WATER-SOLUBLE UNIT-DOSE POUCH
COMPRISING CHELANT

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Appl. No.: 13/477,100
Filed: May 22, 2012

Foreign Application Priority Data
May 23, 2011 (EP) ..................................... 11 167 079.0

Publication Classification

<table>
<thead>
<tr>
<th>Int. Cl.</th>
<th>2006.01</th>
</tr>
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<tr>
<td>B65D 30/02</td>
<td>(2006.01)</td>
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<td>B65D 30/22</td>
<td>(2006.01)</td>
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<td>D06L 3/02</td>
<td>(2006.01)</td>
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The present invention contemplates a water-soluble unit-dose pouch, comprising at least a first compartment wherein the first compartment comprises a powder composition, and wherein the powder composition comprises an oxygen bleach source and between 2% and 20% by weight of the water-soluble unit-dose pouch of a salt of citric acid. It also contemplates methods of using the pouch in a fabric washing machine.
WATER-SOLUBLE UNIT-DOSE POUCH COMPRISING CHELANT

FIELD OF THE INVENTION

[0001] The present invention contemplates water-soluble unit-dose pouches comprising chelant, that exhibit reduced patchy colour damage on coloured fabrics during the wash.

BACKGROUND TO THE INVENTION

[0002] Unitised doses of detergents and bleaching compositions have been found to be both attractive and convenient to consumers. Indeed, a “unit-dose” is easy to handle and avoids the need of the consumer to measure the product, thereby giving rise to more precise dosing and avoiding wasteful overdosing or under-dosing. Often the unit-dose is in the form of a water-soluble pouch comprising a powder or liquid composition. This has the added benefit of allowing the user to handle the unit-dose without directly handling the cleaning composition contained within.

[0003] A preferred bleaching agent is an oxygen bleach. When used in conjunction with bleach activators, oxygen bleaches offer excellent cleaning. The oxygen bleach source and the bleach activator are powder ingredients. In the presence of water, the oxygen bleach source and the bleach activator react together to form free oxygen.

[0004] A number of bleach activators are known in the art. An exemplary bleach activator is tetraacetyl ethylene diamine (TAED). Often TAED is used in conjunction with an oxygen bleach source such as sodium percarbonate to remove tough beverage stains such as coffee, tea, red wine, black grape juice etc. However a problem associated with the combination of an oxygen bleach source and high levels of TAED is that it may cause patchy discoloration damage. Patchy discoloration damage is where the cleaning composition causes localized colour bleaching on the fabrics. This is then seen by the consumer as white patches on coloured fabrics. This is at least partly due to the slower dissolution and dispersion rates associated with water-soluble unit-dose pouches as compared to powder or liquid compositions independent of being in a water-soluble unit-dose pouch. This slower dissolution means that it is possible to have localized bleaching of the fabrics due to the unit-dose pouch being present on a particular area of the fabric. As it dissolves, there is a localized higher level of TAED in that one particular area of the fabrics, which results in more intense bleaching activity.

[0005] Therefore, there is a need in the art for a water-soluble unit-dose pouch that has effective stain removal on tough beverage stains, but shows reduced patchy discoloration damage on coloured fabrics.

[0006] It was surprisingly found that water-soluble unit-dose pouches that comprise a powder composition that comprises an oxygen bleach source and a salt of citric acid exhibited the same effective stain removal performance as the combination of an oxygen bleach source and high levels of TAED, but exhibited reduced patchy discoloration damage of fabrics.

SUMMARY OF THE INVENTION

[0007] A first aspect of the present invention is a water-soluble unit-dose pouch comprising at least a first compartment, wherein the first compartment comprises a powder composition, and wherein the powder composition comprises an oxygen bleach source and a salt of citric acid, and wherein the salt of citric acid is present between 2% and 20% by weight of the water-soluble unit-dose pouch.

[0008] A second aspect of the present invention is a method of treating fabrics comprising the steps of:

[0009] a) Placing fabrics in the drum of a fabric washing machine;

[0010] b) Adding a water-soluble unit-dose pouch according to the present invention to the drum of the fabric washing machine;


DETAILED DESCRIPTION

[0012] The Pouch

[0013] The water-soluble unit-dose pouch of the present invention (herein referred to as pouch), comprises at least a first compartment. The first compartment comprises a powder composition. The pouch is typically a closed structure, made of materials described herein, enclosing a volume space. Preferably, the volume space is separated into at least two compartments.

[0014] The pouch can be of any form, shape and material which is suitable to hold the composition, e.g. without allowing the release of the composition from the pouch prior to entry of the pouch with water. The exact execution will depend, for example, on the type and amount of the composition in the pouch, the number of compartments in the pouch, and on the characteristics required from the pouch to hold, protect and deliver or release the composition(s).

[0015] Preferably, the water-soluble unit-dose pouch comprises at least a first compartment and a second compartment. In one embodiment, the water-soluble unit-dose pouch comprises at least a first compartment and a second compartment, wherein the second compartment comprises a second composition. Preferably, the second composition is selected from the group comprising, liquid, gel, powder, granule or tablet. Most preferably, the second compartment comprises a liquid composition.

