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(54) Title: IMPROVEMENTS RELATING TO BENEFIT AGENT DELIVERY

(57) **Abstract:** The invention provides particles comprising a waxy solid and a polymeric deposition aid having no overall cationic charge, wherein the polymeric deposition aid is partially embedded in the waxy solid. These are intended for use in laundry treatment composition and preferably have a fabric benefit agent dispersed in, or dissolved in, the waxy solid. Preferably the waxy solid comprises at least one of: silicone waxes, hydrocarbon waxes or sucrose polyester materials. Preferably the deposition aid comprises at least one of: non-hydrolysable cotton-substantive polymer, hydrolysable cotton-substantive polymer or polyester-substantive polymer. In a particularly preferred embodiment the polymeric deposition aid comprises at least one of: a) tamarind gum, guar gum, locust bean gum, b) gums derived from Tara, Fenugreek, Aloe, Chia, Flaxseed, Psyllium seed, quince seed, or, c) xanthan, gellan, welan, rhamsan, dextran, curdlan, pullulan, scleroglucan, schizophyllan, chitin, hydroxyalkyl cellulose, arabinan, de-branched arabinan, arabinoxylan, galactan, pectic galactan, galactomannan, glucomannan, lichenan, mannan, pachyman, rhamnogalacturonan, acacia gum, agar, alginates, carrageenan, chitosan, clavan, hyaluronic acid, heparin, inulin, cellodextrins, cellulose and derivatives thereof, or, d) is a phthalate containing polymer.



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IMPROVEMENTS RELATING TO BENEFIT AGENT DELIVERY**Technical Field**

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The present invention is concerned with the delivery of benefit agents, particularly from a laundry composition.

10 **Background to the Invention**

Perfumes, silicones and other “benefit agents” are important and expensive components of laundry detergent compositions. While the present invention will be described with particular reference to perfumes and silicones it is applicable to
15 other benefit agents.

It is important to ensure that perfumes are not lost during processing or on storage of, for example, laundry detergent compositions and that they deposit onto articles being washed. Being volatile, perfumes are prone to evaporate from a
20 composition, especially if the composition is subjected to elevated temperatures. Methods for deposition of perfume components and other benefit agents from laundry compositions are diverse and perfume is often incorporated into a carrier or other delivery system. Carrier systems for perfumes are typically based on encapsulation or entrapment of the perfume within a matrix. The perfume may
25 simply be emulsified but deposition onto a substrate, such as fabric, is often inefficient and problems with poor retention or stability may be found. Preparation of materials by diffusion of the perfume into a carrier can suffer from complex preparation, long times of diffusion, poor retention of the perfume in the matrix and subsequent poor substrate deposition.

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Commercially available perfume encapsulates include those based on aminoplast materials and the patent literature states that these may be surface modified with materials to improve deposition on surfaces such as textiles. These capsules can be difficult to prepare, and as they are formed by reactive chemistry, some care
5 must be taken to avoid unwanted side-reactions. Materials for surface modification include neutral polymers such as locust bean gum (LBG) and cationic materials such as cationic guar gums. WO 07/62833, for example, relates to compositions which comprise core-shell encapsulated perfume particles decorated with locust bean gum. Some slightly anionic materials such as
10 sulphonated PET polymers have also been suggested as suitable materials.

Many known approaches involve the poly-condensation of small molecules to form polymeric encapsulates. WO 2008/145547 discloses a process for the manufacture of core-shell particles by emulsion polymerisation. WO 2007/062733
15 discloses the polymerisation of a polymer/perfume latex in the presence of LBG. GB 2432851 discloses polymerised particles containing sugar polyester as a benefit agent and a deposition aid such as LBG: similar particles are disclosed in EP 1741775. WO 03/014278 relates in general terms to the principle of using hydrolysable polysaccharide ethers as deposition aids.

20 WO 2008/143862 and WO 2008/127766 relate to an emulsion of an unencapsulated benefit agent (which can be a silicone) which is associated with a cationic deposition aid.

25 EP 1479376 discloses a method of improving the deposition of a hydrophilic benefit agent from a personal product composition which comprises mixing the benefit agent with a molten structuring agent (which can be a wax) and then cooling.

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WO 2009/042197: discloses a thickening system which includes both a hydrated thickening silica, a polyol and a water soluble/swellable polymer (which may be xanthan gum).

- 5 US 6162890: discloses further compositions which comprise both a "Carbowax" materials (a PEG 1000) and a phthalate polymer.

EP 0324042: discloses a process which uses both "kettle wax" and CMC (see example 10). More generally this reference discloses a phthalate based polyester
10 being used in combination with a colloidal silica.

WO 2006/101866: discloses products (see page 22) which contain glycerin, "Candelilla Wax" and Xanthan gum.

- 15 US 6686327: (see example 1 and 5) discloses a process in which a mixture is formed which comprises paraffin wax and some polymers which are put through a range of processes for making detergent antifoam particles.

20 **Brief Description of the Invention**

We have now determined that good deposition can be obtained if particles are formed of a waxy solid which has been surface-modified with a polymeric deposition aid that does not have an overall cationic charge. These particles can
25 be formed by allowing the polymeric deposition aid to interact with an emulsion in which the waxy solid, in molten form, comprises the dispersed phase. When the wax is allowed to solidify, the polymeric deposition aid becomes trapped at the surface of the particles.

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In the context of the present invention, a 'waxy solid' is a water-insoluble material which has a melting point in the range 30-150 Celsius. In the context of the present invention, a polymeric deposition aid is a polymer which comprises at least one region that is substantive to a substrate.

5

Accordingly, the present invention provides particles comprising a waxy solid and a polymeric deposition aid having no overall cationic charge, wherein the polymeric deposition aid is partially embedded in the waxy solid.

