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

(11) EP 3 415 592 A1

(12) # EUROPEAN PATENT APPLICATION

(43) Date of publication:
19.12.2018 Bulletin 2018/51

(21) Application number: **17176226.3**

(22) Date of filing: **15.06.2017**

(51) Int Cl.:
C11D 3/00 *(2006.01)* ***C11D 3/386*** *(2006.01)*
C11D 17/04 *(2006.01)*

(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

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(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 and comprising an enzyme, the composition has a pH of 6,5-8,8 in diluted form

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Description

FIELD OF THE INVENTION

[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 enzyme.

[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 stain removal performance of enzymes formulated into 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 an enzyme; 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 in a water-soluble unit dose article to improve the performance of an enzyme.

DETAILED DESCRIPTION OF THE INVENTION

<u>Water-soluble unit dose article</u>

[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.

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

[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 composition. 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.

[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 an enzyme. Preferably, the solid laundry detergent composition comprises an enzyme, preferably between 0.001% and 1%, more preferably between 0.02% and 0.5%, even more preferably between 0.003% and 0.1% by weight of the solid laundry detergent composition of the enzyme.

[0022] Preferably, the enzyme is selected from the group comprising hemicellulases, peroxidases, proteases, cellulases, xylanases, lipases, phospholipases, esterases, cutinases, pectinases, keratanases, reductases, oxidases, phenoloxidases, lipoxygenases, ligninases, pullulanases, tannases, pentosanases, malanases, ß-glucanases, arabinosidases, hyaluronidase, chondroitinase, laccase, and amylases, or mixtures thereof.

[0023] A typical combination is a cocktail of conventional applicable enzymes like protease, lipase, cutinase and/or cellulase in conjunction with amylase.

[0024] A typical combination is an enzyme cocktail that may comprise, for example, a protease and lipase in conjunction with amylase. Preferably the composition comprises a lipase.

[0025] Preferred enzymes could include a protease. Suitable proteases include metalloproteases and serine proteases, including neutral or alkaline microbial serine proteases, such as subtilisins (EC 3.4.21.62). In one aspect, such suitable protease may be of microbial origin. The suitable proteases include chemically or genetically modified mutants of the aforementioned suitable proteases. In one aspect, the suitable protease may be a serine protease, such as an alkaline microbial protease or/and a trypsin-type protease. Examples of suitable neutral or alkaline proteases include:

(a) subtilisins (EC 3.4.21.62), including those derived from Bacillus, such as Bacillus lentus, B. alkalophilus, B. subtilis, B. amyloliquefaciens, Bacillus pumilus and Bacillus gibsonii.

(b) trypsin-type or chymotrypsin-type proteases, such as trypsin (*e.g.*, of porcine or bovine origin), including Fusarium protease and chymotrypsin proteases derived from Cellumonas.

(c) metalloproteases, including those derived from Bacillus amyloliquefaciens.
(d) subtilisin proteases derived from the Bacillus sp TY-145 , NCIMB 40339, especially the variants described with substitutions and/or deletions at positions 171, 173, 175 or 179.

[0026] Preferred proteases include those derived from Bacillus gibsonii, Bacillus amyloliquefaciens, Bacillus sp. TY-145 or Bacillus Lentus.

[0027] Suitable commercially available protease enzymes include those sold under the trade names Alcalase®, Savinase®, Primase®, Durazym®, Polarzyme®, Kannase®, Liquanase®, Liquanase Ultra®, Savinase Ultra®, Ovozyme®, Neutrase®, Blaze®, Everlase® and Esperase® by Novozymes A/S (Denmark), those sold under the tradename Maxatase®, Maxacal®, Maxapem®, Properase®, Purafect®, Purafect Prime®, Purafect Ox®, FN3®, FN4®, Excellase®, Ultimase®, Purafect OXP® and the Preferenz P® series by DuPont International Biosciences, those sold under the tradename Opticlean® and Optimase® by Solvay Enzymes, those available from BASF, namely BLAP (sequence shown in Figure 29 of US 5,352,604 with the following mutations S99D + S101 R + S103A + V104I + G159S, hereinafter referred to as BLAP), BLAP R (BLAP with S3T + V4I + V199M + V205I + L217D), BLAP X (BLAP with S3T + V4I + V205I) and BLAP F49 (BLAP with S3T + V4I + A194P + V199M + V205I + L217D) - all from BASF; and KAP (Bacillus alkalophilus subtilisin with mutations A230V + S256G + S259N) from Kao.