[0016] In another embodiment, the water-soluble unit-dose pouch comprises three compartments, wherein the first compartment comprises a powder composition and the second and third compartments comprise compositions selected from the group comprising liquid, gel, powder, granule or tablet. Preferably, the second and third compartments comprise liquid compositions. In a further embodiment, the water-soluble unit-dose pouch comprises four compartments, wherein the first compartment comprises a powder composition and the second, third and fourth compartments comprise compositions selected from the group comprising liquid, gel, powder, granule or tablet. Preferably, the second, third and fourth compartments comprise liquid compositions. It is advantageous to have multiple compartments in a single water-soluble unit-dose pouch. This allows the combination of incompatible components and components requiring dry or liquid environments.

[0017] The compartments of the pouches can be separate, but are preferably conjoined in any suitable manner. Most preferably the second and third or subsequent compartments are superimposed on the first compartment. In one embodiment, the third compartment may be superimposed on the second compartment, which is in turn superimposed on the first compartment in a sandwich configuration. Alternatively the second and third compartments are superimposed on the first compartment. However it is also envisaged that the first, second and third and subsequent compartments may be attached to one another in a side by side relationship. The
compartments may be packed in a string, each compartment being individually separable by a perforation line. Hence each compartment may be individually torn-off from the remainder of the string by the end-user, for example, so as to pre-treat or post-treat a fabric with a composition from a compartment.

[0018] In a preferred embodiment the present pouch comprises three compartments consisting of a large first compartment and two smaller compartments. The second and third smaller compartments are superimposed on the first large compartment. The size and geometry of the compartments are chosen such that this arrangement is achievable.

[0019] The pouch is preferably made of a film material wherein the film material is soluble or dispersible in water, and has a water-solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereafter using a glass-fiber filter with a pore size of about 20 microns.

[0020] 50 grams±0.1 gram of pouch material is added in a pre-heated 400 ml beaker and 245ml±1 ml of distilled water is added. This is stirred vigorously on a magnetic stirrer set at 600 rpm, for 30 minutes. 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.

[0021] Preferred pouch materials are polymeric materials, preferably polymers which are formed into a film or sheet. The pouch material can, for example, be obtained by casting, blow-moulding, extrusion or blown extrusion of the polymeric material, as known in the art.

[0022] 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, poly-carboxylic acids and salts, polyvinylalcohol or peptides, polyamides, polyacrylamide, copolymers of maleic/polyacrylic acids, polyacrylamides including starch and gelatin, natural gums such as xanthan and carrageen. More preferred polymers are selected from polycrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polyacrylates, and most preferably selected from polyvinyl alcohols, polyvinyl alcohol copolymers and hydroxypolypropyl methylcellulose (HPMC), and combinations thereof. 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.

[0023] Mixtures of polymers can also be used as the pouch material. This can be beneficial to control the mechanical and/or dissolution properties of the pouch or compartments of the pouch, depending on the application thereof and the required needs. Suitable mixtures include for example mixtures wherein one polymer has a higher water-solubility than another polymer, and/or one polymer has a higher mechanical strength than another polymer. Also suitable are mixtures of polymers having different weight average molecular weights, for example a mixture of PVA or a copolymer thereof of a weight average molecular weight of about 10,000-40,000, preferably around 20,000, and of PVA or copolymer thereof, with a weight average molecular weight of about 100,000 to 300,000, preferably around 150,000. Also suitable herein are polymer blend compositions, for example comprising hydrolytically degradable and water-soluble polymer blends such as polylactide and polyvinyl alcohol, obtained by mixing polylactide and polyvinyl alcohol, typically comprising about 1-35% by weight polylactide and about 65% to 99% by weight polyvinyl alcohol. Preferred for use herein are polymers which are from about 60% to about 98% hydrolysed, preferably about 80% to about 90% hydrolysed, to improve the dissolution characteristics of the material.

[0024] Naturally, different film material and/or films of different thickness may be employed in making the compartments of the present invention. A benefit in selecting different films is that the resulting compartments may exhibit different solubility or release characteristics.

[0025] Most preferred pouch materials are PVA films known under the trade reference Monosol MR630, as sold by Chris-Craft Industrial Products of Gary, Ind., US, and PVA films of corresponding solubility and deformability characteristics. Other films suitable for use herein include films known under the trade reference PT film or the K-series of films supplied by Aicello, or VTF-HP film supplied by Kuraray.

[0026] The pouch material herein can also comprise one or more additive ingredients. For example, it can be beneficial to add plasticisers, for example glycerol, ethylene glycol, diethylene glycol, propylene glycol, sorbitol and mixtures thereof. Other additives include functional detergent additives to be delivered to the wash water, for example organic polymeric dispersants, etc.

[0027] For reasons of deformability, pouches or pouch compartments containing a component which is liquid will preferably contain an air bubble having a volume of up to about 50%, preferably up to about 40%, more preferably up to about 30%, more preferably up to about 20%, more preferably up to about 10% of the volume space of said compartment.

