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(54) **Title:** IMPROVEMENTS RELATING TO FABRIC TREATMENT COMPOSITIONS

(57) **Abstract:** A fabric cleaning composition for use in a laundering process which comprises: a) at least one anionic surfactant, b) at least one nonionic surfactant, c) a powder of botanical origin, and d) a polymeric deposition aid at a level of from 0.01 to 2% by total weight of the composition, wherein the powder of botanical origin has a particle size of from 0.1 to 100 µm, preferably from 0.5 to 50 and most preferably from 1 to 45 µm.

## **IMPROVEMENTS RELATING TO FABRIC TREATMENT COMPOSITIONS**

### **Technical Field**

The present invention relates to fabric cleaning compositions comprising micro powders of botanical origin.

### **Background**

Using clay particles to impart softness to fabrics from the wash or from the rinse is well known in the art. Similarly the use of nano-particles of silica or titanium dioxide and micro-encapsulated perfume particles are now quite well documented for enhanced perfume impact.

US2003/0087788 (Rhodia Inc) discloses water dispersible granules comprising a hydrophobic fragrance in the form of droplets, which is finely divided in and encapsulated by a water-soluble/dispersible solid organic matrix, which may be polypeptides of plant or synthetic origin, such as soya or wheat.

EP0908171 (Gem Energy Industry Ltd) discloses a dry powder herbal cleaning composition containing (a) a herb which acts as a soap or detergent; (b) a herb which acts as a foaming accelerating agent; (c) a herb which acts as a foaming agent; (d) a herb which acts as a pigmentation or colour restoring or maintenance agent; and (e) a herb which acts as a conditioner.

WO00/77153 (Unilever plc) discloses a detergent tablet containing (a) particles which contain organic surfactant and detergency builder; and (b) water insoluble but water swellable particles of plant material which contain both cellulose and lignin, such as coconut husk, obtainable by fragmentation of plant material without separation of its fibres into a liquid dispersion.

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We have now discovered that micronised powders of botanical origin can be deposited onto fabrics from a laundry wash product to impart fabrics with benefit agents or for novel fragrance enhancement. Examples of such powders include ginger for fragrance; lime, lemon or orange rind for freshness; flower petals for  
5 perfume; exfoliating agents such as oat kernel flour, rice or rice bran flour; various pigments of natural origin, such as turmeric powder for anti-inflammatory benefit; seeds such as cardamom and coriander for perfuming; various dried herbs such as mint, oregano, green tea, bay leaf and tea-tree for perfuming and anti-microbial benefits.

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### **Definition of the Invention**

In a first aspect, the present invention provides a fabric cleaning composition for use in a laundering process which comprises:

15

- a) at least one anionic surfactant,
- b) at least one nonionic surfactant,
- c) a powder of botanical origin, and
- d) a polymeric deposition aid at a level of from 0.01 to 2% by total weight of  
20 the composition,

wherein the powder of botanical origin has a particle size of from 0.1 to 100  $\mu\text{m}$ , preferably from 0.5 to 50 and most preferably from 1 to 45  $\mu\text{m}$ .

25 In a second aspect of the present invention there is provided a process for treating fabric comprising the step of treating a fabric article with a composition as defined in any preceding claim.

In a further aspect, the present invention provides a fabric article comprising the  
30 botanical micro-particles, resulting from the treatment process of the second aspect of the invention.

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In a yet further aspect of the invention, there is provided a method of preparing a composition, comprising the step of pulverizing the powder of botanical origin to a particle size of from 0.1 to 100 microns preferably from 0.2 to 90 microns, more preferably from 0.5 to 60, even more preferably from 1 to 45 microns and most preferably from 2 to 30 microns.

### **Detailed Description of the Invention**

#### **The Powders of Botanical Origin**

10

The present invention requires the use of ultra-fine powders. The powders suitable for use in the compositions of the invention are sparingly soluble, preferably insoluble particulate structures of botanical origin. They are preferably aromatic and as such preferably comprise one or more aromatic essential oils.

15

The powders of the present invention can be of any shape and are not necessarily spherical and can have aspect ratio (ratio of length to diameter) greater than 1. Within the context of this invention, by micronised, ultra-fine, or finely divided, is meant particle sizes having a maximum axis of from 0.1 to 100 microns preferably from 0.2 to 90 microns, more preferably from 0.5 to 60, even more preferably from 1 to 45 microns and most preferably from 2 to 30 microns.

20

Particles of suitable size may be obtained by any suitable process, for example pulverizing, milling, etc. Fractionation, for example by sieving, may be used to separate the required particle size range.

25

The powder is a material of botanical origin, and may be derived from any part of the plant, such as tubers, fruits, flowers, flower parts (calyx, stamen, etc), seeds, beans, pods, pith, barks, stalks, stems, leaves, roots, rhizomes, husks, skins, rinds and mixtures thereof.

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Examples of aromatic powders include vanilla bean powder, ginger powder, cinnamon powder, cocoa powder, clove powder, cardamom powder, coriander powder, cumin powder, caraway powder, dill powder, chili powder, cyanine powder, turmeric powder, coffee bean powder, tea leaf powder, tea-tree powder, 5 blackberry leaf powder, Lemon balm leaf powder, herb powders (for example, rosemary, thyme, sweet bay, sage, tarragon, mint, basil, balm, lemon verbena, red bergamot, marjoram, lavender and oregano), chamomile powder, fennel powder, lime rind powder, lemon rind powder, grapefruit rind powder, tangerine rind powder, mandarin rind powder, lemon grass powder, apple pomace powder, 10 Lemon myrtle powder, red clover powder, liquorice powder and mixtures thereof.