[0028] Suitable alpha-amylases include those of bacterial or fungal origin. Chemically or genetically modified mutants (variants) are included. A preferred alkaline alpha-amylase is derived from a strain of Bacillus, such as Bacillus licheniformis, Bacillus amyloliquefaciens, Bacillus stearothermophilus, Bacillus subtilis, or other Bacillus sp., such as Bacillus sp. NCIB 12289, NCIB 12512, NCIB 12513, DSM 9375, DSM 12368, DSMZ no. 12649, KSM AP1378, KSM K36 or KSM K38. Preferred amylases include:

(a) the variants described in WO 94/02597, WO 94/18314, WO96/23874 and WO 97/43424, especially the variants with substitutions in one or more of the following positions versus the enzyme listed as SEQ ID No. 2 in WO 96/23874: 15, 23, 105, 106, 124, 128, 133, 154, 156, 181 , 188, 190, 197, 202, 208, 209, 243, 264, 304, 305, 391, 408, and 444.
(b) the variants described in USP 5,856,164 and WO99/23211, WO 96/23873, WO00/60060 and WO 06/002643, especially the variants with one or more substitutions in the following positions versus the AA560 enzyme listed as SEQ ID No. 12 in WO 06/002643:

26, 30, 33, 82, 37, 106, 118, 128, 133, 149, 150, 160, 178, 182, 186, 193, 203, 214, 231, 256, 257, 258, 269, 270, 272, 283, 295, 296, 298, 299, 303, 304, 305, 311, 314, 315, 318, 319, 339, 345, 361, 378, 383, 419, 421, 437, 441, 444, 445, 446, 447, 450, 461, 471, 482, 484, preferably that also contain the deletions of D183* and G184*.

(c) variants exhibiting at least 85%, preferably 90% identity with SEQ ID No. 4 in WO06/002643, the wild-type enzyme from Bacillus SP722, especially variants with deletions in the 183 and 184 positions and variants described in WO 00/60060, WO11/100410 and WO13/003659, particularly those with one or more substitutions at the following positions versus SEQ ID No. 4 in WO06/002643:

51, 52, 54, 109, 304, 140, 189, 134, 195, 206, 243, 260, 262, 284, 347, 439, 469, 476 and 477.

(d) variants exhibiting at least 95% identity with the wild-type enzyme from Bacillus sp.707 (SEQ ID NO:7 in US 6,093, 562), especially those comprising one or more of the following mutations M202, M208, S255, R172, and/or M261. Preferably said amylase comprises one or more of M202L, M202V, M202S, M202T, M202I, M202Q, M202W, S255N and/or R172Q. Particularly preferred are those comprising the M202L or M202T mutations.
(e) variants described in WO 09/149130, preferably those exhibiting at least 90% identity with SEQ ID NO: 1 or SEQ ID NO:2 in WO 09/149130, the wild-type enzyme from Geobacillus Stearophermophilus or a truncated version thereof.
(f) variants described in WO10/115021, especially those exhibiting at least 75%, or at least 85% or at least 90% or at least 95% with SEQ ID NO:2 in WO10/115021, the alpha-amylase derived from Bacillus sp. TS-23.

[0029] Suitable commercially available alpha-amylases include DURAMYL®, LIQUEZYME®, TERMAMYL®, TERMAMYL ULTRA®, NATALASE®, SUPRAMYL®, STAINZYME®, STAINZYME PLUS®, EVEREST®, FUNGAMYL® and BAN® (Novozymes A/S, Bagsvaerd, Denmark), KEMZYM® AT 9000 Biozym Biotech Trading GmbH Wehlistrasse 27b A-1200 Wien Austria, RAPIDASE®, PURASTAR®, ENZYSIZE®, OPTISIZE HT PLUS®, POWERASE® and PURASTAR OXAM®, PREFERENZ® S series, including PREFERENZ S1000 and PREFERENZ S110 (DuPont Industrial Biosciences, Palo Alto, California) and KAM® (Kao, 14-10 Nihonbashi Kayabacho, 1-chome, Chuo-ku Tokyo 103-8210, Japan). In one aspect, suitable amylases include NATALASE®, EVEREST®, PREFERENZ S1000®, STAINZYME® and

STAINZYME PLUS® and mixtures thereof.

