric abrasion, enhance fibre-fibre interactions and reduce dye loss from the fabric. A preferred hydrophobically modified cellulose is described in WO99/14245. Other preferred fabric integrity agents are polymeric components and/or oligomeric components that are obtainable, preferably obtained, by a process comprising the step of condensing imidazole and epichlorhydrin.

[0046] A preferred adjunct component is a salt. Preferably, the detergent composition comprises one or more salts. The salts can act as alkalinity agents, buffers, builders, co-builders, encrustation inhibitors, fillers, pH regulators, stability agents, and combinations thereof. Typically, the detergent composition comprises (by weight of the composition) from 5% to 60% salt. Preferred salts are alkali metal salts of aluminate, carbonate, chloride, bicarbonate, nitrate, phosphate, silicate, sulphate, and combinations thereof. Other preferred salts are alkaline earth metal salts of aluminate, carbonate, chloride, bicarbonate, nitrate, phosphate, silicate, sulphate, and combinations thereof. Especially preferred salts are sodium sulphate, sodium carbonate, sodium bicarbonate, sodium silicate, sodium sulphate, and combinations thereof. Optionally, the alkali metal salts and/or alkaline earth metal salts may be anhydrous.

[0047] A preferred adjunct component is a soil release agent. Preferably, the detergent composition comprises one

or more soil release agents. Typically, soil release agents are polymeric compounds that modify the fabric surface and prevent the redeposition of soil on the fabric. Preferred soil release agents are copolymers, preferably block copolymers, comprising one or more terephthalate unit. Preferred soil release agents are copolymers that are synthesised from dimethylterephthalate, 1,2-propyl glycol and methyl capped polyethyleneglycol. Other preferred soil release agents are anionically end capped polyesters.

[0048] A preferred adjunct component is a soil suspension agent. Preferably, the detergent composition comprises one or more soil suspension agents. Preferred soil suspension agents are polymeric polycarboxylates. Especially preferred are polymers derived from acrylic acid, polymers derived from maleic acid, and co-polymers derived from maleic acid and acrylic acid. In addition to their soil suspension properties, polymeric polycarboxylates are also useful co-builders for laundry detergents. Other preferred soil suspension agents are alkoxyated polyalkylene imines. Especially preferred alkoxyated polyalkylene imines are ethoxyated polyethylene imines, or ethoxyated-propoxyated polyethylene imine. Other preferred soil suspension agents are represented by the formula:



wherein, n=from 10 to 50 and x=from 1 to 20. Optionally, the soil suspension agents represented by the above formula can be sulphated and/or sulphonated.

Softening system

[0049] The detergent compositions of the invention may comprise softening agents for softening through the wash such as clay optionally also with flocculant and enzymes.

[0050] Further more specific description of suitable detergent components can be found in WO97/11151.

Examples

[0051] The following are examples of the invention.

Example A

[0052]

Table 1

Raw Material	% Slurry
EDDS (ethylenediamine - N,N'- disuccinic acid (S,S isomer) in the form of its sodium salt)	2.8
MgSO ₄	1.1
Maleic acid/acrylic acid copolymer (salt form) Sokalan CP5 (tradename from BASF)	10.8
HEDP (1,1-hydroxyethane diphosphonic acid)	5.6
Soap	6.5
Zeolite (anhydrous basis)	37.0
Miscellaneous	5.6
Water	30.6
Total parts	100.0

[0053] A homogenous aqueous slurry of the components shown above was made up with a moisture content of 30.6%. The slurry was heated to 80°C and fed under high pressure, (6,000-7,000 kPa), into a conventional counter-current spray drying tower with an air inlet temperature of 300-310°C. The atomised slurry was dried to produce a granular solid which was then cooled and sieved to remove oversize (>2mm). Fine (<0.15mm) material was elutriated with the exhaust air in the spray-drying tower and collected in a containment system. The finished granules had a moisture content of about 10% by weight, a bulk density of 383 g/l and a particle size distribution such that 56.4% by weight of the granules were between 150-710 microns in size. The particles formed were free-flowing. The spray-dried powder had a composition as shown in table 2 below.

Table 2

Raw Material	% Spray dried granule
EDDS	3.7
MgSO ₄	1.5
MA/AA (Sokalan CP5)	14.0
HEDP	7.3
Soap	8.4
Zeolite A	47.9
Miscellaneous	7.4
Water	9.8
Total Parts	100.0

These particles of example A are incorporated into the following solid laundry detergent compositions according to the invention (Table 3). Amounts given below are percentages by weight of the fully formulated detergent composition.

