

(11) EP 3 415 591 A1

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:

19.12.2018 Bulletin 2018/51

(51) Int Cl.:

C11D 3/00 (2006.01) C11D 17/04 (2006.01)

C11D 3/42 (2006.01)

(21) Application number: 17176195.0

(22) Date of filing: 15.06.2017

(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 RS SE SI SK SM TR

Designated Extension States:

BA ME

Designated Validation States:

MA MD

(71) Applicant: The Procter & Gamble Company Cincinnati, OH 45202 (US)

(72) Inventors:

 SOMERVILLE ROBERTS, Nigel Patrick Newcastle-upon-Tyne, NE12 9TS (GB)

- BROOKER, Alan Thomas Newcastle upon Tyne, NE12 9TS (GB)
- PICKERING, Carly Newcastle upon Tyne, NE12 9TS (GB)
- BUJIS, Ned Peter Newcastle upon Tyne, NE12 9TS (GB)
- (74) Representative: Pickford, James Lawrence
 Procter & Gamble
 Technical Centres Limited
 Whitley Road
 Longbenton
 Newcastle upon Tyne NE12 9TS (GB)

(54) WATER-SOLUBLE UNIT DOSE ARTICLE COMPRISING A SOLID LAUNDRY DETERGENT COMPOSITION

(57) Water-soluble unit dose article containing solid laundry detergent composition and water-soluble film; the solid detergent composition comprises a whitening agent and has a low pH.

EP 3 415 591 A1

Description

15

20

30

35

40

45

FIELD OF THE INVENTION

⁵ **[0001]** The present invention relates to water-soluble unit dose articles containing solid laundry detergent compositions and methods of using them.

BACKGROUND OF THE INVENTION

[0002] Water-soluble laundry unit dose articles are known and are liked by consumers due to their ease and efficiency of use in the laundry operation. Water-soluble unit dose articles comprise water-soluble film defining at least one internal compartment. A laundry detergent composition is housed within the internal compartment. Upon exposure to water, the water-soluble film dissolves/disintegrates releasing the laundry detergent composition into the surrounding water.

[0003] The laundry detergent composition may be a solid laundry detergent composition. Such detergent compositions comprise known laundry detergent cleaning actives. One preferred active ingredient is an optical brightener.

[0004] Due to their compact form, water-soluble unit dose articles have limited space for formulating the solid laundry detergent compositions. This can result in lower than desired cleaning performance as extra detergent composition cannot simply be added to compensate for any under-performance. Addition of further detergent composition to the water-soluble unit dose article would require making the unit dose article bigger making it less convenient and also could contribute to negatives such as impaired dissolution when additional water soluble film would be required to accommodate the bigger water soluble unit dose article and/or reduced structural stability of the unit dose article especially when further stretching the original water soluble film to accommodate the bigger water soluble unit dose article (the film may not provide sufficient structural stability for the volume of solid detergent composition added).

[0005] Therefore, there is a need in the art to provide improved performance of said water-soluble unit dose articles without addition of increased levels of solid laundry detergent composition to the unit dose articles.

[0006] It was surprisingly found that a water-soluble unit dose article comprising a solid laundry detergent composition having a particular pH (upon dilution of the solid laundry detergent composition) provided improved fabric whiteness performance of a whitening agent formulated in said solid laundry detergent composition as compared to water-soluble unit dose articles having a solid laundry detergent composition having a higher pH (upon dilution of the solid laundry detergent composition) as known from the art.

SUMMARY OF THE INVENTION

[0007] A first aspect of the present invention is a water-soluble unit dose article comprising a water-soluble film and a solid laundry detergent composition, wherein the water-soluble film defines a first internal compartment and wherein the solid laundry detergent composition is comprised within the first internal compartment, and wherein the water soluble unit dose article composition comprises a whitening agent preferably selected from a hueing dye and an optical brightener preferably an optical brightener; and

wherein the solid composition at 1wt% dilution in deionized water at 20°C, has an equilibrium pH in the range of from 6.5 to 8.8, preferably between 6.7 and 8.5.

[0008] A second aspect of the present invention is a method of washing comprising the steps of adding the water-soluble unit dose article according to the present invention to sufficient water to dilute the solid laundry detergent composition by a factor of at least 300 fold to create a wash liquor and contacting fabrics to be washed with said wash liquor.

[0009] A third aspect of the present invention is the use of a solid laundry detergent composition comprising a non-soap surfactant and having at 1wt% dilution in deionized water at 20°C an equilibrium pH in the range of from 6.5 to 8.8

DETAILED DESCRIPTION OF THE INVENTION

50 Water-soluble unit dose article

[0010] The present invention is to a water-soluble unit dose article comprising a water-soluble film and a solid laundry detergent composition.

[0011] The water-soluble film is described in more detail below.

⁵⁵ **[0012]** The solid laundry detergent composition is described in more detail below.

in a water-soluble unit dose article to improve the performance of an optical brightener.

[0013] The water-soluble unit dose article comprises the water-soluble film shaped such that the unit-dose article comprises at least a first internal compartment surrounded by the water-soluble film. The compartment should be understood as meaning a closed internal space within the unit dose article, which holds the solid laundry detergent com-

position. The unit dose article may comprise a first water-soluble film and a second water-soluble film sealed to one another such to define the internal compartment. The water-soluble unit dose article is constructed such that the solid laundry detergent composition is comprised within the first internal compartment. The water-soluble unit dose article is constructed such that the solid laundry detergent composition does not leak out of the compartment during storage. However, upon addition of the water-soluble unit dose article to water, the water-soluble film dissolves and releases the contents of the internal compartment into the wash liquor.