10 According to the further aspect of the present invention there is provided a process for the preparation of particles of a waxy solid with partially embedded polymeric deposition aid wherein the polymeric deposition aid is contacted with an emulsion of which the waxy solid, in molten form, comprises the dispersed phase.

15 The waxy solid can itself be a benefit agent if it is, for example, a silicone wax which provides a lubrication benefit. In the alternative, or additionally, further benefit agents, for example perfume, may be dispersed in, or dissolved in, the waxy solid.

20 It is particularly preferred that, for the embodiments as liquids laundry detergent compositions, the dosage in which the composition is applied is such that the wash liquor produced has a surfactant (other than soap) concentration of less than 1 g/L, more preferably less than 0.5 g/L. This is a low level of surfactant as compared with conventional wash liquors. It is also preferred that the dosage of a
25 liquid detergent product containing the cue is less than 30ml and preferably less than 25ml. This is a low dosage as compared with conventional doses of laundry compositions. Such low dosage and/or low surfactant products have environmental advantages in that the products require transport of smaller quantities of material and have a reduced usage of surfactants. Such
30 formulations have comparatively little space for the incorporation of benefit agents

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and therefore means for effective delivery of benefit agents are particularly advantageous.

5 **Detailed Description of the Invention**

In order that the invention may be better understood it will be further described below with reference to preferred features.

10 • **Preferred Waxy Solids**

Preferred waxy solids with a melting point in the lower half of the 30-150 Celsius range will show, in the extreme cases, controlled release of materials through contact with the skin and exhibit good spreading. Those waxy solids with melting
15 points in the upper half of the range will generally exhibit better storage.

Preferably the melting point will lie in the range 30-90 Celsius as this will facilitate production of the surface modified particles in heated aqueous emulsions without the use of pressure.

20

Suitable waxy solids include silicone waxes, hydrocarbon waxes and sucrose polyester materials. Mixtures of the same may be employed.

Typically, the particles comprising the waxy solid will comprise 0.2-3g per dose of
25 a detergent product.

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- **Preferred Polymeric Deposition Aids**

Preferred polymers are selected from non-hydrolysable cotton-substantive polymers, hydrolysable cotton-substantive polymers and polyester-substantive polymers.

Preferred polysaccharide polymers, whether hydrolysable or not may be derived from a broad range of polysaccharides. Preferably, the polysaccharide is selected from the group consisting of: tamarind gum (preferably consisting of xyloglucan polymers), guar gum, locust bean gum (preferably consisting of galactomannan polymers), and other industrial gums and polymers, which include, but are not limited to, Tara, Fenugreek, Aloe, Chia, Flaxseed, Psyllium seed, quince seed, xanthan, gellan, welan, rhamsan, dextran, curdlan, pullulan, scleroglucan, schizophyllan, chitin, hydroxyalkyl cellulose, arabinan (preferably from sugar beets), de- branched arabinan (preferably from sugar beets), arabinoxylan (preferably from rye and wheat flour), galactan (preferably from lupin and potatoes), pectic galactan (preferably from potatoes), galactomannan (preferably from carob, and including both low and high viscosities), glucomannan, lichenan (preferably from icelandic moss), mannan (preferably from ivory nuts), pachyman, rhamnogalacturonan, acacia gum, agar, alginates, carrageenan, chitosan, clavan, hyaluronic acid, heparin, inulin, cellodextrins, cellulose, cellulose derivatives and mixtures thereof.

Preferred non-hydrolysable cotton-substantive deposition aids include non-hydrolysable polysaccharides. The polysaccharide preferably has a β -1,4-linked backbone.

Preferably the polysaccharide is a cellulose, a cellulose derivative, or another β -1,4-linked polysaccharide having an affinity for cellulose, such as polymannan, polyglucan, polyglucomannan, polyxyloglucan and polygalactomannan or a

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mixture thereof. More preferably, the polysaccharide is selected from the group consisting of polyxyloglucan and polygalactomannan. For example, preferred polysaccharides are locust bean gum, tamarind xyloglucan, guar gum or mixtures thereof. Most preferably, the deposition aid is locust bean gum.

5

Preferred hydrolysable cotton-substantive deposition aids include hydrolysable polysaccharides. These comprise a polysaccharide which has been modified to render it more water soluble by means of a group covalently attached to the polysaccharide by means of hydrolysable bond. Preferred groups may for

10 example be independently selected from one or more of acetate, propanoate, trifluoroacetate, 2- (2-hydroxy-1-oxopropoxy) propanoate, lactate, glycolate, pyruvate, crotonate, isovalerate cinnamate, formate, salicylate, carbamate, methylcarbamate, benzoate, gluconate, methanesulphonate, toluene, sulphonate, groups and hemiester groups of fumaric, malonic, itaconic, oxalic, maleic,
15 succinic, tartaric, aspartic, glutamic, and malic acids.

Preferred amongst such hydrolysable deposition aids is cellulose mono acetate.

Most preferably the polyester-substantive deposition aid is a phthalate containing
20 polymer, more preferably a polymer having one or more nonionic hydrophilic components comprising oxyethylene, polyoxyethylene, oxypropylene or polyoxypropylene segments, and, one or more hydrophobic components comprising terephthalate segments. Typically, oxyalkylene segments of these deposition aids will have a degree of polymerization of from 1 to about 400,
25 although higher levels can be used, preferably from 100 to about 350, more preferably from 200 to about 300.

One type of preferred deposition aid is a copolymer having random blocks of ethylene terephthalate and polyethylene oxide terephthalate.

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Another preferred polymeric deposition aid is polyester with repeat units of ethylene terephthalate units contains 10-15% by weight of ethylene terephthalate units together with 90-80% by weight of polyoxyethylene terephthalate units, derived from a polyethylene glycol of average molecular weight 0.2kD-40kD.