[0028] The pouch of the present invention may be made using any suitable equipment and method. Single compartment pouches are made using vertical, but preferably horizontal form filling techniques commonly known in the art. The film is preferably dampened, more preferably heated to increase the malleability thereof. Even more preferably, the method also involves the use of a vacuum to draw the film into a suitable mould. The vacuum drawing the film into the mould can be applied for 0.2 to 5 seconds, preferably 0.3 to 3 or even more preferably 0.5 to 1.5 seconds, once the film is on the horizontal portion of the surface. This vacuum may preferably be such that it provides an under-pressure of between −100 mbar to −1000 mbar, or even from −200 mbar to −600 mbar.

[0029] The moulds, in which the pouches are made, can have any shape, length, width and depth, depending on the required dimensions of the pouches. The moulds can also vary in size and shape from one to another, if desirable. For example, it may be preferred that the volume of the final pouches is between 5 and 300 ml, or even 10 and 150 ml or even 20 and 100 ml and that the mould sizes are adjusted accordingly.

[0030] Heat can be applied to the film, in the process commonly known as thermoforming, by any means. For example the film may be heated directly by passing it under a heating element or through hot air, prior to feeding it onto the surface or once on the surface. Alternatively it may be heated indi-
rectly, for example by heating the surface or applying a hot item onto the film. Most preferably the film is heated using an infra red light. The film is preferably heated to a temperature of 50 to 120°C, or even 60 to 90°C. Alternatively, the film can be wetted by any means, for example directly by spraying a wetting agent (including water, solutions of the film material or plasticizers for the film material) onto the film, prior to feeding it onto the surface or once on the surface, or indirectly by wetting the surface or by applying a wet item onto the film.

[0031] Once a film has been heated/wetted, it is drawn into an appropriate mould, preferably using a vacuum. The filling of the moulded film can be done by any known method for filling (preferably moving) items. The most preferred method will depend on the product form and speed of filling required. Preferably the moulded film is filled by in-line filling techniques. The filled, open pouches are then closed, using a second film, by any suitable method. Preferably, this is also done while in horizontal position and in continuous, constant motion. Preferably the closing is done by continuously feeding a second film, preferably water-soluble film, and/or into the open pouches and then preferably sealing the first and second film together, typically in the area between the moulds and thus between the pouches.

[0032] Preferred methods of sealing include heat sealing, solvent welding, and solvent or wet sealing. It is preferred that only the area which is to form the seal, is treated with heat or solvent. The heat or solvent can be applied by any method, preferably on the closing material, preferably only on the areas which are to form the seal. If solvent or wet sealing or welding is used, it may be preferred that heat is also applied. Preferred wet or solvent sealing/welding methods include selectively applying solvent onto the area between the moulds, or on the closing material, for example, spraying or printing this onto these areas, and then applying pressure onto these areas, to form the seal. Sealing rolls and belts as described above (optionally also providing heat) can be used, for example.

[0033] The formed pouches can then be cut by a cutting device. Cutting can be done using any known method. It may be preferred that the cutting is also done in continuous manner, and preferably with constant speed and preferably while in horizontal position. The cutting device can, for example, be a sharp item or a hot item, whereby in the latter case, the hot item 'burns' through the film/sealing area.

[0034] In one embodiment of the present invention, the water-soluble unit-dose pouch comprises at least two compartments. The different compartments of a multi-compartment pouch may be made together in a side-by-side style and consecutive pouches are not cut. Alternatively, the compartments can be made separately. According to this process and preferred arrangement, the pouches are made according to the process comprising the steps of:

[0035] a) forming an first compartment (as described above);
[0036] b) forming a recess within some or all of the closed compartment formed in step (a), to generate a second moulded compartment superposed above the first compartment;
[0037] c) filling and closing the second compartments by means of a third film;
[0038] d) sealing said first, second and third films; and
[0039] e) cutting the films to produce a multi-compartment pouch.

[0040] Said recess formed in step b is preferably achieved by applying a vacuum to the compartment prepared in step a).

[0041] Alternatively the second, and optionally third, compartment(s) can be made in a separate step and then combined with the first compartment. A particularly preferred process comprises the steps of:

[0042] a) forming a first compartment, optionally using heat and/or vacuum, using a first film on a forming machine;
[0043] b) filling said first compartment with a first composition;
[0044] c) on a second forming machine, deforming a second film, optionally using heat and vacuum, to make a second and optionally third moulded compartment;
[0045] d) filling the second and optionally third compartments;
[0046] e) sealing the second and optionally third compartments;
[0047] f) placing the sealed second and optionally third compartments onto the first compartment;
[0048] g) sealing the first, second and optionally third compartments; and
[0049] h) cutting the films to produce a multi-compartment pouch

[0050] The first and second forming machines are selected based on their suitability to perform the above process. The first forming machine is preferably a horizontal forming machine. The second forming machine is preferably a rotary drum forming machine, preferably located above the first forming machine.