[0030] In one aspect, such enzymes may be selected from the group consisting of: lipases, including "first cycle lipases". In one aspect, the lipase is a first-wash lipase, preferably a variant of the wild-type lipase from Thermomyces lanuginosus comprising one or more of the T231R and N233R mutations. The wild-type sequence is the 269 amino acids (amino acids 23 - 291) of the Swissprot accession number Swiss-Prot 059952 (derived from Thermomyces lanuginosus (Humicola lanuginosa)). Preferred first cycle lipases include those with mutations at one or more of the following positions: 27, 38, 58, 60, 83, 96, 111, 150, 163, 227, 231, 233, 254, 255 and 256. Further preferred lipase variants have a net charge that is more positive than the wild-type lipase. The net charge can be calculated by assigning a charge of -1 to the anionic groups (D and E) and a net charge of +1 to the cationic groups (R and K) and comparing to the wild-type. Preferred mutations include D27R, G38A, G91A, D96G, D96E, D111A, G163K, G225R, T231R, N233R, D254S, P256T, S58A, V60S, S83T, A150G, L227G, I255A and/or P256K. Preferred variants include those with mutations:

(a) T231R + N233R;
(b) D27R + G38A + D96E + D111A + G163K + T231R + N233R + D254S + P256T
(c) G91A + D96G + G225R + T231R + N233R

[0031] Preferred lipases would include those sold under the tradenames Lipex®, Lipoclean®, Calipso® and Lipolex®.

[0032] In one aspect, other preferred enzymes include fungal and microbial-derived endoglucanases exhibiting endo-beta-1,4-glucanase activity (E.C. 3.2.1.4), including a bacterial polypeptide endogenous to a member of the genus Bacillus which has a sequence of at least 90%, 94%, 97% and even 99% identity to the amino acid sequence SEQ ID NO:2 in US7,141,403B2) and mixtures thereof. Suitable endoglucanases are sold under the tradenames Celluclean®, Carezyme®, Celluzyme®, Carezyme Premium® and Whitezyme® (Novozymes A/S, Bagsvaerd, Denmark).

[0033] Other preferred enzymes include pectate lyases sold under the tradenames Pectawash®, Pectaway®, Xpect® and mannanases sold under the tradenames Mannaway® (all from Novozymes A/S, Bagsvaerd, Denmark), and Preferenz F® and Purabrite® (DuPont International Biosciences, Palo Alto, California).

[0034] Deoxyribonuclease (DNase): Suitable deoxyribonucleases (DNases) are any enzyme that catalyzes the hydrolytic cleavage of phosphodiester linkages in the DNA backbone, thus degrading DNA. According to the invention, a DNase which is obtainable from a bacterium is preferred; in particular a DNase which is obtainable from a Bacillus is preferred; in particular a DNase which is obtainable from Bacillus subtilis or Bacillus licheniformis is preferred.

[0035] Perhydrolases: Suitable perhydrolases are capable of catalyzing a perhydrolysis reaction that results in the production of a peracid from a carboxylic acid ester (acyl) substrate in the presence of a source of peroxygen (e.g., hydrogen peroxide). While many enzymes perform this reaction at low levels, perhydrolases exhibit a high perhydrolysis:hydrolysis ratio, often greater than 1. Suitable perhydrolases may be of plant, bacterial or fungal origin. Chemically modified or protein engineered mutants are included.

[0036] Examples of useful perhydrolases include naturally occurring Mycobacterium perhydrolase enzymes, or variants thereof. An exemplary enzyme is derived from Mycobacterium smegmatis.

[0037] Oxidases/peroxidases: Suitable oxidases and peroxidases (or oxidoreductases) include various sugar oxidases, laccases, peroxidases and haloperoxidases.

[0038] Suitable peroxidases include those comprised by the enzyme classification EC 1.1 1 .1.7, as set out by the Nomenclature Committee of the International Union of Biochemistry and Molecular Biology (IUBMB), or any fragment derived therefrom, exhibiting peroxidase activity.