5 Examples of this class of polymer include the commercially available material ZELCON 5126 (from DuPont) and MILEASE T (from ICI). Examples of related polymers can be found in US 4702857.

Another preferred polymeric deposition aid is a sulfonated product of a
10 substantially linear ester oligomer comprised of an oligomeric ester backbone of terephthaloyl and oxyalkyleneoxy repeat units and terminal moieties covalently attached to the backbone. These soil release agents are described fully in US 4968451. Other suitable polymeric soil release agents include the terephthalate polyesters of US 4711730, the anionic end-capped oligomeric esters
15 of US 4721580, and the block polyester oligomeric compounds of US 4702857.

Preferred polymeric deposition aids also include the soil release agents of U.S. 4877896 which discloses anionic, especially sulfoaroyl, end-capped terephthalate esters.

20

Still another preferred deposition aid is an oligomer with repeat units of terephthaloyl units, sulfoisoterephthaloyl units, oxyethyleneoxy and oxy-1,2-propylene units. The repeat units form the backbone of the oligomer and are preferably terminated with modified isethionate end-caps. A particularly preferred
25 deposition aid of this type comprises about one sulfoisophthaloyl unit, 5 terephthaloyl units, oxyethyleneoxy and oxy-1,2-propyleneoxy units in a ratio of from about 1.7 to about 1.8, and two end-cap units of sodium 2-(2-hydroxyethoxy)-ethanesulfonate. Said soil release agent also comprises from about 0.5% to about 20%, by weight of the oligomer, of a crystalline-reducing

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stabilizer, preferably selected from the group consisting of xylene sulfonate, cumene sulfonate, toluene sulfonate, and mixtures thereof.

The deposition aid may be straight or branched.

5

The deposition aid may be a block copolymer comprising at least two regions which differ in their hydrophobic/hydrophilic nature. Examples of the same include locust bean gum with silicone grafted thereto. Advantageously, this provides a portion of the molecule (the silicone) which facilitates adsorption onto the waxy-
10 solid particle and a deposition promoting portion.

The preferred molecular weight of the polymeric deposition aid is in the range of from about 5kD to about 500kD, preferably 10kD-500kD, more preferably 20kD-300kD.

15

Preferably, the polymer is present at levels such that the ratio polymer:waxy solid is in the range 1:50-3:1, preferably 1:20-1:3.

20

While the polymer will often be a sufficient emulsifier for the waxy solid when molten, it will in some embodiments be advantageous to add a co-emulsifier for the waxy solid. Suitable co-emulsifiers include nonionic surfactants, typically fatty (C10-C16) alcohols ethoxylated with 10-20 moles of ethylene oxide, alkyl polyglucosides and n=1-3 sucrose fatty acid emulsifiers.

25

- Preferred Particle Sizes

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The preferred average particle sizes for the waxy solid particles (as measured by Dynamic Light Scattering and Scanning Electron Microscopy) is between 50 microns and 20nm. Preferred particle sizes are determined by the precise application. Where the benefit agent is carried in the waxy solid preferred

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average particle sizes are 1-25 microns, preferably 3-10 microns. Where the waxy solid is itself the benefit agent, average particle sizes will generally be in the range 40-800nm.

5 • Benefit Agents

A variety of benefit agents can be incorporated using this invention. Preferred benefit agents include, but not limited to, the following:

- 10 a) silicone oils, resins, and modifications thereof such as linear and cyclic polydimethylsiloxanes, amino-modified, allcyl, aryl, and alkylaryl silicone oils,
- 15 b) perfume components including fragrance, perfumery, and essential oils and resins (both free and encapsulated);
- 20 c) organic sunscreen actives, for example, octylmethoxy cinnamate;
- 25 d) antimicrobial agents, for example, 2-hydroxy-4,2,4- trichlorodiphenylether;
- 20 e) ester solvents; for example, isopropyl myristate;
- 25 f) lipids and lipid like substance, for example, cholesterol;
- 25 g) hydrocarbons such as paraffins, petrolatum, and mineral oil
- 25 h) fish and vegetable oils
- 25 i) hydrophobic plant extracts;

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- j) pigments including inorganic compounds with hydrophobically-modified surface and/or dispersed in an oil or a hydrophobic liquid.

The functionality of the benefit agent is preferably at least one of that of a
5 perfume, enzyme, antifoam, shading dye and/or pigment, detergency builder, fabric conditioning agent (for example water-insoluble quaternary ammonium materials and/or silicones), sunscreen, antioxidant, reducing agent, sequestrant, colour care additive, density matching polymer, photo-bleach, emollient and/or antimicrobial agent.

10

Preferred benefit agents include perfumes, lubricants any other oily materials. The most preferred benefit agents are perfume components. Perfume components include both odiferous materials and pro-fragrance materials.

- 15 The pro-fragrance can, for example, be a food lipid. Food lipids typically contain structural units with pronounced hydrophobicity. The majority of lipids are derived from fatty acids. In these 'acyl' lipids the fatty acids are predominantly present as esters and include mono-, di-, triacyl glycerols, phospholipids, glycolipids, diol lipids, waxes, sterol esters and tocopherols. In their natural state, plant lipids
20 comprise antioxidants to prevent their oxidation. While these may be at least in part removed during the isolation of oils from plants some antioxidants may remain. These antioxidants can be pro-fragrances. In particular, the carotenoids and related compounds including vitamin A, retinol, retinal, retinoic acid and provitamin A are capable of being converted into fragrant species including the
25 ionones, damascones and damscenones. Preferred pro-fragrance food lipids include olive oil, palm oil, canola oil, squalene, sunflower seed oil, wheat germ oil, almond oil, coconut oil, grape seed oil, rapeseed oil, castor oil, corn oil, cottonseed oil, safflower oil, groundnut oil, poppy seed oil, palm kernel oil, rice bran oil, sesame oil, soybean oil, pumpkin seed oil, jojoba oil and mustard seed

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oil. Perfume components which are odiferous materials are described in further detail below.