[0014] During manufacture, a first water-soluble film may be shaped to comprise an open compartment into which the solid laundry detergent composition is added. A second water-soluble film is then laid over the first film in such an orientation as to close the opening of the compartment. The first and second films are then sealed together along a seal region.

10

15

20

30

35

40

45

50

55

[0015] The unit dose article may comprise more than one compartment, even at least two compartments, or even at least three compartments. The compartments may be arranged in superposed orientation, i.e. one positioned on top of the other. In such an orientation the unit dose article will comprise at least three films, top, middle and bottom. Alternatively, the compartments may be positioned in a side-by-side orientation, i.e. one orientated next to the other. The compartments may even be orientated in a 'tyre and rim' arrangement, i.e. a first compartment is positioned next to a second compartment, but the first compartment at least partially surrounds the second compartment, but does not completely enclose the second compartment. Alternatively one compartment may be completely enclosed within another compartment.

[0016] Wherein the unit dose article comprises at least two compartments, one of the compartments may be smaller than the other compartment. Wherein the unit dose article comprises at least three compartments, two of the compartments may be smaller than the third compartment, and preferably the smaller compartments are superposed on the larger compartment. The superposed compartments preferably are orientated side-by-side.

[0017] In a multi-compartment orientation, the detergent composition according to the present invention may be comprised in at least one of the compartments. It may for example be comprised in just one compartment, or may be comprised in two compartments, or even in three compartments.

[0018] Each compartment may comprise the same or different compositions. The different compositions could all be in the same form, or they may be in different forms.

[0019] The water-soluble unit dose article may comprise at least a first compartment and a second compartment, preferably at least a first compartment, a second compartment and a third compartment. Preferably, the compartments are arranged in a side-by-side arrangement, a superposed arrangement or a mixture thereof. Preferably, at least the second compartment, more preferably at least the second compartment and the third compartment are superposed onto the first compartment. The second compartment and the third compartment are preferably arranged in a side-by-side arrangement superposed onto the first compartment.

[0020] The first i.e. bottom compartment preferably comprises the free flowing solid detergent composition. The second and subsequent compartments i.e. superposed compartments comprise a liquid, a solid or a mixture thereof, preferably a liquid. All compartments might comprise a gas in addition to the enclosed compositions, preferably will comprise a gas. Without wishing to be bound by theory, such a gas will create an 'air space' in the compartment and will facilitate free flowing of the enclosed compositions, and furthermore can act as an additional barrier against eventual compartment to compartment active migration through the film. Preferably the gas is air.

[0021] The water-soluble unit dose article comprises a whitening agent. Preferably, the solid laundry detergent composition comprises the whitening agent. The solid laundry detergent composition may comprise between 0.05% and 2%, preferably between 0.1% and 1.5%, even more preferably between 0.2% and 1% by weight of the solid laundry detergent composition of the whitening agent. The whitening agent is preferably selected from a hueing dye and an optical brightener preferably an optical brightener.

[0022] The solid laundry detergent composition may comprise between 0.05% and 2%, preferably between 0.1% and 1.5%, even more preferably between 0.2% and 1% by weight of the solid laundry detergent composition of the optical brightener. The optical brightener is preferably selected from hydrophobic brighteners, stilbene brighteners, biphenyl type brighteners, or a mixture thereof, preferably selected from brightener 36, brightener 49, brightener 15 or a mixture thereof, most preferably brightener 49.

[0023] The brightener may comprise stilbenes, such as brightener 15 and brightener 36. Other suitable brighteners are hydrophobic brighteners, and biphenyl type brighteners such as brightener 49 (4,4'-bis(2-sulfostyryl)biphenyl). The brightener may be in micronized particulate form, having a weight average particle size in the range of from 3 to 30 micrometers, or from 3 micrometers to 20 micrometers, or from 3 to 10 micrometers. The brightener can be alpha or beta crystalline form.

[0024] Suitable brighteners include: di-styryl biphenyl compounds, e.g. Tinopal® CBS-X, di-amino stilbene di-sulfonic acid compounds, e.g. Tinopal® DMS pure Xtra and Blankophor® HRH, and Pyrazoline compounds, e.g. Blankophor® SN, and coumarin compounds, e.g. Tinopal® SWN.

[0025] Preferred brighteners are: sodium 2 (4-styryl-3-sulfophenyl)-2H-napthol[1,2-d]triazole, disodium 4,4'-bis{[(4-anifino-6-(N methyl-N-2 hydroxyethyl)amino 1 ,3,5- triazin-2-yl)];amino}stilbene-2-2' disulfonate, disodium 4,4'-bis{[(4-anifino-6-(N methyl-N-2 hydroxyethyl)amino 1 ,3,5- triazin-2-yl)]}stilbene-2-2' disulfonate, disodium 4,4'-bis{[(4-anifino-6-(N methyl-N-2 hydroxyethyl)amino 1 ,4,4'-bis{[(4-anifino-6-(N methyl-N-2 hydroxyethyl)amino 1 ,4,4'-bis{[(4-anifino-6-(N methyl-N-2 hydroxyethyl)amino 1 ,4,4'-bis{[(4-anifino-6-(N methyl)amino 1 ,4,4'-bis{[(4-anifino-6-(N methyl)amino 1 ,4,4'-bis{[(4-anifino-6-(N methyl)am

anilino-6-morpholino-I,3,5-triazin-2-yl)]amino} stilbene-2-2' disulfonate, and disodium 4,4'- bis(2-sulfostyryl)biphenyl. A suitable fluorescent brightener is C.I. Fluorescent Brightener 260, which may be used in its beta or alpha crystalline forms, or a mixture of these forms.