[0051] It will be understood moreover that by the use of appropriate feed stations, it is possible to manufacture multi-compartment pouches incorporating a number of different or distinctive compositions and/or different or distinctive liquid, gel or paste compositions.

[0052] Solid Composition

[0053] The water-soluble unit-dose pouch comprises at least a first compartment comprising a solid composition. The solid composition of the present invention comprises an oxygen bleach source, and a salt of citric acid.

[0054] The Oxygen Bleach Source

[0055] As an essential ingredient, the solid composition according to the present invention comprises an oxygen bleach source. Preferably said oxygen bleach is a peroxygen source, more preferably a hydrogen peroxide source.

[0056] In one embodiment, the peroxygen source is an inorganic perhydrate salt. Inorganic perhydrate salts are normally the alkali metal salts. Preferably, the inorganic perhydrate salt is selected from the group comprising perchlorate, percarbonate, perphosphates and persulfate salts. In another embodiment, the perhydrate salt is selected from perborate and percarbonate. In one embodiment, the perhydrate salt is percarbonate, preferably sodium percarbonate.

[0057] Sodium percarbonate has the formula corresponding to 2Na₅₂₃₇O₇.₃H₂O₂. To enhance storage stability, the sodium percarbonate can be coated, for example, with a further mixed salt of an alkali metal sulphate and/or carbonate. The weight ratio of the mixed salt coating material to the sodium percarbonate is from 1:2000 to 1:4, more preferably from 1:99 to 1:9, and most preferably from 1:49 to 1:19. Preferably, the mixed salt is of sodium sulphate and sodium carbonate which has the general formula Na₅₂₃₇O₇.₃Na₂CO₃ wherein n is from 0.1 to 3, preferably n is from 0.3 to 1.0 and most preferably n is from 0.2 to 0.5. In one embodiment, the sodium percarbonate is coated with Boric acid.
Other suitable oxygen bleach sources include per- sulphates, particularly potassium per- sulphate $K_2S_2O_8$ and sodium per- sulphate $Na_2S_2O_8$.

Commercially available carbonate/sulphate coated sodium per- carbonate may include a low level of a heavy metal sequester such as ethylenediaminetetraacetic acid (EDTA), 1-hydroxyethylidene 1,1-diphosphonic acid (HEDP) or an aminophosphonic acid, that is incorporated during the manufacturing process. These sequesterants are used, as decomposition of oxygen bleach source, for example peroxide, is strongly catalyzed by free heavy metals (mainly Fe, Cu, Co, Mn, Cr). Thus the presence of sequesterants decreases the level of free heavy metals present at equilibrium, and consequently reduces the peroxide decomposition.

Preferred heavy metal sequesterants for incorporation as described herein include the organic phosphonates and amino alkylene poly(alkylene phosphonates) such as the alkali metal ethane 1-hydroxy diposphonates, the nitrilo trimethylene phosphonates, the ethylene diamine tetra methylene phosphonates and the diethylen triamine penta methylene phosphonates.

In another embodiment, the oxygen bleach source comprises a peroxy carboxylic acid (hereafter referred to as peracid). Preferred peracids are those having general formula:

$$\text{CON}-(R)\text{-COOH}$$

wherein $R$ is selected from C1-4 alkyl and $n$ is an integer of from 1 to 5.

In a particularly preferred aspect of the present invention the peracid has the formula such that $R$ is CH$_2$ and $n$ is 5 i.e. phthaloyl amino-peroxy caproic acid or PAP. The peracid is preferably used as a substantially water-insoluble solid compound and is available from Solvay/Ausimont under the tradename Euroco®.

Typically, the compositions of the present invention comprise from 8.5% to 70%, preferably from 10% to 60% and more preferably from 15% to 55%, by weight of the water-soluble unit-dose pouch, of an oxygen bleach source.

Salt of Citric Acid

The solid composition of the present invention comprises between 2% and 20% by weight of the water-soluble unit-dose pouch of a salt of citric acid. Preferably, the salt of citric acid is present at a concentration of between 3% and 20%, more preferably between 5% and 20%, more preferably between 7.5% and 15%, most preferably between 10% and 13% by weight of the water-soluble unit-dose pouch.

The level of salt of citric acid present is measured as a concentration by weight of the entire pouch. This is because the invention requires a particular level of salt of citric acid per unit-dose in order to give the benefit. This then allows for the presence of other compositions, i.e. lower levels of powder composition due to the addition of a liquid composition for example.

By ‘salt of citric acid’ we herein mean any salt of citric acid. Preferred salts of citric acid are those selected from the group comprising sodium citrate, calcium citrate, potassium citrate, ammonium citrate, magnesium citrate and mixtures thereof. The term ‘sodium citrate’ refers to any of the sodium salts of citric acid. In one embodiment, the salt of citric acid is sodium citrate, preferably monosodium citrate, disodium citrate, trisodium citrate and mixtures thereof. In one embodiment, the salt of citric acid is potassium citrate, preferably tripotassium citrate. In another embodiment, the salt of citric acid is calcium citrate, preferably tribasic calcium citrate. In yet another embodiment, the salt of citric acid is magnesium citrate, preferably tribasic magnesium citrate.