- Perfumes as Benefit Agents

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The perfume is typically present in an amount of from 10-85% by total weight of the particle, preferably from 20 to 75% by total weight of the particle. The perfume suitably has a molecular weight of from 50 to 500.

10 Useful components of the perfume include materials of both natural and synthetic origin. They include single compounds and mixtures. Specific examples of such components may be found in the current literature, e.g., in Fenaroli's Handbook of Flavour Ingredients, 1975, CRC Press; Synthetic Food Adjuncts, 1947 by M. B. Jacobs, edited by Van Nostrand; or Perfume and Flavour Chemicals by
15 S. Arctander 1969, Montclair, N.J. (USA). These substances are well known to the person skilled in the art of perfuming, flavouring, and/or aromatizing consumer products, i.e., of imparting an odour and/or a flavour or taste to a consumer product traditionally perfumed or flavoured, or of modifying the odour and/or taste of said consumer product.

20

By perfume in this context is not only meant a fully formulated product fragrance, but also selected components of that fragrance, particularly those which are prone to loss, such as the so-called 'top notes'.

25 Top notes are defined by Poucher (Journal of the Society of Cosmetic Chemists 6(2):80 [1955]). Examples of well known top-notes include citrus oils, linalool, linalyl acetate, lavender, dihydromyrcenol, rose oxide and cis-3-hexanol. Top notes typically comprise 15-25%wt of a perfume composition and in those embodiments of the invention which contain an increased level of top-notes it is
30 envisaged at that least 20%wt would be present within the particle.

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Typical perfume components which it is advantageous to employ in the embodiments of the present invention include those with a relatively low boiling point, preferably those with a boiling point of less than 300, preferably 100-250 Celsius.

5

It is also advantageous to encapsulate perfume components which have a low LogP (i.e. those which will be partitioned into water), preferably with a LogP of less than 3.0. These materials, of relatively low boiling point and relatively low LogP have been called the "delayed blooming" perfume ingredients and include

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the following materials:

Allyl Caproate, Amyl Acetate, Amyl Propionate, Anisic Aldehyde, Anisole, Benzaldehyde, Benzyl Acetate, Benzyl Acetone, Benzyl Alcohol, Benzyl Formate, Benzyl Iso Valerate, Benzyl Propionate, Beta Gamma Hexenol, Camphor Gum, 15 Laevo-Carvone, d-Carvone, Cinnamic Alcohol, Cinamyl Formate, Cis-Jasmone, cis-3-Hexenyl Acetate, Cuminic Alcohol, Cyclal C, Dimethyl Benzyl Carbinol, Dimethyl Benzyl Carbinol Acetate, Ethyl Acetate, Ethyl Aceto Acetate, Ethyl Amyl Ketone, Ethyl Benzoate, Ethyl Butyrate, Ethyl Hexyl Ketone, Ethyl Phenyl Acetate, Eucalyptol, Eugenol, Fenchyl Acetate, Flor Acetate (tricyclo Decenyl Acetate), 20 Frutene (tricyclo Decenyl Propionate), Geraniol, Hexenol, Hexenyl Acetate, Hexyl Acetate, Hexyl Formate, Hydratropic Alcohol, Hydroxycitronellal, Indone, Isoamyl Alcohol, Iso Menthone, Isopulegyl Acetate, Isoquinolone, Ligustral, Linalool, Linalool Oxide, Linalyl Formate, Menthone, Menthyl Acetphenone, Methyl Amyl Ketone, Methyl Anthranilate, Methyl Benzoate, Methyl Benyl 25 Acetate, Methyl Eugenol, Methyl Heptenone, Methyl Heptine Carbonate, Methyl Heptyl Ketone, Methyl Hexyl Ketone, Methyl Phenyl Carbiny Acetate, Methyl Salicylate, Methyl-N-Methyl Anthranilate, Nerol, Octalactone, Octyl Alcohol, p-Cresol, p-Cresol Methyl Ether, p-Methoxy Acetophenone, p-Methyl Acetophenone, Phenoxy Ethanol, Phenyl Acetaldehyde, Phenyl Ethyl Acetate,

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Phenyl Ethyl Alcohol, Phenyl Ethyl Dimethyl Carbinol, Prenyl Acetate, Propyl Bornate, Pulegone, Rose Oxide, Safrole, 4-Terpinenol, Alpha-Terpinenol, and /or Viridine

5 It is commonplace for a plurality of perfume components to be present in a formulation. In the encapsulates of the present invention it is envisaged that there will be four or more, preferably five or more, more preferably six or more or even seven or more different perfume components from the list given of delayed blooming perfumes given above present in the particles.

10

Another group of perfumes with which the present invention can be applied are the so-called 'aromatherapy' materials. These include many components also used in perfumery, including components of essential oils such as Clary Sage, Eucalyptus, Geranium, Lavender, Mace Extract, Neroli, Nutmeg, Spearmint,

15 Sweet Violet Leaf and Valerian. By means of the present invention these materials can be transferred to textile articles that will be worn or otherwise come into contact with the human body (such as handkerchiefs and bed linen).

- Laundry Treatment Compositions

20

The particles of the invention may be incorporated into laundry compositions. The particles are typically included in said compositions at levels of from 0.001% to 10%, preferably from 0.005% to 5%, most preferably from 0.01% to 3% by weight of the total composition.

25

The active ingredient in the compositions is preferably a surface active agent or a fabric conditioning agent. More than one active ingredient may be included. For some applications a mixture of active ingredients may be used.

- 15 -

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, especially, an aqueous based liquid. In particular the compositions may be used in laundry compositions, especially in liquid, gel, powder or tablet laundry composition.