[0026] The solid laundry detergent composition may comprise between 0.05% and 2%, preferably between 0.1% and 1.5%, even more preferably between 0.2% and 1% by weight of the solid laundry detergent composition of the hueing dye. The hueing dye may comprise polymeric or non-polymeric dyes, pigments, or mixtures thereof. Preferably the hueing dye comprises a polymeric dye, comprising a chromophore constituent and a polymeric constituent. The chromophore constituent is characterized in that it absorbs light in the wavelength range of blue, red, violet, purple, or combinations thereof upon exposure to light. In one aspect, the chromophore constituent exhibits an absorbance spectrum maximum from about 520 nanometers to about 640 nanometers in water and/or methanol, and in another aspect, from about 560 nanometers to about 610 nanometers in water and/or methanol.

[0027] Although any suitable chromophore may be used, the dye chromophore is preferably selected from benzodifuranes, methine, triphenylmethanes, napthalimides, pyrazole, napthoquinone, anthraquinone, azo, oxazine, azine, xanthene, triphenodioxazine and phthalocyanine dye chromophores. Mono and di-azo dye chromophores are preferred.

[0028] The hueing dye may comprise a dye polymer comprising a chromophore covalently bound to one or more of at least three consecutive repeat units. It should be understood that the repeat units themselves do not need to comprise a chromophore. The dye polymer may comprise at least 5, or at least 10, or even at least 20 consecutive repeat units. [0029] The repeat unit can be derived from an organic ester such as phenyl dicarboxylate in combination with an oxyalkyleneoxy and a polyoxyalkyleneoxy. Repeat units can be derived from alkenes, epoxides, aziridine, carbohydrate including the units that comprise modified celluloses such as hydroxyalkylcellulose; hydroxypropyl cellulose; hydroxypropyl methylcellulose; hydroxybutyl cellulose; and, hydroxybutyl methylcellulose or mixtures thereof. The repeat units may be C2-C4 alkyleneoxy groups, sometimes called alkoxy groups, preferably derived from C2-C4 alkylene oxide. The repeat units may be C2-C4 alkoxy groups, preferably ethoxy groups.

[0030] For the purposes of the present invention, the at least three consecutive repeat units form a polymeric constituent. The polymeric constituent may be covalently bound to the chromophore group, directly or indirectly via a linking group. Examples of suitable polymeric constituents include polyoxyalkylene chains having multiple repeating units. In one aspect, the polymeric constituents include polyoxyalkylene chains having from 2 to about 30 repeating units, from 2 to about 20 repeating units, from 2 to about 10 repeating units or even from about 3 or 4 to about 6 repeating units. Non-limiting examples of polyoxyalkylene chains include ethylene oxide, propylene oxide, glycidol oxide, butylene oxide and mixtures thereof.

Water-soluble film

10

20

30

35

40

45

50

55

[0031] The film of the present invention is soluble or dispersible in water. The water-soluble film preferably has a thickness of from 20 to 150 micron, preferably 35 to 125 micron, even more preferably 50 to 110 micron, most preferably about 76 micron.

[0032] Preferably, the film has a water-solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out here after using a glass-filter with a maximum pore size of 20 microns:

 $5 \text{ grams} \pm 0.1 \text{ gram of film material is added in a pre-weighed 3L beaker and } 2L \pm 5 \text{ml of distilled water is added.}$ This is stirred vigorously on a magnetic stirrer, Labline model No. 1250 or equivalent and 5 cm magnetic stirrer, set at 600 rpm, for 30 minutes at 30°C. Then, the mixture is filtered through a folded qualitative sintered-glass filter with a pore size as defined above (max. 20 micron). The water is dried off from the collected filtrate by any conventional method, and the weight of the remaining material is determined (which is the dissolved or dispersed fraction). Then, the percentage solubility or dispersability can be calculated.

[0033] Preferred film materials are preferably polymeric materials. The film material can, for example, be obtained by casting, blow-moulding, extrusion or blown extrusion of the polymeric material, as known in the art.

[0034] Preferred polymers, copolymers or derivatives thereof suitable for use as pouch material are selected from polyvinyl alcohols, polyvinyl pyrrolidone, polyalkylene oxides, acrylamide, acrylic acid, cellulose, cellulose ethers, cellulose esters, cellulose amides, polyvinyl acetates, polycarboxylic acids and salts, polyaminoacids or peptides, polyamides, polyacrylamide, copolymers of maleic/acrylic acids, polysaccharides including starch and gelatine, natural gums such as xanthum and carragum. More preferred polymers are selected from polyacrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polymethacrylates, and most preferably selected from polyvinyl alcohols, polyvinyl alcohol copolymers and hydroxypropyl methyl cellulose (HPMC), and combinations thereof. Preferably, the level of polymer in the pouch material, for example a PVA polymer, is at least 60%. The polymer can have any weight average molecular

weight, preferably from about 1000 to 1,000,000, more preferably from about 10,000 to 300,000 yet more preferably from about 20,000 to 150,000.

[0035] Mixtures of polymers and/or copolymers can also be used as the pouch material, especially mixtures of polyvinylalcohol polymers and/or copolymers, especially mixtures of polyvinylalcohol homopolymers and/or anionic polyvinylalcohol copolymers preferably selected from sulphonated and carboxylated anionic polyvinylalcohol copolymers especially carboxylated anionic polyvinylalcohol copolymers. Most preferably the water soluble film comprises a blend of a polyvinylalcohol homopolymer and a carboxylated anionic polyvinylalcohol copolymer.