Some chelating agents have a tendency to cause the powder phase of the water-soluble unit-dose pouch to become yellow. It was surprisingly found that the presence of sodium citrate provides chelating activity whilst minimising the unwanted side effect of yellowing of the powder composition.

Low Level of TAED

Preferably, the water-soluble unit-dose pouch of the present invention comprises less than 1% by weight of the water-soluble unit-dose pouch of tetracetyl ethylene diamine (TAED). In another embodiment, the water-soluble unit-dose pouch comprises between 0.0001% and 1% by weight of the water-soluble unit-dose pouch of tetracetyl ethylene diamine (TAED). In yet another embodiment, the water-soluble unit-dose pouch is devoid of tetracetyl ethylene diamine (TAED).

TAED is a peracid-delivering bleach activator (opposed to diacyl peroxide-delivering bleach activator) used in conjunction with an oxygen bleach source such as sodium percarbonate is very effective at removing tough beverage stains such as coffee, tea, red wine, black grape juice etc. However a problem associated with the combination of an oxygen bleach source and TAED is patchy discoloration damage. Patchy discoloration damage is where the cleaning composition causes localized colour bleaching on the fabrics. This is then seen as white patches on coloured fabrics.

It was surprisingly found that water-soluble unit-dose pouches that comprise an oxygen bleach source, low levels of TAED or no TAED, and between 2% and 20%, preferably 3% and 20%, preferably 5% and 20%, more preferably 7.5% and 15%, most preferably between 10% and 13% by weight of the water-soluble unit-dose pouch of sodium citrate exhibited the same effective stain removal performance as the combination of an oxygen source and higher levels of TAED (5-10% by weight of the composition), but exhibited reduced patchy discoloration damage of fabrics.

Diacyl Peroxide Bleach Activators

In one embodiment, the solid composition according to the present invention comprises a diacyl peroxide bleach activator. Diacyl peroxide bleach activators boost the cleaning power of the oxygen-based bleaching agent via a perhydrolysis reaction brought about by nucleophilic attack on the diacyl peroxide bleach activator by a perhydroxide anion.

The diacyl peroxide bleach activator that may be used in the present has the general formula:

$$\text{R}^-\text{C}^-\text{LG}$$

wherein $R$ is an alkyl group, linear or branched, containing from about 1 to 11 carbon atoms and I.G is a suitable leaving group.
Generally, a suitable leaving group is electrophilic and is stable such that the rate of the reverse reaction is negligible. This facilitates the nucleophilic attack by the perhydroxide anion. The leaving group must also be sufficiently reactive for the reaction to occur within the optimum time frame, for example during the wash cycle. However, if the leaving group is too reactive, the bleach activator will be difficult to stabilize. These characteristics are generally parallelled by the pKa of the conjugate acid of the leaving group, although exceptions to this convention are known. The conjugate acid of the leaving group in accordance with the present invention preferably has a pKa in a range from about 4 to about 13, more preferably from about 6 to about 11, and most preferably from about 8 to about 11.

Preferably, the leaving group has the formula:

\[
\begin{align*}
\text{O} & \quad \text{Y} \\
\text{R} & \quad \text{X}
\end{align*}
\]

wherein Y is selected from the group consisting of SO₃⁻, M⁺, COO⁻/M⁺, SO₄⁻/M⁺, PO₄⁻/M⁺, PO₃⁻/M⁺, (N¹R²)₂X⁻ and O⁻N(R²)₂. M⁺ is a cation and X⁻ is an anion, both of which provide solubility to the bleach activator, and R² is an alkyl chain containing from about 1 to about 4 carbon atoms or H. In accordance with the present invention, M⁺ is preferably an alkali metal, with sodium being most preferred. Preferably, X⁻ is a hydroxide, methylsulfate or acetate anion.

Other suitable leaving groups have the following formulas:

\[
\begin{align*}
\text{O} & \quad \text{R}^3 \\
\text{Y} & \quad \text{R}^3
\end{align*}
\]

wherein Y is the same as described above and R³ is an alkyl chain containing from about 1 to about 8 carbon atoms, H or R².

While numerous diacyl peroxide bleach activators as described above are suitable for use in the present invention, a preferred diacyl peroxide bleach activator has the formula:

\[
\begin{align*}
\text{R} & \quad \text{O} \\
\text{C} & \quad \text{O} \\
\text{SO}_3^- & \quad \text{Na}
\end{align*}
\]

wherein R is an alkyl chain, linear or branched, containing from 1 to 11 carbon atoms. More preferably, R is an alkyl chain, linear or branched, containing from 3 to 11, even more preferably from 8 to 11.

Most preferably, according to the present invention, the diacyl peroxide bleach activator has the formula:

\[
\begin{align*}
\text{CH}_2-(\text{CH}_2)_3 & \quad \text{O} \\
\text{O} & \quad \text{SO}_3^- \\
\text{Na} & \quad \text{Na}
\end{align*}
\]

which is also referred to as sodium n-nonyloxybenzene sulfonate (hereinafter referred to as “NOBS”).