5

The compositions of the present invention are preferably laundry compositions, especially main wash (fabric washing) compositions or rinse-added softening compositions. The main wash compositions may include a fabric softening agent and the rinse-added fabric softening compositions may include surface-active compounds, particularly non-ionic surface-active compounds.

10

The detergent compositions of the invention may contain a surface-active compound (surfactant) which may be chosen from soap and non soap anionic, cationic, non-ionic, amphoteric and zwitterionic surface active compounds and mixtures thereof. Many suitable surface active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

15

The preferred detergent-active compounds that can be used are soaps and synthetic non soap anionic, and non-ionic compounds.

20

The compositions of the invention may contain linear alkylbenzene sulphonate, particularly linear alkylbenzene sulphonates having an alkyl chain length of from C8 to C15. It is preferred if the level of linear alkylbenzene sulphonate is from 0 wt% to 30 wt%, more preferably from 1 wt% to 25 wt%, most preferably from 2 wt% to 15 wt%, by weight of the total composition.

25

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

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secondary alkyl sulphates, particularly C8 to C15 primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

5

The compositions of the invention may also contain non-ionic surfactant. Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C8 to C20 aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C10 to C15 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).

10

15 It is preferred if the level of non-ionic surfactant is from 0 wt% to 30 wt%, preferably from 1 wt% to 25 wt%, most preferably from 2 wt% to 15 wt%, by weight of the total composition.

Any conventional fabric conditioning agent may be used in the compositions of the present invention. The conditioning agents may be cationic or non-ionic. If the fabric conditioning compound is to be employed in a main wash detergent composition the compound will typically be non-ionic. For use in the rinse phase, typically they will be cationic. They may for example be used in amounts from 0.5% to 35%, preferably from 1% to 30% more preferably from 3% to 25% by weight of the composition.

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Suitable cationic fabric softening compounds are substantially water-insoluble quaternary ammonium materials comprising a single alkyl or alkenyl long chain having an average chain length greater than or equal to C20 or, more preferably, compounds comprising a polar head group and two alkyl or alkenyl chains having

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an average chain length greater than or equal to C14. Preferably the fabric softening compounds have two long chain alkyl or alkenyl chains each having an average chain length greater than or equal to C16. Most preferably at least 50% of the long chain alkyl or alkenyl groups have a chain length of C18 or above. It is preferred if the long chain alkyl or alkenyl groups of the fabric softening compound are predominantly linear.

Quaternary ammonium compounds having two long-chain aliphatic groups, for example, distearyldimethyl ammonium chloride and di(hardened tallow alkyl) dimethyl ammonium chloride, are widely used in commercially available rinse conditioner compositions. Other examples of these cationic compounds are to be found in "Surfactants Science Series" volume 34 ed. Richmond 1990, volume 37 ed. Rubingh 1991 and volume 53 eds. Cross and Singer 1994, Marcel Dekker Inc. New York".

Any of the conventional types of such compounds may be used in the compositions of the present invention.

The fabric softening compounds are preferably compounds that provide excellent softening, and are characterised by a chain melting $L\beta$ to $L\alpha$ transition temperature greater than 25 Celsius, preferably greater than 35 Celsius, most preferably greater than 45 Celsius. This $L\beta$ to $L\alpha$ transition can be measured by differential scanning calorimetry as defined in "Handbook of Lipid Bilayers", D Marsh, CRC Press, Boca Raton, Florida, 1990 (pages 137 and 337).

Substantially water-insoluble fabric softening compounds are defined as fabric softening compounds having a solubility of less than 1×10^{-3} wt% in demineralised water at 20 Celsius. Preferably the fabric softening compounds have a solubility of less than 1×10^{-4} wt%, more preferably from less than 1×10^{-8} to 1×10^{-6} wt%.

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Especially preferred are cationic fabric softening compounds that are water-insoluble quaternary ammonium materials having two C12-22 alkyl or alkenyl groups connected to the molecule via at least one ester link, preferably two ester links. Di(tallowoxyloxyethyl) dimethyl ammonium chloride and/or its hardened
5 tallow analogue is an especially preferred compound of this class.

A second preferred type comprises those derived from triethanolamine (hereinafter referred to as 'TEA quats') as described in for example US 3915867. Suitable materials are, for example, N-methyl-N,N,N-triethanolamine ditallowester
10 or di-hardened-tallowester quaternary ammonium chloride or methosulphate. Examples of commercially available TEA quats include Rewoquat WE18 and Rewoquat WE20, both partially unsaturated (ex. WITCO), Tetranyl AOT-1, fully saturated (ex. KAO) and Stepanex VP 85, fully saturated (ex. Stepan).

15 It is advantageous if the quaternary ammonium material is biologically biodegradable.

It is also possible to include certain mono-alkyl cationic surfactants which can be used in main-wash compositions for fabrics. Cationic surfactants that may be
20 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 R1 is a C8-C22 alkyl group, preferably a C8-C10 or C12-C14 alkyl group, R2 is a methyl group, and R3 and R4, which may be the same or
25 different, are methyl or hydroxyethyl groups); and cationic esters (for example, choline esters).

The choice of surface-active compound (surfactant), and the amount present, will depend on the intended use of the detergent composition. In fabric washing
30 compositions, different surfactant systems may be chosen, as is well known to the

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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, an amount of from 5 to 40 wt% is generally appropriate. Typically the compositions will comprise at least 2 wt% surfactant e.g. 2-60%, preferably 15-40% most preferably 25-35%, by weight of the composition.

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.

The compositions of the invention, when used as main wash fabric washing compositions, will generally also contain one or more detergency builders. The total amount of detergency builder in the compositions will typically range from 5 to 80 wt%, preferably from 10 to 60 wt%, by weight of the compositions.

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 aluminosilicates as disclosed in GB 1 470 250 (Procter & Gamble); and layered 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.