[0036] Preferred films exhibit good dissolution in cold water, meaning unheated distilled water. Preferably such films exhibit good dissolution at temperatures of 24°C, even more preferably at 10°C. By good dissolution it is meant that the film exhibits water-solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out here after using a glass-filter with a maximum pore size of 20 microns, described above.

[0037] Preferred films are those supplied by Monosol under the trade references M8630, M8900, M8779, M8310.

[0038] The film may be opaque, transparent or translucent. The film may comprise a printed area.

[0039] The area of print may be achieved using standard techniques, such as flexographic printing or inkjet printing.

[0040] The film may comprise an aversive agent, for example a bittering agent. Suitable bittering agents include, but are not limited to, naringin, sucrose octaacetate, quinine hydrochloride, denatonium benzoate, or mixtures thereof. Any suitable level of aversive agent may be used in the film. Suitable levels include, but are not limited to, 1 to 5000ppm, or even 100 to 2500ppm, or even 250 to 2000rpm.

20 Solid laundry detergent composition

10

25

30

35

40

45

50

[0041] The first internal compartment comprises a solid laundry detergent composition. The solid laundry detergent composition may comprise solid particulates or may be a single homogenous solid. Preferably, the solid laundry detergent composition comprises particles. This means the solid laundry detergent composition comprises individual solid particles as opposed to the solid being a single homogenous solid. The particles may be free-flowing or may be compacted, preferably free-flowing.

[0042] The particles may be spray-dried particles, agglomerates, extrudates or a mixture thereof. Those skilled in the art will know how to make spray-dried particles, agglomerates or extrudates using techniques commonly known in the art. [0043] The solid particulate laundry detergent composition preferably has a mean particle size of between 400 microns and 1000 microns, more preferably between 450 microns and 850 microns.

[0044] Preferably, the solid particulate laundry detergent composition has a bulk density of between 400 and 1000g/l, more preferably between 500 and 800g/l, as measured through ISO 697 test method.

[0045] Preferably, the solid particulate laundry detergent composition fills between 25% and 95%, preferably between 30% and 90%, more preferably between 40% and 80% of the available volume within the first compartment, the remaining volume preferably filled with a gas. The gas may be any suitable gas. The gas may comprise oxygen, nitrogen, carbon dioxide or a mixture thereof. The gas may be air.

[0046] The first compartment preferably comprises between 1g and 25g, preferably between 5g and 20 g, more preferably between 8g and 18g of the solid particulate laundry composition.

[0047] The solid particulate laundry detergent composition is preferably free flowing within the first internal compartment. That is to say, if the water-soluble unit dose article is moved or repositioned, the solid particulate laundry detergent composition can be seen to freely move, or flow within the first internal compartment. This is opposed to where the solid particulate laundry detergent composition is compressed such as happens when excess air is drawn out of the first internal compartment so that the film contracts and compresses around the solid particulate laundry detergent composition. Such water-soluble unit dose articles comprising compressed solids are commonly known from the art.

[0048] The solid laundry detergent composition comprises a non-soap surfactant. Preferably, the solid laundry detergent composition comprises between 20% and 75%, more preferably between 30% and 70%, most preferably between 40% and 60% by weight of the solid laundry detergent composition of the non-soap surfactant

[0049] The non-soap surfactant may comprise a non-soap anionic surfactant, a non-ionic surfactant or a mixture thereof, preferably a non-soap anionic surfactant. The solid composition may comprise a non-soap anionic surfactant, preferably, the solid laundry detergent composition comprises between 20% and 75%, more preferably between 30% and 70%, most preferably between 40% and 60% by weight of the solid laundry detergent composition of the non-soap anionic surfactant.

[0050] Preferably, the non-soap anionic surfactant comprises linear alkylbenzene sulphonate, alkoxylated alkyl sulphate or a mixture thereof, more preferably a mixture thereof. Preferably, the ratio of linear alkylbenzene sulphonate to alkoxylated alkyl sulphate preferably the ratio of linear alkylbenzene sulphonate to ethoxylated alkyl sulphate is from 1:2 to 20:1, preferably from 1.1:1 to 15:1, more preferably from 1.2:1 to 10:1, even more preferably from 1.3:1 to 5:1, even more preferably from 1.4:1 to 3:1, most preferably from 2:1 to 3:1.

[0051] Preferably, the alkoxylated alkyl sulphate is an ethoxylated alkyl sulphate with an average degree of ethoxylation

of between 0.5 and 7, preferably between 0.5 and 5, more preferably between 0.5 and 3, even more preferably from about 1 to about 2 most preferably 1 and an average alkyl chain length of between 8 and 18. Preferably the alkoxylated alkyl sulphate has an average alkyl chain length between 10 and 16, more preferably between 12 and 14. Preferably, the linear alkylbenzene sulphonate is a C_{10} - C_{16} linear alkylbenzene sulphonate or a C_{11} - C_{14} linear alkylbenzene sulphonate or a mixture thereof.

[0052] When present, preferably the non-ionic surfactant is selected from an alkoxylated alcohol preferably selected from a natural or olefin derived fatty alcohol alkoxylate, an oxo-synthesised fatty alcohol alkoxylate, Guerbet fatty alcohol alkoxylates, alkyl phenol alcohol alkoxylates or a mixture thereof. The alcohol alkoxylate may have an average degree of alkoxylation of between 0.5 and 10, preferably between 1 and 9, more preferably between 3 and 8, more preferably between 5 and 8 or even from about 7 to about 8. The alcohol alkoxylate may have an average alkyl chain length of between 8 and 18, preferably between 10 and 16, more preferably between 12 and 15.