[0039] Suitable peroxidases include those of plant, bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Examples of useful peroxidases include peroxidases from Coprinopsis, e.g., from C. cinerea and variants thereof.

[0040] A peroxidase for use in the invention also include a haloperoxidase enzyme, such as chloroperoxidase, bromoperoxidase and compounds exhibiting chloroperoxidase or bromoperoxidase activity. Haloperoxidases are classified according to their specificity for halide ions. Chloroperoxidases (E.C. 1 .1 1.1 .10) catalyze formation of hypochlorite from chloride ions.

[0041] The haloperoxidase may be a chloroperoxidase. Preferably, the haloperoxidase is a vanadium haloperoxidase, i.e., a vanadate-containing haloperoxidase. In a preferred method of the present invention the vanadate-containing haloperoxidase is combined with a source of chloride ion.

[0042] Haloperoxidases have been isolated from many different fungi, in particular from the fungus group dematiaceous hyphomycetes, such as Caldariomyces, e.g., C. fumago, Alternaria, Curvularia, e.g., C. verruculosa and C. inaequalis, Drechslera, Ulocladium and Botrytis. Haloperoxidases have also been isolated from bacteria such as Pseudomonas, e.g., P. pyrrocinia and Streptomyces, e.g., S. aureofaciens.

[0043] Preferably, the haloperoxidase is derivable from Curvularia sp., in particular Curvularia verruculosa or Curvularia inaequalis, such as C. inaequalis CBS 102.42 6; or C. verruculosa CBS 147.63 or C. verruculosa CBS 444.70; or from Drechslera hartlebii, Dendryphiella salina, Phaeotrichoconis crotalarie , or Geniculosporium sp.

[0044] An oxidase according to the invention include, in particular, any laccase enzyme comprised by the enzyme classification EC 1 .10.3.2, or any fragment derived therefrom exhibiting laccase activity, or a compound exhibiting a similar activity, such as a catechol oxidase (EC 1.10.3.1), an o-aminophenol oxidase (EC 1.10.3.4), or a bilirubin oxidase (EC 1 .3.3.5).

[0045] Preferred laccase enzymes are enzymes of microbial origin. The enzymes may be derived from plants, bacteria or fungi (including filamentous fungi and yeasts).

[0046] Suitable examples from fungi include a laccase derivable from a strain of Aspergillus, Neurospora, e.g., N. crassa, Podospora, Botrytis, Collybia, Fomes, Lentinus, Pleurotus, Trametes, e.g., T. villosa and T. versicolor, Rhizoctonia, e.g., R. solani, Coprinopsis, e.g., C. cinerea, C. comatus, C. friesii, and C. plicatilis, Psathyrella, e.g., P. condelleana, Panaeolus, e.g., P. papilionaceus, Myceliophthora, e.g., M. thermophila, Schytalidium, e.g., S. thermophilum, Polyporus, e.g., P. pinsitus, Phlebia, e.g., P. radiata or Coriolus, e.g., C. irsutus).

[0047] Suitable examples from bacteria include a laccase derivable from a strain of Bacillus. A laccase derived from Coprinopsis or Myceliophthora is preferred; in particular a laccase derived from Coprinopsis cinerea,; or from Myceliophthora thermophila.

[0048] Examples of other oxidases include, but are not limited to, amino acid oxidase, glucose oxidase, lactate oxidase, galactose oxidase, polyol oxidase and aldose oxidase. Oxidases and their corresponding substrates may be used as hydrogen peroxide generating enzyme systems, and thus a source of hydrogen peroxide. Several enzymes, such as peroxidases, haloperoxidases and perhydrolases, require a source of hydrogen peroxide. By studying EC 1.1 .3._, EC 1.2.3._, EC 1.4.3._, and EC 1.5.3._ or similar classes (under the International Union of Biochemistry), other examples of such combinations of oxidases and substrates are easily recognized by one skilled in the art.

Water-soluble film

[0049] 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.

[0050] 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 grams ± 0.1 gram of film material is added in a pre-weighed 3L beaker and 2L ± 5ml 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.

[0051] 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.

[0052] 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.

[0053] 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.

[0054] 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.