Preferred non-aromatic powders include ginseng powder, rice husk flour, wheat husk flour, buckwheat husk flour and mixtures thereof.

15 The powdered material can be in native form or may result from processes such as ageing, fermenting and roasting that would increase or alter the constituent aroma chemicals.

20 Delivery of such powders onto fabrics and clothing by a laundry cleaning product can impart a host of benefits that otherwise would be difficult to deliver from laundry applications. These include long lasting perfume and freshness, anti-bacterial activity, deodorizing activity, skin anti-ageing and well-being activity, change of fabric feel (handle), cooling benefit and warming benefit.

25 The plant powders of the invention can be incorporated into laundry compositions in a similar fashion as other powdered ingredients.

The powder of botanical origin is present in an amount of from 0.1 to 10 wt %, preferably from 0.3 to 6 wt %, more preferably from 0.4 to 4 wt % and most 30 preferably from 0.5 to 3 wt % by weight of the total composition.

- 5 -

The powders of botanical origin suitable for use in the invention are not extracted or isolated materials, such as polymers, for example lignin, starches or proteins, but are plant parts that are powdered in their bulk state. The powders are not intended to include cellulose or cellulose derivatives such as polymeric derivatives  
5 such as cellulose ethers. The plant parts (i.e. pre-powdered) and the powders are not subject to chemical purification, for example, to remove components such as lignin or pigments (but may, of course, be washed or cleaned).

### **The Polymeric Deposition Aid**

10

Deposition of micro-powder from the laundry formulations of the invention onto a substrate may be achieved by any suitable route.

15

For example, by filtration, in which the particle size of the powder is such that the particles are trapped between the fibres of the fabric. The filtration mechanism requires particles or clusters of primary particles of a size comparable with the interyarn pore size.

20

The particle size suitable for filtration is typically in the range of from 1 to 30 microns. Larger particles begin to be visible to the unaided eye, whereas smaller particles tend to be removed in the wash. Particles of around from 5 to 15 microns are preferred as they tend to be invisible to the eye and exhibit good deposition by filtration onto fabrics.

25

Deposition of micro-powders from laundry formulations of the invention is achieved and enhanced over and above the delivery by filtration method, by polymer aided deposition. The polymeric deposition aid is suitably present at a level of from 0.0001 to 2%, preferably from 0.0002 to 1.0 %, more preferably 0.0005 to 0.5 % and most preferably from 0.0005 to 0.05 % by total weight of the

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composition.

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Polymeric deposition agent suitable for use in the present invention include modified natural polymers and synthetic polymers.

Polymers suitable for the deposition of micro-powders are disclosed in

5 WO9709406, particularly high MW polyethylene oxides (PEO) which are used to deposit clay particles in the main wash; EP0299575B1 and WO9527037 disclose high MW PEO, polyacrylates, polyacryl amides, poly vinyl alcohol and poly ethylene imines, which are used to deposit clay particles in the main wash; and EP0387426B1 which utilizes a similar list of polymers as well as guar gums.

10

WO 01/07546 A1 discloses suitable rinse stage polymeric deposition aids for emulsion droplets including cationic guar polymers, cationic polyacrylamides, cationic potato starch, and cationic cellulose derivates.

15 Preferred modified natural polymers suitable for use in the present invention may be selected from the group consisting of cationic starches, cationic guar, cationic cellulose, and nonionic locust bean gums (LBGs). A preferred cationic starch is a cationically modified potato starch.

20 Suitable examples of cationic polymers include cationic guar polymers such as Jaguar (ex Rhone Poulenc), cationic cellulose derivatives such as Celquats (ex National Starch), Flocaid (ex National Starch), cationic potato starch such as SoftGel (ex Aralose) and cationic polyacrylamides such as PCG (ex Allied Colloids).

25

Suitable non-ionic deposition aids include high molecular weight polyethylene glycols, for example PEO WSRN 750 (ex Union Carbide).

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Preferred synthetic polymers, for use as a deposition aid, may be selected from the group consisting of polyethylene oxide (PEO), polyethylene imine (PEI), poly (acrylate), poly (acrylamide), polyethylene terephthalate-polyoxyethylene terephthalate (PET/POET) polymers and mixtures thereof.

5

### **The Anionic and Nonionic Surfactants**

The compositions of the invention comprise at least one anionic surfactant and at least one nonionic surfactant.

10

The compositions of the invention contain at least one anionic surfactant. Examples include alkylbenzene sulfonates, such as linear alkylbenzene sulfonate, particularly linear alkylbenzene sulfonates having an alkyl chain length of C<sub>8</sub>-C<sub>15</sub>. It is preferred that the level of linear alkylbenzene sulfonate is from 0 wt% to 30 wt%, more preferably 1 wt% to 25 wt%, most preferably from 2 wt% to 15 wt%.