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The compositions of the invention preferably contain an alkali metal, preferably sodium, aluminosilicate builder. Sodium aluminosilicates may generally be incorporated in amounts of from 10 to 70% by weight (anhydrous basis), preferably from 25 to 50 wt%.

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The alkali metal aluminosilicate may be either crystalline or amorphous or mixtures thereof, having the general formula: $0.8 \text{ } 1.5 \text{ Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 0.8 \text{ } 6 \text{ SiO}_2$

10

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 SiO₂ units (in the formula above). Both the amorphous and the 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 and X, and mixtures thereof.

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The zeolite may be the commercially available zeolite 4A now widely used in laundry detergent powders. However, according to a preferred embodiment of the invention, the zeolite builder incorporated in the compositions of the invention is maximum 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 weight 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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Especially preferred is zeolite MAP having a silicon to aluminium weight ratio not exceeding 1.07, more preferably about 1.00. The calcium binding capacity of zeolite MAP is generally at least 150 mg CaO per g of anhydrous material.

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Organic builders that may be present include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono ,
5 di- and trisuccinates, carboxymethyloxy succinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl and alkenylmalonates and succinates; and sulphonated fatty acid salts. This list is not intended to be exhaustive.

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

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

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
20 yielding hydrogen peroxide in aqueous solution.

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

Especially preferred is sodium percarbonate having a protective coating against destabilisation by moisture. Sodium percarbonate having a protective coating

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comprising sodium metaborate and sodium silicate is disclosed in GB 2 123 044B (Kao).

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%.

Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxoanoic acid precursors. Especially preferred bleach precursors suitable for use in the present invention are N,N,N',N', tetracetyl 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 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 peroxycarboxylic class of peracids 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 bleach stabilisers include ethylenediamine tetra-acetate (EDTA), the polyphosphonates such as Dequest (Trade Mark) and non phosphate stabilisers such as EDDS (ethylene diamine di succinic acid). These bleach stabilisers are

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also useful for stain removal especially in products containing low levels of bleaching species or no bleaching species.

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

10 Advantageously in the compositions of the invention benefit agents, particularly, perfume components may be employed which are sensitive to bleaches as the encapsulation of, for example, the perfume component within the waxy solid will provide some degree of protection to the perfume component or other benefit agent.

15 The compositions according to the invention may also contain one or more enzyme(s).

Suitable enzymes include the proteases, amylases, cellulases, oxidases, peroxidases and lipases usable for incorporation in detergent compositions.

20 Preferred proteolytic enzymes (proteases) are, catalytically active 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.

25 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 subtilisins which are obtained from particular strains of *B. Subtilis B. licheniformis*, such as the commercially available subtilisins Maxatase (Trade Mark), as supplied by

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Genencor International N.V., Delft, Holland, and Alcalase (Trade Mark), as supplied by Novozymes Industri A/S, Copenhagen, Denmark.

Particularly suitable is a protease obtained from a strain of *Bacillus* having
5 maximum activity throughout the pH range of 8 - 12, being commercially available, e.g. from Novozymes 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
10 (Trade Mark from Miles Kali Chemie, Hannover, West Germany), and Superase (Trade Mark 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
15 be used. Advantageously in the compositions of the invention benefit agents, for example, perfume components, may be employed which are sensitive to enzymes as the encapsulation of the perfume component (or other benefit agent) within the waxy particles will provide some degree of protection to the perfume component (or other benefit agent).

20

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
25 carbonate are also within the scope of the invention.

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
30 is fatty acid soap, suitably present in an amount of from 1 to 5 wt%.

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Other materials that may be present in detergent compositions of the invention include sodium silicate; antiredeposition agents such as cellulosic polymers; soil release polymers (other than any that might be attached to the benefit agent carrying particles); inorganic salts such as sodium sulphate; or lather boosters as appropriate; dyes; coloured speckles; 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 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 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. It is particularly useful to add the perfume particles of the present invention via post-dosing.

Particulate detergent compositions of the invention preferably have a bulk density 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 powder, or by wholly non tower methods such as dry mixing and granulation; in 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).

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Liquid detergent compositions can be prepared by admixing the essential and optional ingredients thereof in any desired order to provide compositions 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.

In order that the present invention may be further understood and carried forth into practice it will be further described with reference to the following examples:

Examples

Example 1: Preparation of Silicone wax emulsion using PET-POET as emulsifier, and enhancement of fabric properties.

Synthesis of polyethylene terephthalate-co-polyoxyethylene terephthalate (PET-POET #170707)

PET-4900 was a polyethylene terphthalate ($M_n=4,900$); poly (ethylene glycol) 20,000 ($M_n=20,000$) was purchased from Fluka; antimony oxide (Sb_2O_3), calcium acetate and 2, 6-ditert-butyl-4-methylphenol (DBMP) were supplied by Sinopharm Chemical Reagent Co., Ltd. All the reagents were used as received without further purification.

The PET-POET sample was prepared utilising a stainless steel reaction kettle which offers mechanical stirring, fine thermo-controlling and high vacuum level. The reaction kettle was supplied by Weihai Auto-control Reaction Kettle Ltd.

Eighty grams of PEG of 20,000 molecular weight and 5 grams of PET-4900 were used for the transesterification polymerisation. Sb_2O_3 (20mg) and calcium

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acetate (20mg) were utilised as the catalyst and 2,6-ditert-butyl-4-methylphenol (80mg) as anti-oxidant. Before heating the reaction mixture, vacuum was applied to the kettle, followed with re-filling with nitrogen. This process was repeated three times; and then under vacuum level below zero mmHg and mechanical stirring at 50 rpm, the temperature was gradually elevated to the reaction temperature of around 260 degrees centigrade and maintained for 5 hours. Transesterification reaction took place with the release of ethylene glycol. The product was taken out of the kettle with a spatula while it was hot, at about 150°C. The viscous polymer cooled down to a hard solid of Mn= 30,000.