This diacyl peroxide bleach activator and those described previously may be readily synthesized by well known reaction schemes or purchased commercially. Those skilled in the art will appreciate that other diacyl peroxide bleach activators beyond those described herein which are readily water-soluble can be used in the solid composition without departing from the scope of the invention.

In one embodiment, the compositions of the present invention comprise from 0.5% to 35%, preferably from 1.5% to 26% and more preferably from 2.5% to 20%, by weight of the pouch of a diacyl peroxide bleach activator having the formula:

\[
\begin{align*}
\text{R} & \quad \text{O} \\
\text{LG} & \quad \text{LG}
\end{align*}
\]

wherein R is an alkyl group, linear or branched, containing from about 1 to 11 carbon atoms and LG is a suitable leaving group.

Optional Ingredients

The water-soluble unit-dose pouches herein may further comprise a variety of other optional ingredients such as surfactants, fillers, chelating agents, radical scavengers, antioxi dants, stabilisers, builders, soil suspending polymer, polymeric soil release agents, dye transfer inhibitor, solvents, sud s controlling agents, sud s booster, brighteners, perfumes, pigments, dyes and the like. The optional ingredients may be present in any of the compartments and compositions of the water-soluble unit-dose pouch.

Method of Use

One aspect of the present invention is a method of treating fabrics comprising the steps of:

a) Placing fabrics in the drum of a fabric washing machine;

b) Adding a water-soluble unit-dose pouch according to the present invention to the drum of the fabric washing machine;

c) Washing the fabrics in the fabric washing machine.

Preferably, in step b) a second fabric cleaning composition is added to the drum or drawer of the washing machine. Thus, the water-soluble unit-dose pouch in this embodiment serves as a fabric additive.

Data

Stain Removal

The stain removal ability of water-soluble unit-dose pouches according to the present invention and ones outside of the claims of the present invention were compared. Fabrics were prepared that contained various stains. These stain fabrics were purchased from WFK, code SBL 2004. Image analysis was used to compare each stain to an unstained fabric control. Software converted images taken into standard colorimetric values and compared these to standards based on the commonly used Macbeth Colour Rendition Chart and assigns each stain a colorimetric value. Eight replicates of each were prepared.

Stained fabrics were then washed place in the drum of Miele Washing machine Novotronic W327 together with 3 kg of clean fabrics (ballast) at 30° C, main wash cycle program Lava/Indossa. To this, commercially available detergent was added (Dash con Actilift Liquido), and also one of two different pouches.
Comparative Pouch A is commercially available as Omino Bianco 100 più Idrocaps Smacchiatore Concentrato. It is a single compartment pouch comprising a powder composition. It was determined that it comprised 5% TAED and 45.1% sodium percarbonate by weight of the pouch, and no salt of citric acid.

A second pouch according to the present invention was prepared and designated Pouch 1. This was a water-soluble unit-dose pouch comprising a powder composition in a first compartment, and a liquid composition in a second compartment. Pouch 1 comprised (percent by weight of the pouch;)

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<tr>
<th>Powder Composition</th>
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<tr>
<td>Sodium Citrate</td>
<td>9.2</td>
</tr>
<tr>
<td>TAED</td>
<td>0.8</td>
</tr>
<tr>
<td>Percarbonate</td>
<td>42.5</td>
</tr>
<tr>
<td>Amylase</td>
<td>1.1</td>
</tr>
<tr>
<td>Carboxy methyl cellulose</td>
<td>0.04</td>
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<tr>
<td>Citric Acid</td>
<td>0.03</td>
</tr>
<tr>
<td>Acrylic acid/maleic acid co-polymer</td>
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<td>HEDP (Na)</td>
<td>2.4</td>
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<td>Linear alkyl benzene</td>
<td>0.006</td>
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<td>Linear alkylbenzene sulfonate</td>
<td>0.7</td>
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</tr>
<tr>
<td>Na₂CO₃</td>
<td>0.07</td>
</tr>
<tr>
<td>Na₂SO₄</td>
<td>5.4</td>
</tr>
<tr>
<td>PEG 4000</td>
<td>0.7</td>
</tr>
<tr>
<td>Palmitic Acid</td>
<td>0.7</td>
</tr>
<tr>
<td>Perfluro microparticles</td>
<td>0.45</td>
</tr>
<tr>
<td>Protease</td>
<td>0.6</td>
</tr>
<tr>
<td>Silica</td>
<td>0.008</td>
</tr>
<tr>
<td>Starch</td>
<td>0.27</td>
</tr>
<tr>
<td>Zeolite A</td>
<td>5.6</td>
</tr>
<tr>
<td>Misc impurities</td>
<td>0.9</td>
</tr>
<tr>
<td>Water</td>
<td>2.5</td>
</tr>
</tbody>
</table>

Following the wash, the stains were analysed again. The percentage decrease in stains following the wash versus prior to the wash as compared to an unstained control was then calculated. Results can be seen in Table 1.