15

The compositions of the invention may contain other anionic surfactants in amounts additional to the percentages quoted above. Suitable anionic surfactants are well-known to those skilled in the art. Examples include primary and secondary alkyl sulfates, particularly C<sub>8</sub>-C<sub>20</sub> primary alkyl sulfates; alkyl ether sulfates; olefin sulfonates; alkyl xylene sulfonates; dialkyl sulfosuccinates; and fatty acid ester sulfonates. Sodium salts are generally preferred.

20

The compositions of the invention also contain at least one non-ionic surfactant.

25

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C<sub>8</sub>-C<sub>20</sub> aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C<sub>10</sub>-C<sub>15</sub> primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

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It is preferred that the level of non-ionic surfactant is from 0.5 wt% to 30 wt%, preferably from 1 wt% to 25 wt%, most preferably from 2 wt% to 15 wt%.

### **The Fabric Cleaning Composition**

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The fabric cleaning composition of the invention is suitable for use in a laundry process. The composition is preferably a main wash cleaning composition, or a softening-in-the-wash composition.

10 The compositions of the invention may be in any physical form e.g. a solid such as a powder or granules, a tablet, a solid bar, a paste, gel or liquid, for example an aqueous based liquid, a spray, a stick, an impregnated substrate, foam or mousse. In particular the compositions may be liquid, powder, or unit dose such as tablet laundry compositions.

15

The liquid products of the invention may have pH ranging from 6 to 12 (for fabric softening-in-the-wash compositions). This pH range preferably remains stable over the shelf life of the product.

20 Compositions in accordance with the invention may comprise at least one further surface-active compound, selected from soaps, cationic surfactants, zwitterionic surfactants, amphoteric surfactants and mixtures thereof. The choice of surface-active compound (surfactant), and the amount present, will depend on the intended use of the detergent composition. In fabric washing compositions,  
25 different surfactant systems may be chosen, as is well known to the skilled formulator, for handwashing products and for products intended for use in different types of washing machine.

30 Many suitable surface-active compounds are available and are described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch. The choice of surface-active compound

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(surfactant), and the amount present, will depend on the intended use of the detergent composition. In fabric washing compositions, different surfactant systems may be chosen, as is well known to the skilled formulator, for handwashing products and for products intended for use in different types of washing machine.

The total amount of surfactant present will also depend on the intended end use and may be as high as 60 wt%, for example, in a composition for washing fabrics by hand. In compositions for machine washing of fabrics, a total amount of from 10 to 40 wt% is generally appropriate. Typically the compositions will comprise at least 2 wt% total surfactant e.g. 2-60%, preferably 15-40% most preferably 25-35%.

Detergent compositions suitable for use in most automatic fabric washing machines generally contain anionic non-soap surfactant, or non-ionic surfactant, or combinations of the two in any suitable ratio, optionally together with soap.

It is possible to include certain mono-alkyl cationic surfactants which can be used in main-wash compositions for fabrics. Cationic surfactants that may be used include quaternary ammonium salts of the general formula  $R_1R_2R_3R_4N^+ X^-$  wherein the R groups are long or short hydrocarbon chains, typically alkyl, hydroxyalkyl or ethoxylated alkyl groups, and X is a counter-ion (for example, compounds in which  $R_1$  is a  $C_8$ - $C_{22}$  alkyl group, preferably a  $C_8$ - $C_{10}$  or  $C_{12}$ - $C_{14}$  alkyl group,  $R_2$  is a methyl group, and  $R_3$  and  $R_4$ , which may be the same or different, are methyl or hydroxyethyl groups); and cationic esters (for example, choline esters).

Amphoteric and zwitterionic surfactants that may be used include alkyl amine oxides, betaines and sulfobetaines.

### Softening in the Wash

Where the composition is a softening from the wash composition, it may comprise a sugar polyester or a softening silicone oil.

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The sugar polyester is preferably selected from the group consisting of sucrose polyesters, glucose polyesters and cellobiose polyesters, and is most preferably a sucrose polyester.

10 The sugar polyester may be liquid, soft solid or solid.

The preferred sucrose polyesters for use in the present invention have 2 to 4 hydrocarbon chains per sugar ring, where the hydrocarbon chain has a length of from 12 to 22 carbon atoms. A particularly preferred sucrose polyester is sucrose  
15 tetraerucate.

An example of a preferred sucrose polyester is Ryoto Sugar Ester ER290 supplied by Mitsubishi Kagaku Foods Corporation, which is a sucrose tetraerucate and according to the manufacturer's specification is mainly Tetraerucate, Pentaerucate  
20 and Hexaerucate and has a HLB value of 2.

The sugar polyester may be pure, or may contain impurities. When present, the impurities are preferably selected from the group consisting of free fatty acid, fatty acid methyl ester, soap, inorganic salts and mixtures thereof.

25

The most preferred SPEs are commercially available, such as Emanon SCR-PK (ex KAO), which is a palm kernel derived SPE containing mainly C<sub>12</sub>-C<sub>14</sub> with about 20% C<sub>18</sub> mono unsaturation and SPE-THSBO (ex Clariant), which is derived from touch hardened soy bean oil, having mainly C<sub>16</sub>-C<sub>18</sub> chains with  
30 about 80% mono and di unsaturation. The average degree of esterification of the above preferred SPEs is between 4.2-4.7.

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SCR-PK contains up to 20% impurities but SPE-THSBO is pure. SCR-PK contains from 4 to 6 wt% of K soap, 2.5 wt% of free fatty acid, from 10 to 15 wt% of fatty acid methyl ester and less than 1 % of KCl.