Table 3

Ingredient	A	B	C	D	E
Example A particles	7%	13%	15%	10%	10%
Sodium linear C ₁₁₋₁₃ alkylbenzene sulfonate	11%	12%	10%	18%	15%
R ₂ N ⁺ (CH ₃) ₂ (C ₂ H ₄ OH), wherein R ₂ = C ₁₂₋₁₄ alkyl group	0.6%	1%			0.6%
Sodium C ₁₂₋₁₈ linear alkyl sulfate condensed with an average of 3 to 5 moles of ethylene oxide per mole of alkyl sulfate		0.3%	2%	2%	
Mid chain methyl branched sodium C ₁₂ -C ₁₄ linear alkyl sulfate	1.4%	1.2%	1%		
Sodium C ₁₂₋₁₈ linear alkyl sulfate	0.7%	0.5%			
C ₁₂₋₁₈ linear alkyl ethoxylate condensed with an average of 3-9 moles of ethylene oxide per mole of alkyl alcohol		3%	2%		
Citric acid	2%	1.5%			2%
Sodium tripolyphosphate (anhydrous weight given)			20%	25%	22%
Sodium carboxymethyl cellulose	0.2%	0.2%		0.3%	
Sodium polyacrylate polymer having a weight average molecular weight of from 3000 to 5000		0.5%	1%		0.7%
Copolymer of maleic/acrylic acid, having a weight average molecular weight of from 50,000 to 90,000, wherein the ratio of maleic to acrylic acid is from 1:3 to 1:4 (Sokalan CP5 from BASF)	1.2%	0.5%			
Diethylene triamine pentaacetic acid	0.2%		0.5%	0.2%	0.3%
Proteolytic enzyme having an enzyme activity of from 15 mg/g to 70 mg/g	0.2%	0.2%	0.5%	0.4%	0.3%
Amylolytic enzyme having an enzyme activity of from 25 mg/g to 50 mg/g	0.2%	0.2%	0.3%	0.4%	0.3%

Table 3 (continued)

Ingredient	A	B	C	D	E
Lipolytic enzyme having an enzyme activity of 5 mg/g to 25 mg/g		0.2%	0.1 %		
Anhydrous sodium perborate monohydrate			20%	5%	8%
Sodium percarbonate	10%	12%			
Magnesium sulfate					
Nonanoyl oxybenzene sulfonate				2%	1.2%
Tetraacetylenediamine	3%	4%	2%	0.6%	0.8%
Brightener	0.1%	0.1%	0.2%	0.1%	0.1%
Sodium carbonate	10%	10%	10%	19%	22%
Sodium sulfate	20%	15%	5%	13%	1%
Zeolite A	20%	15%		2%	14%
Sodium silicate (2.0 R)		0.2%		1%	1%
Crystalline layered silicate	3%	5%	10%		
Photobleach	0.002%				
Polyethylene oxide having a weight average molecular weight from 100 to 10,000	2%	1%			
Perfume spray-on	0.2%	0.5%	0.25%	0.1%	
Starch encapsulated perfume	0.4%				
Silicone based suds suppressor	0.05%	0.05%			0.02%
Miscellaneous and moisture	To 100%	To 100%	To 100%	To 100%	To 100%

Claims

1. A spray- dried particle comprising from 10 to 54 wt % (anhydrous basis) zeolite, from 8 to 75 wt % polycarboxylate polymer, and optional additional ingredients to 100 % by weight.
2. A spray-dried particle according to claim 1 comprising from 12 to 50 wt% polycarboxylate polymer and preferably, from 10 to 50 wt % zeolite (anhydrous basis).
3. A spray-dried particle according to claim 1 or claim 2 in which the polycarboxylate polymer has a molecular weight greater than 10 000.
4. A spray-dried particle according to any preceding claim additionally comprising 0.05 to 20 wt % shear-thinning component.
5. A spray-dried particle according to claim 4 in which the shear-thinning component comprises from 0.5 to 18 wt% soap and/or surfactant, preferably soap.
6. A spray-dried particle according to any preceding claim comprising at least 15 wt % zeolite (anhydrous basis).
7. A spray-dried particle according to any preceding claim additionally comprising chelant, preferably phosphonate or succinate chelant.
8. A spray-dried particle according to any preceding claim additionally comprising magnesium sulphate.
9. A method for making a spray-dried particle according to any preceding claim comprising preparing an aqueous

slurry by adding zeolite to an aqueous solution comprising polycarboxylate polymer.

10. A detergent composition comprising spray-dried particles according to any preceding claim.

5 **11.** A detergent composition according to claim 10 in which the spray-dried particles are incorporated into the detergent composition as part of an agglomerate or extrudate with other detergent adjunct ingredients.

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EUROPEAN SEARCH REPORT

Application Number
EP 03 25 7082

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EUROPEAN SEARCH REPORT

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