Silicone wax emulsion preparation

Silicone waxes were sourced from Clariant, namely, SilCare 41M65 (Stearyl Dimethicone, softening point near 30°C) and SilCare 41M80 (C24-28 alkyl dimethicone, softening point near 65°C).

Emulsions were prepared with deionised water and 1% PET-POET(#170707) as the continuous phase and 5 % of silicone wax as the disperse phase. For the preparation of emulsions containing silicone wax each of them was melted to about 10°C - 20°C above their melting point. The continuous phase was also heated to 80°C - 90°C to match the temperature (the same temperature) of the disperse phase ensuring that it was not boiling. The disperse phase was then added to the continuous phase and homogenised at 13500 rpm for 5 - 20 minutes using a rotor-stator system (Ultraturrax[®] T25 basic, IKA-WERKE GmbH & Co. KG, Staufen, Germany) to form a coarse emulsion. Homogenisation of the coarse emulsion was carried out in a double sealed beaker connected to a water bath to ensure the temperature was maintained above the melting points of the silicone waxes.

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After homogenisation of the coarse emulsion, it was immediately further homogenised at 1200 bar for approximately 2 cycles using the Microfluidizer[®] M-110S (Microfluidics International Corporation, MA-Newton, USA). Samples were collected in sterile containers and left on the bench for cooling until samples
5 reached room temperature (20°C).

Comparative Example A

A comparative (control) sample without any PET-POET#170707 was prepared
10 using a procedure identical to the above, except that Tween 20 ex. Sigma Aldrich (1% in deionized water) was used to prepare the continuous phase.

Model wash evaluation

15 a) Preparation of wash liquor:

The wash liquor was prepared by diluting Persil Small and Mighty[™] (concentrated liquid detergent) with local medium/soft water (typically 6-18°F) to achieve a 2.5g/l concentration. 50ml of wash liquor was added to each 500ml Linitest pot.

20

b) Simulated Wash (Linitest)

62.5µl of 5% silicone wax emulsified with polyester deposition aid (PET POET#170707) and without (comparative example A) were added to the linitest
25 pots containing wash liquor and agitated slightly to ensure mixing. (Washes were done in quadruplicate for each sample and results averaged).

A sample of knitted polyester measuring 21cm by 21cm was placed into each linitest pot containing the wash liquor and silicone wax emulsions, and the pots
30 were sealed.

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The Linitest™ is a laboratory scale washing machine (Ex. Heraeus). The equipment is designed and built to comply with the requirements for international standard test specifications. It is used for small scale detergency and stain removal testing particularly when low liquor to cloth ratios are required.

5

There are various models of the Linitest commercially available. The model used in this case has a single rotation speed of 40 rpm. The carrier is capable of accommodating twelve 500ml steel containers and can be operated at temperatures up to 100°C.

10

The Linitest comprises a 20 litre tank, control system and drive mechanism. Permanent thermostatically controlled tubular heating elements in the base of the tank heat the bath liquor to the required temperature. The stainless steel construction throughout ensures efficient heat transfer to the specimen containers that are mounted on a rotating horizontal carrier driven by a geared motor. The rotating movement of the carrier 'throws' the liquid from one end of the container to the other in a continuous action. This movement simulates the mechanical washing process and additional mechanical action can be obtained by using steel ball bearings or discs.

15

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The Linitest pots were attached to the Linitester cradle and rotated for 45 minutes at 25°C for emulsions based on SilCare 41M65, and at 40°C for emulsions based on SilCare 41M80 to simulate the main wash.

25

At the end of the main wash simulation, each linitest pot was emptied of wash liquor. An 85ml aliquot of water was added to each pot and the pots were resealed and returned to the Linitester to rotate for a further 10 minutes to simulate rinsing. Rinsing was performed at the same temperature as the mainwash. This was repeated one further time, so that each fabric had

30

experienced two simulated rinses.

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After rinsing, the fabrics were squeezed to remove excess liquid and were line dried at ambient temperature.

Mechanical testing of fabric test samples

5

a) Fabric sample preparation

Samples that underwent model wash evaluation were trimmed to 20cm by 20cm in size, then allowed to condition for 24hours in a room with controlled
10 temperature and humidity (20°C; 65% RH) prior to mechanical testing.

b) Evaluation of fabric mechanical properties related to tactile performance.

An indication of increased levels of deposition of silicone based materials to fabric
15 substrates can be obtained by evaluating the change in fabric mechanical properties caused by lubrication of fibres and yarns due to the presence of silicone.

Several methods exist to relate fabric mechanical properties to fabric handle. An
20 internationally recognised method is the Kawabata Evaluation System (KES). In particular one fabric parameter that has been found to relate to tactile properties such as softness and flexibility is the fabric shear hysteresis. In this example, an automated apparatus based on the KES was used to determine fabric shear hysteresis. The fabrics were oriented with the courses perpendicular to the
25 clamping jaws.

A lower value of shear hysteresis (2HG3 parameter) generally corresponds to increased lubrication and silicone deposition.

Table 1: Fabric shear hysteresis results (using SilCare 41M65)

Fabric treatment	Shear hysteresis (2HG3)
Liquid detergent only (no silicone emulsion)	2.30
Comparative example A	2.04
SilCare 41M65/PETPOET#170707	1.72

5

Table 2: Fabric shear hysteresis results (using SilCare 41M80)

Fabric treatment	Shear hysteresis (2HG3)
Liquid detergent only (no silicone emulsion)	2.24
Comparative example A	2.47
SilCare 41M80/PETPOET#170707	1.70

- 10 A reduction in fabric shear hysteresis (related to increased softening) was observed due to incorporation of PETPOET#170707 with silicone wax.