10

30

35

40

55

[0053] The solid composition at 1wt% dilution in deionized water at 20°C has an equilibrium pH in the range of from 6.5 to 8.8, preferably between 6.7 and 8.5, more preferably between 7 and 8. Without wishing to be bound by theory, the specific low pH of the solid laundry detergent composition provides for improved fabric cleaning or treatment performance of the water-soluble unit dose article according to the present invention as compared to water-soluble unit dose articles wherein the solid laundry detergent composition has a higher pH. Such higher pH solid laundry detergent composition formulated into water-soluble unit dose articles are known in the art.

[0054] Those skilled in the art will know how to measure the pH using common known techniques. A preferred method is to obtain a 10g sample accurately weighed to two decimal places, of the solid laundry detergent composition. The sample should preferably be obtained using a Pascall sampler in a dust cabinet. Add the 10g sample to a plastic beaker and add 200 ml of carbon dioxide-free de-ionised water. Agitate using a magnetic stirrer on a stirring plate at 150 rpm until fully dissolved and for at least 15 minutes. Transfer the contents of the beaker to a 1 litre volumetric flask and make up to 1 litre with Cardon dioxide-free de-ionised water. Mix well and take a 100 mls \pm 1 ml aliquot using a 100 mls pipette immediately. Measure and record the pH and temperature of the sample using a pH meter capable of reading to \pm 0.01pH units, with stirring, ensuring temperature is 20°C +/- 0.5°C.

[0055] The solid laundry detergent composition may comprise between 0% and 10% by weight of the solid laundry detergent composition of carbonate salts. The carbonate salts may be selected from sodium carbonate, potassium carbonate, sodium bicarbonate, sodium bicarbonate, burkeite, sequicarbonate, habit modified carbonate, crystal growth modified burkeite or a mixture thereof, preferably sodium carbonate.

[0056] The solid laundry detergent composition may comprise a material selected from zeolite, sodium sulphate, silica, organic acid or a mixture thereof, preferably wherein the solid laundry detergent composition comprises between 10% and 35%, more preferably between 12% and 25% by weight of the solid laundry detergent composition of the material. [0057] The solid laundry detergent composition may comprise an organic acid, preferably between 1% and 10% by weight of the solid laundry detergent composition of an organic acid and/or a salt thereof. Preferably, the organic acid is a carboxylic acid, preferably a polycarboxylic acid, more preferably the organic acid is selected from citric acid, malic acid, lactic acid, propionic acid, valeric acid, caproic acid, carbonic acid, adipic acid, gluconic acid, methylglycinediacetic acid or a mixture thereof, most preferably citric acid. Without wishing to be bound by theory such materials may be used to control the pH of the laundry detergent composition.

[0058] The solid laundry detergent composition may comprise an adjunct ingredient, wherein the adjunct ingredient is preferably selected from cationic polymers, dye transfer inhibitors, chelants including aminocarboxylate and aminophosphonate chelants such as HEDP, enzymes, acrylate-based polymers, perfumes and perfume capsules, polyester terephthalate polymers, PEG-based polymers, ethoxylated polyethyleneimines, polysaccharides, amine oxide, aesthetic dyes, hueing dyes, antifoams, bleaching actives, or a mixture thereof.

⁴⁵ **[0059]** Preferred acrylate-based polymers are acrylate/maleate random copolymers.

[0060] Preferred polysaccharides are cationically-modified polysaccharides. Preferably, the cationically modified polysaccharide is selected from cationic guar gums, cationic cellulosic polymers, and mixtures thereof, most preferably cationic cellulosic polymers even more preferably cationically modified hydroxyethyl cellulose, most preferably, hydroxyethyl cellulose derivatised with trimethyl ammonium substituted epoxide.

50 [0061] Preferably, the particulate laundry detergent composition comprises agglomerates. Preferably, the agglomerates comprise non-soap surfactant, sodium sulphate and silica.

[0062] Another aspect of the present invention is the use of a solid laundry detergent composition comprising a non-soap surfactant and having at 1wt% dilution in deionized water at 20°C an equilibrium pH in the range of from 6.5 to 8.8 in a water-soluble unit dose article as according to the present invention to improve the performance of an optical brightener.

Method of washing

[0063] An aspect of the present invention is a method of washing comprising the steps of adding the water-soluble unit dose article according to the present invention to sufficient water to dilute the solid particulate laundry detergent composition by a factor of at least 300 fold to create a wash liquor and contacting fabrics to be washed with said wash liquor. **[0064]** The method may be performed in a hand wash operation, an automatic laundry washing machine or a mixture thereof.

Process of making

10

15

20

30

35

40

50

55

[0065] Those skilled in the art will know how to make the unit dose article and particulate laundry detergent composition of the present invention using known techniques in the art:

Water soluble pouch making:

[0066] During manufacture, a first water-soluble film may be shaped to comprise an open compartment into which the detergent composition is added. A second water-soluble film is then laid over the first film in such an orientation as to close the opening of the compartment. The first and second films are then sealed together along a seal region using known sealing means such as solvent, heat or a mixture thereof.