<table>
<thead>
<tr>
<th>Stain</th>
<th>Comparative Pouch A</th>
<th>Pouch 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black Grape Juice</td>
<td>97</td>
<td>98</td>
</tr>
<tr>
<td>Blackberry</td>
<td>83</td>
<td>83</td>
</tr>
<tr>
<td>Espresso coffee</td>
<td>71</td>
<td>71</td>
</tr>
<tr>
<td>Green Tea</td>
<td>57</td>
<td>58</td>
</tr>
<tr>
<td>Kraft Balsamic</td>
<td>88</td>
<td>89</td>
</tr>
<tr>
<td>Vinegar</td>
<td>71</td>
<td>75</td>
</tr>
<tr>
<td>Red Wine</td>
<td>45</td>
<td>46</td>
</tr>
</tbody>
</table>

As can be seen from Table 1, both pouches A and 1 gave comparable stain removal ability on all stains tested. Thus 0.8% by weight of the pouch of TAED in combination with between 2% and 20% by weight of the pouch of a salt of citric acid gave comparable cleaning to a pouch comprising 5% TAED by weight of the pouch and no salt of citric acid.

Patchy Damage

The effect of patchy fabric damage of fabrics washed with water-soluble unit-dose pouches according to the present invention and also versus comparative pouch A was tested.

International Association for Soaps, Detergents and Maintenance Products (AISE) standard fabrics were prepared. Fabrics AISE 20, AISE 22 and AISE 29 were used, the difference being each was coloured with a different dye. Each fabric sample was cut to a size of 13 cm by 26 cm. The fabric was then folded to make a 13 cm by 13 cm pocket and the water-soluble unit-dose pouch to be tested placed within this pocket. The fabric pockets containing the pouches were then washed at 30°C in a Bauknecht WA9437 washing machine, on woollens program 9 together with 3 white 100% cotton double sheets to act as ballast. 12 replicates were completed for each fabric and the results averaged.

The different pouches tested according to the present invention can be seen in Table 2 (percentage by weight of the pouch;)

<table>
<thead>
<tr>
<th>Component</th>
<th>Pouch 2</th>
<th>Pouch 3</th>
<th>Pouch 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder composition</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sodium citrate</td>
<td>2.46</td>
<td>2.46</td>
<td>2.46</td>
</tr>
<tr>
<td>TAED</td>
<td>0.38</td>
<td>1.12</td>
<td>1.89</td>
</tr>
<tr>
<td>Percarbonate</td>
<td>46.75</td>
<td>46.75</td>
<td>46.75</td>
</tr>
<tr>
<td>Amylase</td>
<td>1.23</td>
<td>1.23</td>
<td>1.23</td>
</tr>
<tr>
<td>Carboxy methyl cellulose</td>
<td>0.019</td>
<td>0.06</td>
<td>0.09</td>
</tr>
<tr>
<td>Acrylic acid/maleic acid co-polymer</td>
<td>5.87</td>
<td>5.87</td>
<td>5.87</td>
</tr>
<tr>
<td>Linear alkylbenzene sulfonate</td>
<td>0.12</td>
<td>0.12</td>
<td>0.12</td>
</tr>
<tr>
<td>NOBS</td>
<td>3.19</td>
<td>3.19</td>
<td>3.19</td>
</tr>
<tr>
<td>PEG 4000</td>
<td>0.19</td>
<td>0.23</td>
<td>0.23</td>
</tr>
<tr>
<td>Palmitic Acid</td>
<td>0.23</td>
<td>0.23</td>
<td>0.23</td>
</tr>
<tr>
<td>Protease</td>
<td>0.81</td>
<td>0.81</td>
<td>0.81</td>
</tr>
<tr>
<td>Misc impurities</td>
<td>0.22</td>
<td>0.26</td>
<td>0.29</td>
</tr>
<tr>
<td>Water</td>
<td>0.85</td>
<td>0.84</td>
<td>0.84</td>
</tr>
<tr>
<td>Water soluble polycarboxylate</td>
<td>22.6</td>
<td>21.8</td>
<td>21.0</td>
</tr>
</tbody>
</table>

Patchy discolouration damage was then assessed visually by two independent judges. The scale used was as follows: 0—undamaged; 1—slightly damaged; 2—damaged (larger damaged area or several small damaged areas with high intensity); 3—Badly damaged (several large damaged areas with high intensity); 4—very badly damaged (several very large areas with high intensity). The average results of the two judges can be seen in Table 3.