5 The sugar polyester, being non-ionic oil, requires an emulsifier, that is to say, the sugar polyester must be in an emulsified form. The emulsifier is preferably selected from cationic surfactant, anionic surfactant, non-ionic surfactant, and mixtures thereof.

10 Alternatively, the ultra-fine particles of the invention themselves can act as stabilizer and emulsifier for the nonionic softeners such as sugar polyesters (SPEs). Recently the interest in study of solid particles as emulsifies has been re-awakened (Binks, B. P. Current Opinions in Colloid Interface Science, 2002, 7, 21).

15

Most of recent activities on surface active colloidal particles have focused on very low aspect ratio (spherical) particles. Only recently Alargova et al, Langmuir, 2006, 22, 765-774, have shown that high aspect ratio particles can be used for emulsion stabilisation.

20

The compositions of the invention, when used as main wash fabric washing compositions, will generally also contain one or more detergency builder. The total amount of detergency builder in the compositions will typically range from 0 to 80 wt%, preferably from 0 to 60 wt%.

25

Inorganic builders that may be present include sodium carbonate, if desired in combination with a crystallisation seed for calcium carbonate, as disclosed in GB 1 437 950 (Unilever); crystalline and amorphous aluminosilicates, for example, zeolites as disclosed in GB 1 473 201 (Henkel), amorphous aluminosilicates as disclosed in GB 1 473 202 (Henkel) and mixed crystalline/amorphous  
30 aluminosilicates as disclosed in GB 1 470 250 (Procter & Gamble); and layered

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silicates as disclosed in EP 164 514B (Hoechst). Inorganic phosphate builders, for example, sodium orthophosphate, pyrophosphate and tripolyphosphate are also suitable for use with this invention.

- 5 The compositions of the invention preferably contain an alkali metal, preferably sodium, aluminosilicate builder. Sodium aluminosilicates may generally be incorporated in amounts of from 5 to 60% by weight (anhydrous basis), preferably from 10 to 50 wt%, especially from 25 to 50 wt%.
- 10 The alkali metal aluminosilicate may be either crystalline or amorphous or mixtures thereof, having the general formula:  $0.8-1.5 \text{ Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 0.8-6 \text{ SiO}_2$ . These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5  $\text{SiO}_2$  units (in the formula above). Both the amorphous and the
- 15 crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature. Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A
- 20 and X, and mixtures thereof.

The zeolite may be the commercially available zeolite 4A now widely used in laundry detergent powders. In an alternative embodiment of the invention, the zeolite builder incorporated in the compositions of the invention is maximum

25 aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070A (Unilever). Zeolite MAP is defined as an alkali metal aluminosilicate of the zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, and more preferably within the range of from 0.90 to 1.20.

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In the case of zeolite MAP, zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00, is especially preferred. The calcium binding capacity of zeolite MAP is generally at least 150 mg CaO per g of anhydrous material.

5

The zeolites may be supplemented by other inorganic builders, for example, amorphous aluminosilicates, or layered silicates such as SKS-6 ex Clariant.

The zeolite may be supplemented by organic builders. Organic builders that may  
10 be present include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di and trisuccinates, carboxymethyloxy succinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyl iminodiacetates, alkyl- and alkenylmalonates and succinates; and  
15 sulfonated fatty acid salts. This list is not intended to be exhaustive.

Especially preferred organic builders are citrates, suitably used in amounts of from 1 to 30 wt%, preferably from 5 to 30 wt%, more preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in  
20 amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%.

Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form.

25 Builders are suitably present in total amounts of from 10 to 80 wt%, more preferably from 20 to 60 wt%. Builders may be inorganic or organic.

A built composition in accordance with the invention may most preferably comprise from 10 to 80 wt% of a detergency builder (b) selected from zeolites,  
30 phosphates, and citrates.

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The laundry detergent composition will generally comprise other detergent ingredients well known in the art. These may suitably be selected from bleach ingredients, enzymes, sodium carbonate, sodium silicate, sodium sulphate, foam controllers, foam boosters, perfumes, fabric conditioners, soil release polymers, dye transfer inhibitors, photobleaches, fluorescers and coloured speckles.

5 Compositions according to the invention may also suitably contain a bleach system. Fabric washing compositions may desirably contain peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, capable of yielding hydrogen peroxide in aqueous solution.

10

Suitable peroxy bleach compounds include organic peroxides such as urea peroxide, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulfates. Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate.

15

Especially preferred is sodium percarbonate having a protective coating against destabilisation by moisture. Sodium percarbonate having a protective coating comprising sodium metaborate and sodium silicate is disclosed in GB 2 123 044B (Kao).

20

The peroxy bleach compound is suitably present in an amount of from 0.1 to 35 wt%, preferably from 0.5 to 25 wt%. The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 0.1 to 8 wt%, preferably from 0.5 to 5 wt%.

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Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and pernonanoic acid precursors. Especially preferred bleach precursors suitable for use in the present invention are N,N,N',N',-tetracetyl

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ethylenediamine (TAED) and sodium nonanoyloxybenzene sulphonate (SNOBS). The novel quaternary ammonium and phosphonium bleach precursors disclosed in US 4 751 015 and US 4 818 426 (Lever Brothers Company) and EP 402 971A (Unilever), and the cationic bleach precursors disclosed in EP 284 292A and  
5 EP 303 520A (Kao) are also of interest.