Preparation of free-flowing detergent powders :

[0067] Highly preferred are free-flowing detergent powders. Without wishing to be bound by theory, free-flowing detergent powders are found to have improved dissolution when formulated in a water soluble pouch compared to compressed powders, leaving less detergent residues behind accordingly. Highly free-flowing detergent powders can be prepared by the following process.

[0068] Surfactant-containing particles can be prepared by spray-drying, agglomeration or other processes such as drum drying etc. Such agglomerates are preferred due to the high surfactant loading that can be achieved.. However other processes can be used. The surfactant agglomerates preferably contain anionic surfactant, including LAS. An especially preferred feature is that the surfactant agglomerates contain a mixture of anionic surfactants, especially LAS and AES surfactant.

[0069] To improve the flowability and stability of the detergent powder(s), smaller particles are usually removed by sieving. It is especially preferred to sieve the surfactant agglomerates plus other detergent ingredients prior to any subsequent coating step. The surfactant agglomerates, plus other granular detergent ingredients such as HEDP, are sieved to remove particles smaller than 600 microns. The surfactant particles, plus any other detergent ingredients that are optionally added, are then put into a mixer where they are dusted or coated with a fine powder to provide a protective layer on the surface. An example of such a process is where blown powders are coated with non-ionic surfactant as a binder and then zeolite. It has been found, though, that dusting or coating the surfactant agglomerates (and other optional detergent ingredients) in this instance with a blend of micronized sodium sulphate and zeolite gives good results for flowability as well as appearance etc. Inclusion of a liquid binder to help the adhesion of the fine powder(s) to the surface of the larger surfactant agglomerates is also an option.

[0070] A suitable detergent mix can be prepared as follows. Surfactant agglomerates containing a blend of LAS and AExS anionic surfactants are prepared and dried to give particles with a total surfactant activity of 60% comprising a 2:1 blend of LAS to AExS surfactants. The particles contain 20% of hydrophilic silica. A suitable silica is 22S from Evonik. The balance consists of ground sodium sulphate, water and miscellaneous. The fine particles are then removed by sieving the agglomerates on a 600 micron mesh sieve. Oversize particles are removed by sieving the agglomerates through a 1400 micron mesh size sieve.

[0071] 3kg of the sieved surfactant agglomerates are then put into a 6-litre internal volume paddle mixer from Forberg. 300g of ground sodium sulphate (d90 < 100 microns) and 100g of sodium aluminosilicate type 4A are then added to the mixer and the mixer is run at maximum speed for 2 minutes, thus coating the surface of the agglomerates with sulphate and zeolite. The coated agglomerates are then removed and blended with other detergent materials to give a free-flowing detergent mixture suitable for use.

[0072] The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40 mm" is intended to mean "about 40 mm."

EXAMPLES

[0073] The below tests show a consistently improved whiteness performance profile for low versus high pH brightener comprising powder compositions within a water-soluble unit dose article.

Test 1: Whiteness - Improving brightener efficacy:

Test Method:

5

15

30

35

40

45

50

55

[0074] In order to demonstrate the impact in providing improved whiteness of formulating with a brightener in a low pH powder vs in a high pH powder contained within a PVA film pouch, a whiteness maintenance full scale test has been conducted. A short cotton cycle at 40°C and 6 gpg water hardness was selected on a Miele washing machine (model 986). Total run time was 1hour 25 minutes. 2.5 kg cotton ballast loads (sourced from Warwick Equest Ltd. Unit 55, Consett Business Park, Consett, County Durham, DH8 6BN) were added together with a soiled load (8 SBL2004 soiled ballast sheets ex wfk Testgewebe GmbH Christenfeld 10, D-41379 Brüggen-Bracht Germany order ref 10996). White fabric bundles (8 in total containing Terry Towel, Knitted Cotton, Flat Cotton, Polycotton and Polyester sourced from Warwick Equest Ltd. Unit 55, Consett Business Park, Consett, County Durham, DH8 6BN) were added to each machine and washed in either reference product or test product. Four wash cycles were carried out, removing soiled ballast sheets each time and adding four more along with the addition of product. After repeating the wash process four times, whiteness bundles were removed and tumble dried in an electric Miele tumble dryer (Novotronic T430) set to "extra dry". Fabric tracers were then analysed using a bench-top spectrophotometer Konica -Minolta model CM-3630 which when combined with Polaris White Star software (ex Axiphos GmbH Arend-Braye Str. 42, D-79540 Loerrach, Germany) allows the extraction of reflectance data in the range of 360 -740 nm. In order to determine the impact brightener in a low pH powder vs High pH powder on deposition of optical brightener Lab measurements were taken (The lightness, L*, represents the darkest black at L* = 0, and the brightest white at L* = 100. The colour channels, a* and b*, will represent true neutral grey values at a* = 0 and b* = 0. The red/green opponent colours are represented along the a* axis, with green at negative a* values and red at positive a* values. The yellow/blue opponent colors are represented along the b* axis, with blue at negative b* values and yellow at positive b* values) a more negative b value showing higher light emission in the blue region (420-470nm) which is a function of optical brightening agents as they absorb light in the ultraviolet/violet region (340-370nm) and re-emit in the blue.