<table>
<thead>
<tr>
<th>Component</th>
<th>Pouch 2</th>
<th>Pouch 3</th>
<th>Pouch 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid composition</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dipropylene Glycol</td>
<td>7.2</td>
<td>7.2</td>
<td>7.2</td>
</tr>
<tr>
<td>Glycerine</td>
<td>0.375</td>
<td>0.375</td>
<td>0.375</td>
</tr>
<tr>
<td>Propylene glycol</td>
<td>0.027</td>
<td>0.027</td>
<td>0.027</td>
</tr>
<tr>
<td>Deionized water</td>
<td>0.9</td>
<td>0.9</td>
<td>0.9</td>
</tr>
<tr>
<td>Acid Blue 80</td>
<td>0.027</td>
<td>0.027</td>
<td>0.027</td>
</tr>
<tr>
<td>Neadol 91 EOS</td>
<td>0.33</td>
<td>0.33</td>
<td>0.33</td>
</tr>
<tr>
<td>LF244 non-ionic surfactant available from Clorox Film</td>
<td>3.7</td>
<td>3.7</td>
<td>3.7</td>
</tr>
</tbody>
</table>

| M8630 available from Monosol | 2.45 | 2.45 | 2.45 |
TABLE 3 Comparative Fabric Pouch 2 Pouch 3 Pouch 4 Comparative Pouch A

<table>
<thead>
<tr>
<th>Fabric</th>
<th>Pouch 2</th>
<th>Pouch 3</th>
<th>Pouch 4</th>
<th>Comparative Pouch A</th>
</tr>
</thead>
<tbody>
<tr>
<td>AISE 29</td>
<td>0.13</td>
<td>1.13</td>
<td>1.13</td>
<td>2.4</td>
</tr>
<tr>
<td>AISE 20</td>
<td>0.04</td>
<td>1.13</td>
<td>2.17</td>
<td>3.4</td>
</tr>
<tr>
<td>AISE 22</td>
<td>0.21</td>
<td>0.33</td>
<td>0.88</td>
<td>2.6</td>
</tr>
</tbody>
</table>

As can be seen from Table 3, the lower the level of TAED, the lower the degree of patchy discoloration damage.

It is clear from Tables 2 and 3, that the presence of chelant and low levels of TAED results in a comparable degree of stain removal but a lower level of patchy discoloration damage.

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".

Every document cited herein, including any cross referenced or related patent or application, is hereby incorporated herein by reference in its entirety unless expressly excluded or otherwise limited. The citation of any document is not an admission that it is prior art with respect to any invention disclosed or claimed herein or that it alone, or in any combination with any other references or references, teaches, suggests or discloses any such invention. Further, to the extent that any meaning or definition of a term in this document conflicts with any meaning or definition of the same term in a document incorporated by reference, the meaning or definition assigned to that term in this document shall govern.

While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

What is claimed is:

1. A water-soluble unit-dose pouch comprising at least a first compartment, wherein the first compartment comprises a powder composition, and wherein the powder composition comprises an oxygen bleach source and a salt of citric acid, and wherein the salt of citric acid is present between 2% and 20% by weight of the water-soluble unit-dose pouch.

2. The water-soluble unit-dose pouch of claim 1, wherein the salt of citric acid is present at a concentration of between 3% and 20% by weight of the water-soluble unit-dose pouch.

3. The water-soluble unit-dose pouch of claim 1 wherein the salt of citric acid is selected from the group comprising sodium citrate, calcium citrate, potassium citrate, ammonium citrate, magnesium citrate and mixtures thereof.

4. The water-soluble unit-dose pouch of claim 3, wherein the salt of citric acid is sodium citrate.

5. The water-soluble unit-dose pouch of claim 1, wherein the oxygen bleach source is a peroxy-based bleach source.

6. The water-soluble unit-dose pouch of claim 5, wherein the peroxy-source is selected from the group comprising sodium percarbonate, potassium percarbonate, sodium perborate, potassium perborate, phthaloyl amino-peroxy caproic acid and mixtures thereof.

7. The water-soluble unit-dose pouch of claim 1, wherein the powder composition comprises a bleach activator having the formula:

![Bleach Activator Formula](image)

wherein R is an alkyl group, linear or branched, containing from about 1 to 11 carbon atoms and LG is a suitable leaving group.

8. The water-soluble unit-dose pouch of claim 7, wherein the bleach activator has the formula:

![Bleach Activator Formula](image)

wherein R is an alkyl chain, linear or branched, containing from 1 to 11 carbon atoms.

9. The water-soluble unit-dose pouch of claim 8, wherein the powder composition comprises n-nonanoxyloxybenzenesulphonate.

10. The water-soluble unit-dose pouch of claim 1, wherein the powder comprises between 0.00001% and 1% by weight of the water-soluble unit-dose pouch of tetroctyl ethylene diamine.

11. The water-soluble unit-dose pouch of claim 1, wherein the powder is devoid of tetroctyl ethylene diamine.

12. The water-soluble unit-dose pouch of claim 1, wherein the powder is made of a pouch material, and wherein the pouch material comprises a polyvinyl alcohol polymer.

13. The water-soluble unit-dose pouch of claim 1 comprising at least a first compartment and a second compartment.

14. The water-soluble unit-dose pouch of claim 13, wherein the second compartment comprises a liquid composition.

15. A method of treating fabrics comprising the steps of:

a. Placing fabrics in the drum of a fabric washing machine;

b. Adding a water-soluble unit-dose pouch according to any preceding claims to the drum of the fabric washing machine;

c. Washing the fabrics in the fabric washing machine.

* * * * *