[0055] Preferred films are those supplied by Monosol under the trade references M8630, M8900, M8779, M8310.
[0056] The film may be opaque, transparent or translucent. The film may comprise a printed area.
[0057] The area of print may be achieved using standard techniques, such as flexographic printing or inkjet printing.
[0058] 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.

Solid laundry detergent composition

[0059] 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.
[0060] 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.
[0061] 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.
[0062] 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.
[0063] 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.
[0064] 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.
[0065] 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.
[0066] The enzyme may be added in a liquid form or a solid form. When added in a liquid form, the enzyme raw material may either contain or be blended with stabilizers including glycols, such as monopropylene glycol, calcium salts, sorbitol, reversible protease inhibitors such as boron derivatives, particularly phenyl boronic acid derivatives such as 4-formyl phenyl boronic acid, and/or peptide inhibitorsm, particularly peptide aldehydes.
[0067] The enzyme may also be added in a solid form or as a capsule. Solid forms would include granules that can be made by fluid bed coating such as layered granules. Preferably said microcapsules and granules are coated with a polymer that provides triggered release via an ionic strength trigger such that said granule and/or capsule is stable in product but upon dilution in water will release its enzyme payload. Examples of such polymeric coatings include cellulose derivatives, such as hydroxypropyl methyl cellulose derivatives, particularly hydroxyl propyl methyl cellulose phthalate and cellulose acetate phthalate. A further preferred polymeric coating is polyvinyl alcohol. It is further preferred that any capsules and/or granules are density-matched to the surrounding liquid matrix to promote stability and prevent settling out of a visible phase. In a further aspect the enzymes can be added as capsules and/or microcapsules derived from interfacial polymerization reaction of a polyamine, preferably a branched polyamine. Said microcapsules can be made by recation of polyamines, such as those sold under the Lupasol tradename by BASF with an acid chloride.
[0068] 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
[0069] 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.
[0070] 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.

[0071] 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 1 to 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.

[0072] 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 a degree of ethoxylation of between 0.5 and 10, preferably between 1 and 9, more preferably between 3 and 8, most 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.

[0073] 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.

[0074] 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 ± 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 ±0.01pH units, with stirring, ensuring temperature is 20°C +/- 0.5°C.

[0075] 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.

[0076] 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.

[0077] 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.

[0078] The solid laundry detergent composition may comprise an adjunct ingredient, wherein the adjunct ingredient is preferably selected from cationic polymers, brighteners, dye transfer inhibitors, chelants including aminocarboxylate and aminophosphonate chelants such as HEDP, 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.

[0079] Preferred acrylate-based polymers are acrylate/maleate random copolymers.

[0080] 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.

[0081] Preferably, the particulate laundry detergent composition comprises agglomerates. Preferably, the agglomerates comprise non-soap surfactant, sodium sulphate and silica.

[0082] 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 enzyme, especially a lipase enzyme.

Method of washing

[0083] 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.
[0084] The method may be performed in a hand wash operation, an automatic laundry washing machine or a mixture thereof.

Process of making

[0085] 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 :

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 :

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.

[0086] 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.
[0087] 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.
[0088] 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.
[0089] 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.
[0090] 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

[0091] The below stain removal test shows a consistently improved performance profile for low versus high pH enzyme comprising powder compositions within a water soluble pouch.

Test Method:

[0092] In order to demonstrate the impact in providing improved cleaning performance of formulating a low pH powder vs a high pH powder contained within a PVA film pouch, a stain removal 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 3622), 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 (4 SBL2004 soiled ballast sheets ex wfk Testgewebe GmbH Christenfeld 10, D-41379 Brüggen-Bracht Germany). Stain sets (sourced from Warwick Equest Ltd. Unit 55, Consett Business Park, Consett, County Durham, DH8 6BN) were added to each machine and washed in either high pH reference product or low pH 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, stain sets were removed and tumble dried in an electric Miele tumble dryer (Novotronic T430) set to "extra dry". Stains were then analysed using a Stain Removal Index (SRI), the higher the SRI value the better the stain removal performance is. ΔSRI denoted the difference in SRI between the Reference and Test products.