The bleach system can be either supplemented with or replaced by a peroxyacid, examples of such peracids can be found in US 4 686 063 and US 5 397 501 (Unilever). A preferred example is the imido peroxy-carboxylic class of peracids  
10 described in EP A 325 288, EP A 349 940, DE 382 3172 and EP 325 289. A particularly preferred example is phthalimido peroxy caproic acid (PAP). Such peracids are suitably present at 0.1 - 12%, preferably 0.5 - 10%.

A bleach stabiliser (transition metal sequestrant) may also be present. Suitable  
15 bleach stabilisers include ethylenediamine tetra-acetate (EDTA), diethylenetriamine pentaacetate (DTPA), the polyphosphonates such as Dequest (Trade Mark), ethylenediamine tetramethylene phosphonate (EDTMP) and diethylenetriamine pentamethylene phosphate (DETPMP) and non-phosphate stabilisers such as EDDS (ethylene diamine disuccinate). These bleach stabilisers are also useful for  
20 stain removal especially in products containing low levels of bleaching species or no bleaching species.

An especially preferred bleach system comprises a peroxy bleach compound (preferably sodium percarbonate optionally together with a bleach activator), and a  
25 transition metal bleach catalyst as described and claimed in EP 458 397A, EP 458 398A and EP 509 787A (Unilever).

The compositions according to the invention may also contain one or more enzyme(s). Suitable enzymes include the proteases, amylases, cellulases,  
30 oxidases, peroxidases and lipases usable for incorporation in detergent compositions. Preferred proteolytic enzymes (proteases) are, catalytically active



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protein materials which degrade or alter protein types of stains when present as in fabric stains in a hydrolysis reaction. They may be of any suitable origin, such as vegetable, animal, bacterial or yeast origin.

- 5 Proteolytic enzymes or proteases of various qualities and origins and having activity in various pH ranges of from 4-12 are available and can be used in the instant invention. Examples of suitable proteolytic enzymes are the subtilins which are obtained from particular strains of B. Subtilis B. licheniformis, such as the commercially available subtilisins Maxatase (Trade Mark), as supplied by Gist  
10 Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark.

Particularly suitable is a protease obtained from a strain of Bacillus having maximum activity throughout the pH range of 8-12, being commercially available,  
15 e.g. from Novo Industri A/S under the registered trade-names Esperase (Trade Mark) and Savinase (Trade-Mark). The preparation of these and analogous enzymes is described in GB 1 243 785. Other commercial proteases are Kazusase (Trade Mark obtainable from Showa-Denko of Japan), Optimase (Trade Mark from Miles Kali-Chemie, Hannover, West Germany), and Superase (Trade Mark  
20 obtainable from Pfizer of U.S.A.).

Detergency enzymes are commonly employed in granular form in amounts of from about 0.1 to about 3.0 wt%. However, any suitable physical form of enzyme may be used.

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The compositions of the invention may contain alkali metal, preferably sodium, carbonate, in order to increase detergency and ease processing. Sodium carbonate may suitably be present in amounts ranging from 1 to 60 wt%, preferably from 2 to 40 wt%. However, compositions containing little or no sodium  
30 carbonate are also within the scope of the invention.

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Powder flow may be improved by the incorporation of a small amount of a powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate copolymer, or sodium silicate. One preferred powder structurant is fatty acid soap, suitably present in an amount of from 1 to 5 wt%. The amount of  
5 sodium silicate may suitably range from 0.1 to 5 wt%.

Other materials that may be present in detergent compositions of the invention include sodium silicate; antiredeposition agents such as cellulosic polymers; soil release polymers; inorganic salts such as sodium sulfate; lather control agents or  
10 lather boosters as appropriate; proteolytic and lipolytic enzymes; dyes; coloured speckles; perfumes; foam controllers; fluorescers and decoupling polymers. This list is not intended to be exhaustive.

The detergent composition when diluted in the wash liquor (during a typical wash  
15 cycle) will typically give a pH of the wash liquor from 7 to 10.5 for a main wash detergent.

Particulate detergent compositions are suitably prepared by spray-drying a slurry of compatible heat-insensitive ingredients, and then spraying on or post-dosing  
20 those ingredients unsuitable for processing via the slurry. The skilled detergent formulator will have no difficulty in deciding which ingredients should be included in the slurry and which should not.

Particulate detergent compositions of the invention preferably have a bulk density  
25 of at least 400 g/litre, more preferably at least 500 g/litre. Especially preferred compositions have bulk densities of at least 650 g/litre, more preferably at least 700 g/litre.

Such powders may be prepared either by post-tower densification of spray-dried  
30 powder, or by wholly non-tower methods such as dry mixing and granulation; in

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both cases a high-speed mixer/granulator may advantageously be used. Processes using high-speed mixer/granulators are disclosed, for example, in EP 340 013A, EP 367 339A, EP 390 251A and EP 420 317A (Unilever).

- 5 The micro-powders of the invention are particularly well suited to incorporation into detergent powders.

Liquid detergent compositions can be prepared by admixing the essential and optional ingredients thereof in any desired order to provide compositions  
10 containing components in the requisite concentrations. Liquid compositions according to the present invention can also be in compact form which means it will contain a lower level of water compared to a conventional liquid detergent.