Test products:

[0075]

LAS/AE1S Agglomerate

Constituent	%w/w Base Powder
LAS Linear alkyl benzene sulfonate	50
C12-14 Alky Ethoxylate (1) Sulphate	20
Sodium Sulphate	7.5
Silica	18.75
Free water	3.75
Total	100

Liquid Additive Mix

Constituent	%w/w
Ethoxylated Polyethyleneimine (PEI600E020 - 80%)	28
PEG-Vinyl Acetate co-polymer (72.5%)	38.4
Nonionic surfactant (C24AE7)	33.6
Total	100

Test Base - Low pH

Constituent	%w/w Base Powder
LAS/AE1S Agglomerate	67.04
Carboxymethyl cellulose (98%) (Finnfix GDA ex CP Kelco)	1.77
Texcare SRA300 Soil release polymer	0.54
Na HEDP Etidronic Acid (86.8%)	14.25
Zeolite	2.20
Acusol 4445N Polymer (92.6%)	4.08
Dow Corning GP-4314 Powdered Antifoam(12% active)	2.34
Lipase (18.5mg/g)	1.99
Stainzyme Plus (14.4mg/g)	1.15
Protease	1.74
Cellulase (15.6mg/g)	1.41
Mannanase (4mg/g)	1.52
Total	100.0

Reference Base - High pH

Constituent	%w/w Base Powder
LAS/AE1S Agglomerate	51.84
Carboxymethyl cellulose (98%) (Finnfix GDA ex CP Kelco)	1.37
Texcare SRA300 Soil release polymer	0.42
Na HEDP Etidronic Acid (86.8%)	11.0
Zeolite	1.70
Acusol 4445N Polymer (92.6%)	3.15
Dow Corning GP-4314 Powdered Antifoam(12% active)	1.81
Lipase (18.5mg/g)	1.54
Stainzyme Plus (14.4mg/g)	0.89
Protease	1.34
Cellulase (15.6mg/g)	1.08
Mannanase (4mg/g)	1.17
Sodium Carbonate	22.93
Total	100

Products Tested

[0076]

- High pH Reference: 13.74g of High pH reference base powder formulation & 0.11g of Brightener 49 Tinopal CBS contained in a PVA film* pouch & 1.25ml of liquid additive mix formulation.
- Low pH Test formulation (according to the invention): 10.62g Low pH test base formulation & 0.11g of Brightener 49 Tinopal CBS contained in a PVA film* & 1.25ml liquid additive mix formulation.

[0077] * PVA Film: M9400 provided by Monosol LLC, 707 East 80th Place, Suite 301, Merrillville, IN 4641

Results

10

15

20

25

30

35

40

45

50

[0078] As can be seen from the b-values tabulated below, brightener inclusion in the low pH test formulation according to the invention provides a more negative average b value across 4 fabrics tested vs brightener inclusion in high pH reference formulation outside the scope of the invention. This more negative b value results in brighter fabrics.

b * Value Low pH vs High pH with Brightener				
Fabric Type	High pH Ref	Low pH Test		
Terry Towel	-15.16	-15.49		
Polycotton	-14.69	-15.82		
Polyester	-14.6	-14.17		
Knitted Cotton	-13.24	-13.73		
Average	-14.42	-14.80		

Claims

- A water-soluble unit dose article comprising a water-soluble film and a solid laundry detergent composition, wherein
 the water-soluble film defines a first internal compartment and wherein the solid laundry detergent composition is
 comprised within the first internal compartment, and wherein the water-soluble unit dose article composition comprises a whitening agent preferably selected from a hueing dye and an optical brightener preferably an optical
 brightener; and
 - wherein the solid composition at 1wt% dilution in deionized water at 20°C, has an equilibrium pH in the range of from 6.5 to 8.8, preferably between 6.7 and 8.5.
- 2. The water-soluble unit dose article according to claim 1, wherein the solid laundry detergent composition comprises from 0wt% to 10wt% of carbonate salt, preferably wherein the carbonate salts are selected from sodium carbonate, potassium carbonate, sodium bicarbonate, sodium bicarbonate, burkeite, sequicarbonate habit modified carbonate, crystal growth modified burkeite or a mixture thereof, preferably sodium carbonate.
- 3. The water-soluble unit dose article according to any preceding claims wherein the solid laundry detergent composition comprises the whitening agent and wherein preferably the solid laundry detergent composition comprises between 0.05% and 2%, preferably between 0.1% and 1.5%, even more preferably between 0.2% and 1% by weight of the solid laundry detergent composition of the whitening agent.
- **4.** The water-soluble unit dose article according to any preceding claims, wherein the optical brightener is selected from hydrophobic brighteners, stilbene brighteners, biphenyl brighteners or a mixture thereof, preferably selected from brightener 49, brightener 15, brightener 36 or a mixture thereof, most preferably brightener 49.
- 5. The water-soluble unit dose article according to any preceding claims wherein the solid laundry detergent composition comprises a non-soap anionic surfactant and wherein preferably the non-soap anionic surfactant comprises linear alkylbenzene sulphonate, alkoxylated alkyl sulphate or a mixture thereof, more preferably a mixture thereof wherein the ratio of linear alkylbenzene sulphonate to alkoxylated alkyl sulphate preferably the ratio of linear alkylbenzene sulphonate to ethoxylated alkyl sulphate is from 1:2 to 20:1, preferably from 1.1:1 to 15:1, more preferably from 1.2:1 to 10:1, even more preferably from 1.3:1 to 5:1, even more preferably from 1.4:1 to 3:1, most preferably from 2:1 to 3:1.
- 6. The water-soluble unit dose article according to claim 5 wherein the solid laundry detergent composition comprises between 20% and 75%, preferably between 30% and 70%, more preferably between 40% and 60% by weight of the solid laundry detergent composition of the non-soap anionic surfactant.