Test products:

LAS/AE1S Agglomerate

[0093]

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

[0094]

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

[0095]

Constituent	%w/w Base Powder
LAS/AE1S Agglomerate	71.81

(continued)

Constituent	%w/w Base Powder
Carboxymethyl cellulose (98%) (Finnfix GDA ex CP Kelco)	1.90
Brightener 49 Tinopal® CBS-X	1.25
Texcare SRA300 Soil release polymer	0.57
Na HEDP Editronic Acid (86.8%)	15.27
Acusol 4445N Polymer (92.6%)	4.36
Zeolite	2.35
Dow Corning GP-4314 Powdered Antifoam (12% active)	2.50
Total	100

Reference Base - High pH

[0096]

Constituent	%w/w Base Powder
LAS/AE1S Agglomerate	55.53
Carboxymethyl cellulose (98%) (Finnfix GDA ex CP Kelco)	1.46
Brightener 49 Tinopal® CBS-X	0.96
Texcare SRA300 Soil release polymer	0.44
NaHEDP Etidronic acid (86.6%)	11.80
Zeolite	1.82
Acusol 4445N polymer (92.6%	3.37
Dow Corning GP-4314 Powdered Antifoam (12% active)	1.94
Lipase (18.5mg/g)	1.72
Stainzyme Plus (14.4mg/g)	0.99
Protease	1.49
Cellulase (15.6mg/g)	1.20
Mannanase (4mg/g)	1.30
Sodium Carbonate	24.56
Total	100

Enzyme cocktail:

[0097]

Constituent	%w/w Base Powder
Lipase (18.5mg/g)	25.54
Stainzyme Plus (14.4mg/g)	14.82
Protease	22.29
Cellulase (15.6mg/g)	17.95
Mannanase (4mg/g)	19.40

(continued)

Constituent	%w/w Base Powder
Total	100

Products Tested

[0098]

- High pH Ref A : 13.07 g of High pH Ref base formulation contained in a PVA film* pouch & 1.25ml of liquid additive mix.
- Low pH Test leg A : 9.92 g of Low pH Test base formulation contained in a PVA film* pouch & 1.25ml of liquid additive mix.
- High pH Ref B : 13.07 g of High pH Ref base formulation and 0.83g of enzyme cocktail contained in a PVA film* pouch & 1.25ml of liquid additive mix.
- Low pH Test leg B : 9.92 g of Low pH Test base formulation and 0.83g of enzyme cocktail contained in a PVA film* pouch & 1.25ml of liquid additive mix.

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

Results

[0099] The results tabulated below show that there is improved stain removal of greasy stains in a low versus a high pH formulation, the performance delta getting magnified in presence versus in absence of enzyme technology especially lipase technology.

Soil	High pH Ref without enzyme	Low pH Test without enzyme	High pH Ref with enzyme	Low pH Test with enzyme
Burnt butter	38.3	46.7	50.0	59.9
Cooked beef	18.9	25.6	31.8	41.8
Dyed Bacon	45.5	48.8	50.5	53.2
Make-up	21.8	28.9	24.1	33.9
Average	*31.1*	*37.5*	*39.1*	*47.2*

Claims

1. 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 an enzyme; 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 enzyme and wherein preferably the solid laundry detergent composition comprises between 0.001% and 1%, preferably between 0.02% and 0.5%, even more preferably between 0.003% and 0.1% by weight of the solid laundry detergent composition of the enzyme.

4. The water-soluble unit dose article according to any preceding claims, wherein the enzyme is selected from the

group comprising hemicellulases, peroxidases, proteases, cellulases, xylanases, lipases, phospholipases, esterases, cutinases, pectinases, keratanases, reductases, oxidases, phenoloxidases, lipoxygenases, ligninases, pullulanases, tannases, pentosanases, malanases, ß-glucanases, arabinosidases, hyaluronidase, chondroitinase, laccase, and amylases, or mixtures thereof, preferably proteases, amylases, lipases, cellulases and mixtures thereof, preferably lipases.

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.

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 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 enzyme.

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EUROPEAN SEARCH REPORT

Application Number
EP 17 17 6226

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X	DE 10 2014 218952 A1 (HENKEL AG & CO KGAA [DE]) 24 March 2016 (2016-03-24) * paragraphs [0121] - [0128]; examples * -----	1-14	TECHNICAL FIELDS SEARCHED (IPC)
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The present search report has been drawn up for all claims

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Place of search	Date of completion of the search	Examiner
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X : particularly relevant if taken alone
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