The fabric treatment compositions of the invention can also contain adjuvants that  
15 are normal in the cosmetic, pharmaceutical and/or dermatological field, such as hydrophilic or lipophilic gelling agents, hydrophilic or lipophilic active agents, preserving agents, antioxidants, solvents, fragrances, fillers, screening agents, bactericides, odour absorbers, photobleaches (singlet oxygen or radical type) and dyestuffs. The amounts of these various adjuvants are those conventionally used  
20 in the field under consideration and are, for example, from 0.01 to 20% of the total weight of the composition. Examples of suitable biocides for use in the present invention include Proxel (1,2-benzisothiazolin-3-one), available from, for example, Univar, Avecia and Uniqema; and Kathon CG (Methylchloroisothiazolinone and Methylisothiazolinone), available from Rhom and Haas.

25

### **Substrate**

When used in laundering, the substrate may be any substrate onto which it is desirable to deposit the botanical particles and which is subjected to treatment  
30 such as a washing process.

In particular, the substrate may be a textile fabric and preferably comprises cotton, polyester and polycotton.

### **Treatment**

5

The treatment of the substrate with the composition of the invention can be made by any suitable method such as washing, soaking or rinsing of the substrate but also by direct application such as spraying, rubbing, spotting, smearing, etc.

- 10 The treatment may involve contacting the substrate with an aqueous medium comprising the material of the invention.

The treatment may be provided as a spray composition e.g., for domestic (or industrial) application to fabric in a treatment separate from a conventional  
15 domestic laundering process. Suitable spray dispensing devices are disclosed in WO 96/15310 (Procter & Gamble) and are incorporated herein by reference.

The compositions of the invention may be used to provide a perfume and/or odour benefit, for example a reduction in malodour, to fabric during a laundering  
20 process.

### **Examples**

Embodiments of the invention are now illustrated with reference to the following  
25 non-limiting examples. Unless stated otherwise, all proportions are given in weight percent by weight of the total composition.

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Example 1: Preparation of powders for use in the invention

**(a) Centrifugal milling**

5 Plant powders having a particle size within the claimed range were prepared using a Retsch ZM1000 Ultra Centrifugal Mill operating at a speed of 10,000 rpm. The mill was fitted with a 24 tooth rotor and a 0.12 mm screen.

To avoid heating of the materials during milling, they were co-mixed with dry-ice  
10 before being fed into the mill.

**(b) Size fractionation**

The fine powders from the centrifugal milling were then separated into different  
15 size fractions using Endecotts Ltd (England) Test Sieve.

Sieves of different aperture sizes were stacked on top of each other and 10 g of the powder placed on the top sieve and the sieve stack placed on a Retsch AS200 Sieve Shaker to allow the powder separation into different size fractions.  
20 The Retsch controls were set to shake for 5 minutes with 10 second vibration intervals at an amplitude of 1.5 mm. The sieve stack was removed and manually tapped a few times between the intervals.

Some aromatic botanical powders became heavily agglomerated during the  
25 milling process. This made size fractionation by shaking impractical. However the powder could be brushed through the sieves with ease to allow separation into different size ranges.

Table 1 gives two examples of the size fractions that remained on each sieve from  
30 10g of the starting powder.

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Table 1: Weight fractions remaining on the sieve from 10g of starting powder after 30 minutes of shaking (for ginger) and brushing (for cardamom).

Sieve aperture size ( $\mu\text{m}$ )	Ginger (g)	Cardamom (g)
180	1.75	0.34
125	1.23	0.55
90	1.56	1.22
75	0.93	0.93
63	1.39	2.19
45	1.91	3.25
38	0.68	0.76
< 38	0.3	0.0
total retrieved (g)	9.75	9.24

- 5 In all the following particle deposition experiments the < 45  $\mu\text{m}$  size fractions were used.

Example 2: Detergent compositions 1 to 4 comprising herbs and spices, according to the invention

10

Table 2 shows liquid detergent formulations in accordance with the invention. All concentrations are based on 100% ingredient.

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Composition	1	2	3	4
Nonionic 7EO, branched (100%)	8.50	3.00	3.00	2.66
Fatty acid 7908 (100%)	1.00			
Fatty acid 5908 (100%)		2.30	2.30	2.30
LAS acid (97%)		8.00	8.00	7.10
SLES 3EO (70%)	8.50	3.00	3.00	2.66
Citric acid		3.00	2.80	2.80
NaCl (solution) - PS (26%)	2.00	2.00	2.10	2.10
NaOH solution (50%)	0.27	3.30	3.30	3.30
(Proxel) 1,2-benzisothiazolin-3-one (20%)	0.016	0.016	0.016	0.016
Boric acid (100%)	0.50			
Calcium Chloride		0.10	0.10	0.10
Tinopal CBS-X	0.020	0.020	0.020	0.020
DTPMPA (Na7-Dequest 2066, 32 %)	0.700	1.000	1.000	1.000
Relase Ultra 16L EX	0.39			
Savinase 16L Ultra		0.38	0.36	0.36
Stainzyme 12L		0.10	0.10	0.10
Perfume	0.39	0.35	0.35	0.35
Aztec Spice*	2.50	0.00	0.00	0.00
Harmony*		2.00		
Fruiteria*			3.00	
Citrus orchid*				2.50
Wirral Water	70.21	65.43	63.55	64.63

\* Aztec Spice = Cocoa shells 60%, Cinnamon 5%, Vanilla beans 20%, Cloves 5%,  
Ginger 10%.