7. The water-soluble unit dose article according to any preceding claims wherein the solid laundry detergent composition comprises between 1% and 10% by weight of the solid laundry detergent composition of an organic acid and wherein preferably the organic acid is a carboxylic acid, preferably a polycarboxylic acid, more preferably the organic acid is selected from citric acid, malic acid, lactic acid, propionic acid, valeric acid, caproic acid, carbonic acid, adipic acid, gluconic acid, methylglycinediacetic acid or a mixture thereof, most preferably citric acid.

5

10

15

20

30

45

50

55

- **8.** The water-soluble unit dose article according to any preceding claims wherein the solid laundry detergent composition is a free flowing particulate solid, a compressed particulate solid or a mixture thereof and has a mean particle size between 400 microns and 1000 microns, preferably between 450 microns and 850 microns.
- 9. The water-soluble unit dose article according to any preceding claims wherein the solid laundry detergent composition has a bulk density of between 400 and 1000g/l preferably between 500 and 800g/l.
- **10.** The water-soluble unit dose article according to any preceding claims wherein the first compartment comprises between between 1g and 25g, preferably between 5g and 20 g, preferably between 8g and 18g of the solid laundry composition.
 - 11. The water-soluble unit dose article according to any preceding claims wherein the water-soluble film comprises polyvinyl alcohol, preferably wherein the water-soluble film comprises polyvinyl alcohol polymer or copolymer, preferably a blend of polyvinylalcohol polymers and/or polyvinylalcohol copolymers, more preferably selected from sulphonated and carboxylated anionic polyvinylalcohol copolymers especially carboxylated anionic polyvinylalcohol copolymers, most preferably a blend of a polyvinylalcohol homopolymer and a carboxylated anionic polyvinylalcohol copolymer.
- 12. The water-soluble unit dose article according to any preceding claims wherein the water-soluble unit dose article comprises at least a first compartment and a second compartment, preferably at least a first compartment, a second compartment and a third compartment.
 - 13. The water-soluble unit dose article according to claim 12 wherein the compartments are arranged in a side-by-side arrangement, a superposed arrangement or a mixture thereof, preferably wherein at least one compartment, preferably wherein at least two compartments are superposed onto a third compartment, even more preferably wherein at least one of the superposed compartments comprises a liquid, preferably wherein the both superposed compartments comprise a liquid.
- 14. A method of washing comprising the steps of adding the water-soluble unit dose article according to any preceding claims to sufficient water to dilute the solid laundry detergent composition by a factor of at least 300 fold to create a wash liquor and contacting fabrics to be washed with said wash liquor.
- **15.** Use of a solid laundry detergent composition comprising a non-soap surfactant and having at 1wt% dilution in deionized water at 20°C an equilibrium pH in the range of from 6.5 to 8.8 in a water-soluble unit dose article to improve the performance of an optical brightener.

11



EUROPEAN SEARCH REPORT

Application Number EP 17 17 6195

	DOCUMENTS CONSIDE	RED TO BE RELEVANT				
Category	Citation of document with indi of relevant passag		Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)		
X	US 2015/275157 A1 (S [GB] ET AL) 1 Octobe * paragraphs [1159] [0108] *	r 2015 (2015-10-01)	1,2,8-14	INV. C11D3/00 C11D3/42 C11D17/04		
Х	US 2009/011970 A1 (E THEOPHILE [BE]) 8 January 2009 (2009 * claims; examples *		1-4,7-14			
Х	US 2003/087784 A1 (S PATRIC [GB] ET AL) 8 * claims; examples *	OMERVILE-ROBERTS NIGEL May 2003 (2003-05-08)	1-5,7-14			
Х	WO 01/83667 A1 (PROC 8 November 2001 (200 * claims; examples *		1-14			
				TECHNICAL FIELDS		
				SEARCHED (IPC)		
				C11D		
	The present search report has be					
Place of search Munich		Date of completion of the search 4 December 2017	Pfa	nnenstein, Heide		
C	ATEGORY OF CITED DOCUMENTS	T : theory or principle				
	icularly relevant if taken alone	E : earlier patent doc after the filing date	ument, but publis			
Y : particularly relevant if combined with another D : document cited in the application document of the same category L : document cited for other reasons A : technological background		D : document cited in	the application			
		& : member of the sa	&: member of the same patent family, corresponding			
		· -				

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 17 17 6195

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

04-12-2017

Patent document cited in search report		Publication date		Patent family member(s)	Publication date
US 2015275157	A1	01-10-2015	CA CA CN CN EP EP US WO WO	2940222 A1 2940420 A1 2940425 A1 106133125 A 106133127 A 106164234 A 2924106 A1 2924107 A1 2924108 A1 2015275155 A1 2015275156 A1 2015275157 A1 2015148777 A1 2015148780 A1 2015148782 A1	01-10-2015 01-10-2015 01-10-2015 16-11-2016 16-11-2016 23-11-2016 30-09-2015 30-09-2015 30-09-2015 01-10-2015 01-10-2015 01-10-2015 01-10-2015 01-10-2015
US 2009011970	A1	08-01-2009	AT EP ES US	503826 T 2014756 A1 2364193 T3 2009011970 A1	15-04-2011 14-01-2009 26-08-2011 08-01-2009
US 2003087784	A1	08-05-2003	NON	E	
WO 0183667	A1	08-11-2001	AU BR CA CN EP GB JP MX WO	4356201 A 0110271 A 2404689 A1 1426459 A 1283862 A1 2361688 A 2003531953 A PA02010585 A 0183667 A1	12-11-2001 18-02-2003 08-11-2001 25-06-2003 19-02-2003 31-10-2001 28-10-2003 10-03-2003 08-11-2001

© Lorentz Control Cont