Fruiteria = Apple pomace 20%, Lemon grass 5%, Orange peel 20%, Lemon peel  
20%, Blackberry leaves 15%, Ginger 10%, Mandarin peel 10%.

Harmony = Lemon balm leaves 30%, Chamomile 30%, Sage leaves 20%, Lime  
10%, Lemon myrtle 5%, Red Clover 5%.

Citrus orchid = Orange peel 40%, Lemon peel 20%, Mandarin peel 15%, Lime  
peel 15%, Lavendar 10%.

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Example 3: Evaluation of deposition of powders onto fabrics from compositions 1 to 4

To evaluate the deposition of particles from the wash a Linitester was used, which  
5 simulates the tumbling action of an automatic washing machine, on a smaller scale of 100 ml.

The level of deposition of the aromatic particles from a wash solution can be quantified by measuring the turbidity of transmission of the wash solution before  
10 and after the solution is contacted with the fabrics. To simulate the conditions of the wash environment the following procedure was observed:-

A surfactant solution comprising a mixture of anionic and nonionic surfactant (5g of Synperonic A7 and 5.5g of neutralized LAS (90% active) in 1 litre of water) was  
15 prepared.

A buffer solution (2.42g  $\text{CaHCO}_3$  and 7.55g of  $\text{Ca}_2\text{CO}_3$  in a litre of water) was also prepared.

20 Unlike the full detergent compositions of Table 2, the above surfactant/buffer solution remains transparent under dilution for the simulated wash experiment and hence enables turbidity measurement originating from the botanical powders only.

Finally, aqueous solutions of three different deposition polymers were prepared,  
25 namely Softgel BDA, locust bean gum (LBG) and Ucare JR-30M. These polymer solutions were prepared at 0.1% and 2% respectively and from these solutions appropriate levels dosed into the Linitester.

Softgel BDA (Avebe) is a cationic potato starch with low, 0.05%, degree of  
30 substitution (cationic charge). Ucare polymer JR-30M (Amerchol) is a cationic hydroxyethyl cellulose with a 1.5-2.2% degree of substitution. LBG (Sigma) is a



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locust bean gum polysaccharide and has no charge but capable of cellulose recognition.

The following process was carried out:-

5

1. The surfactant solution (10 ml) and buffer solution (10 ml) were added to a Linitester pot. Water (60 ml) was then added along with the polymer solution and the botanical particles (0.015g). Finally, further water was added to a total of 100 g total solution.

10

2. The Linitester temperature was set to 40°C.

3. After 5 minutes (i.e. before deposition) a 5 ml sample was taken for light transmission measurement.

4. A 20 x 20 cm<sup>2</sup> fabric monitor was added to the pot.

5. The Linitester was then run for 20 minutes.

15

6. At end of run, the fabric was removed, spun and line dried.

7. A second 5 ml sample (i.e. after deposition) was withdrawn for transmission measurement.

8. The samples were analyzed on a spectrometer for percent transmission at 560 nm.

20

Note: a blank containing the surfactant solution, the monitor and the polymer solution was run and used as a blank for the transmission measurements. In this way a more accurate estimate of the deposition could be obtained.

25

Table 3 below summarizes the deposition from the detergent wash solution for mint particles < 45 µm in the presence of Softgel BDA. Deposition is expressed as a percentage of the total amount of particles in the composition.

30

To convert the % transmission to % deposition, a calibration curve was obtained for % transmission as a function of micropowder concentration.

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Table 3: Deposition of mint particles onto cotton, polycotton and polyester in the presence of Softgel BDA, Locust Bean Gum and Polymer JR.

Polymer	Transmission % before deposition	Transmission % after deposition	level of deposition %
<b>Softgel (wt%)</b>	<b>Cotton</b>		
0.00 (control)	36.4	66.0	37.1
0.0005	44.2	95.9	92.4
0.0010	43.1	99.2	98.6
0.0020	40.1	96.3	93.2
<b>LBG (wt%)</b>			
0.0010	34.2	84.0	70.4
0.0020	30.5	86.6	75.3
<b>JR (wt%)</b>			
0.0010	40.2	74.2	52.3
0.0020	40.1	80.3	63.6
<b>Softgel (wt%)</b>	<b>Polycotton</b>		
0.00 (control)	36.4	58.2	22.7
0.0005	44.2	88.5	78.7
0.0010	43.1	93.9	88.8
0.0020	40.1	96.5	93.6
<b>Softgel (wt%)</b>	<b>Polyester</b>		
0.00 (control)	36.4	44.0	8.9
0.0005	44.2	79.5	62.0
0.0010	43.1	77.8	58.9
0.0020	40.1	81.0	64.9

- 5 The controls (zero polymer levels in the wash solution) for the three fabric types indicate the level of deposition that is achieved by the filtration mechanism.

The results show that high levels of deposition of particles of botanical origin can be delivered onto fabrics from the wash, using a deposition aid polymer.

10

It is surprising that Softgel BDA with its low cationic charge density of only 0.05% works so well in an anionic detergent solution for example compared to polymer JR with about 2% charge density.

**CLAIMS**

1. A fabric cleaning composition for use in a laundering process which comprises:

5

- a) at least one anionic surfactant,
- b) at least one nonionic surfactant,
- c) a powder of botanical origin, and
- d) a polymeric deposition aid at a level of from 0.0001 to 2% by total weight of the composition,

10

wherein the powder of botanical origin has a particle size of from 0.1 to 100  $\mu\text{m}$ , preferably from 0.5 to 50 and most preferably from 1 to 45  $\mu\text{m}$ .

- 15 2. A composition according to claim 1, wherein the powder of botanical origin is selected from tubers, fruits, flowers, flower parts (calyx, stamen, etc), seeds, beans, pods, pith, barks, stalks, stems, leaves, roots, rhizomes, husks, skins, rinds and mixtures thereof.

- 20 3. A composition according to claim 2, wherein the powder is selected from vanilla bean powder, ginger powder, cinnamon powder, cocoa powder, clove powder, cardamom powder, coriander powder, cumin powder, caraway powder, dill powder, chili powder, cyanine powder, turmeric powder, coffee bean powder, tea leaf powder, tea-tree powder, blackberry leaf powder, Lemon balm leaf powder, herb powders, chamomile powder, fennel powder, lime rind powder, lemon rind powder, grapefruit rind powder, tangerine rind powder, mandarin rind powder, lemon grass powder, apple pomace powder, Lemon myrtle powder, red clover powder, liquorice powder and mixtures thereof.

30

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4. A composition according to claim 3, wherein the herbs are selected from rosemary, thyme, sweet bay, sage, tarragon, mint, basil, balm, lemon verbena, red bergamot, marjoram, lavender and oregano.
- 5 5. A composition according to claim 1 or claim 2, wherein the powder is selected from ginseng powder, rice husk flour, wheat husk flour, buckwheat husk flour and mixtures thereof.
- 10 6. A composition according to any preceding claim, wherein the powder of botanical origin is present in an amount of from 0.1 to 10 wt %, preferably from 0.3 to 5 wt %, most preferably from 0.5 to 3 wt % by weight of the total composition.
- 15 7. A composition according to any preceding claim, wherein the botanical powder is in native state, aged, matured, fermented or roasted.
8. A composition according to any preceding claim in which the polymeric deposition agent is a modified natural polymer or a synthetic polymer.
- 20 9. A composition according to claim 8, wherein the modified natural polymer is selected from the group consisting of cationic starches, cationic guar, cationic cellulose, and nonionic locust bean gums (LBGs).
- 25 10. A composition according to claim 9, wherein the cationic starch is a cationically modified potato starch.
- 30 11. A composition according to claim 8, wherein the synthetic polymer is selected from the group consisting of polyethylene oxide (PEO), polyethylene imine (PEI), poly (acrylate), poly (acrylamide), polyethylene terephthalate-polyoxyethylene terephthalate (PET/POET) polymers and mixtures thereof.

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12. A process for treating fabric comprising the step of treating a fabric article with a composition as defined in any preceding claim.
- 5 13. A fabric article comprising particles, resulting from the treatment process of claim 12.
- 10 14. A method of preparing a composition as defined by any one of claims 1 to 11, comprising the step of pulverizing the powder of botanical origin to a particle size of from 0.1 to 100  $\mu\text{m}$ , preferably from 0.5 to 50 and most preferably from 1 to 45  $\mu\text{m}$ .
- 15 15. Use of a composition as defined in any preceding claim to provide a perfume and/or odour benefit to fabric during a laundering process.

# INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2009/065887

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> INV. C11D3/382 C11D3/37 C11D3/22		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b>		
Minimum documentation searched (classification system followed by classification symbols) C11D		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 840 669 A (NEELAKANTAN KAMESWARAN [IN]) 24 November 1998 (1998-11-24) column 1, line 29 - column 2, line 34 column 2, lines 58-60 claims; examples	13
A	US 5 898 026 A (YIANAKOPOULOS GEORGES [BE] ET AL) 27 April 1999 (1999-04-27) claims; examples	1-15
A	US 2004/110652 A1 (HAFKAMP RUDOLFUS JOHANNES HEND [NL] ET AL) 10 June 2004 (2004-06-10) paragraphs [0011], [0040]; claims; examples	1-15
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<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C.		
<input checked="" type="checkbox"/> See patent family annex.		
* Special categories of cited documents :		
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	
"E" earlier document but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone	
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.	
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family	
"P" document published prior to the international filing date but later than the priority date claimed		
Date of the actual completion of the international search  <div style="text-align: center; font-weight: bold;">11 February 2010</div>	Date of mailing of the international search report  <div style="text-align: center; font-weight: bold;">23/02/2010</div>	
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer  <div style="text-align: center; font-weight: bold;">Péntek, Eric</div>	

**INTERNATIONAL SEARCH REPORT**

International application No  
PCT/EP2009/065887

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>WO 2007/147698 A (UNILEVER PLC [GB]; UNILEVER NV [NL]; UNILEVER HINDUSTAN [IN]) 27 December 2007 (2007-12-27) page 4, line 6 - page 8, line 2; claims; examples</p> <p align="center">-----</p>	1-15
A	<p>US 2002/119904 A1 (BOSKAMP JELLES VINCENT [NL] ET AL) 29 August 2002 (2002-08-29) paragraphs [0001], [0044]; claims; examples</p> <p align="center">-----</p>	1-15

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Information on patent family members

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