Determination of amount of silicone deposited on polyester

- 15 The amount of silicone deposited onto polyester fabric as a result of the model wash evaluation was determined by analysis of elemental silicon using X-ray Fluorescence (Phillips PW2400). The mean result from analysis of four fabric samples per treatment is shown in tables 3 and 4.
- 20 The limit of detection for Si using this technique is 10ppm. For the purposes of averaging, where levels were below the limit of detection (<10ppm Si), values were considered to be equal to 10ppm.

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Table 3: Silicon deposition results (using SilCare 41M65)

Fabric treatment	Si (ppm)
Liquid detergent only (no silicone emulsion)	12
Comparative example A	11
SilCare 41M65/PETPOET#170707	39

5

Table 4: Silicon deposition results (using SilCare 41M80)

Fabric treatment	Si (ppm)
Liquid detergent only (no silicone emulsion)	11
Comparative example A	16
SilCare 41M80/PETPOET#170707	33

A significant increase in silicone deposition was observed due to incorporation of PETPOET#170707 with silicone wax.

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Example 2: Preparation of Silicone wax/ perfume emulsion using PET-POET as emulsifier, and enhancement of fabric properties from wash delivery.

Synthesis of polyethylene terephthalate-co-polyoxyethylene terephthalate (PET-POET #170707)

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PETPOET#170707 was synthesised in an identical manner to that described in example 1.

20 **Model perfume blend:**

A model perfume formulation containing top, middle and base notes was prepared by blending components as shown in Table 5.

Table 5: Blend of perfume components.

Note Type	Wt%	Component
Top	20	Manzanate
	20	Limonene
	20	Dihydro myrcenol
	20	Benzyl acetate
	20	Anis aldehyde
Middle	35	OTBCA
	5	Delta damascone
	20	C12 Aldehyde
	20	Verdyl acetate
	20	Beta ionone
Base	20	Bangalol
	20	Iso E super
	20	Hexyl cinnamic aldehyde
	20	Cyclopentadecanolide
	20	2 phenyl ethyl 2-phenyl acetate

5 **Silicone wax emulsion preparation**

Silicone wax was sourced from Clariant, namely, SilCare 41M65 (Stearyl Dimethicone, softening point near 30°C).

- 10 Emulsions were prepared with deionised water and 1% PET-POET(#170707) as the continuous phase and 5 % of silicone wax, and 2.5% of model perfume as the disperse phase. For the preparation of emulsions, the silicone wax was melted to about 10°C - 20°C above its melting point, and model perfume was added. The continuous phase was also heated to 80°C - 90°C to match the temperature (the
- 15 same temperature) of the disperse phase ensuring that it was not boiling. The disperse phase was then added to the continuous phase and homogenised at 13500 rpm for 5 - 20 minutes using a rotor-stator system (Ultraturrax® T25 basic, IKA-WERKE GmbH & Co. KG, Staufen, Germany) to form a coarse emulsion. Homogenisation of the coarse emulsion was carried out in a double sealed beaker

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connected to a water bath to ensure the temperature was maintained above the melting points of the silicone wax.

After homogenisation of the coarse emulsion, it was immediately further
5 homogenised at 1200 bar for approximately 2 cycles using the Microfluidizer[®] M-110S (Microfluidics International Corporation, MA-Newton, USA). Samples were collected in sterile containers and left on the bench for cooling till samples reached room temperature (20°C).

10 **Wash evaluation**

The performance of silicone wax/ perfume/ PET-POET#170707 particles was evaluated on modern polyester fabric in a washing machine trial.

15 Two identical washloads were prepared comprising 950g of 100% polyester fleece with anti-pill finish and 750g of woven 65% polyester /35% viscose with Teflon finish.

Each washload was laundered on a 40°C express cycle in local medium/soft
20 water (typically 6-18°F) using a Miele Honeycomb care W1714 machine, with 1400rpm spin. One washload was washed in the presence of Persil Small and Mighty liquid laundry detergent (35ml dose via dosing ball placed inside the wash drum). The other washload was washed in the presence of liquid detergent containing silicone wax/perfume/PET-POET#170707 particles (7g of 5% silicone
25 wax; 2.5% perfume; 1%PET-POET#170707 emulsion stirred into 35ml of Persil Small and Mighty liquid laundry detergent)

The polyester fleece was tumble dried (Miele Novotronic T430, normal+ setting), and the polyester/viscose was line dried.

Mechanical testing of fabric

a) Fabric sample preparation

5 Twelve 20cm by 20cm samples were cut from the polyester/viscose fabric that underwent wash evaluation, and were allowed to condition for 24 hours in a room with controlled temperature and humidity (20°C; 65% RH) prior to mechanical testing.

10 b) Evaluation of fabric mechanical properties related to tactile performance.

An indication of increased levels of deposition of silicone based materials to fabric substrates can be obtained by evaluating the change in fabric mechanical properties caused by lubrication of fibres and yarns due to the presence of
15 silicone.

Several methods exist to relate fabric mechanical properties to fabric handle. An internationally recognised method is the Kawabata Evaluation System (KES). In particular one fabric parameter that has been found to relate to tactile properties
20 such as softness and flexibility is the fabric shear hysteresis. In this example, an automated apparatus based on the KES was used to determine fabric shear hysteresis. The fabrics were oriented with the warp threads perpendicular to the clamping jaws.

25 A lower value of shear hysteresis (2HG5 parameter) generally corresponds to increased lubrication and silicone deposition.

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Table 6: Fabric shear hysteresis results (polyester/viscose):

Fabric treatment	Shear hysteresis (2HG5)
Liquid detergent only (no silicone emulsion)	4.17
SilCare 41M65/perfume/PETPOET#170707	3.90

A reduction in fabric shear hysteresis (related to increased softening) was achieved through use of silicone wax/perfume particles with PETPOET#170707 incorporated.

Example 3: Preparation of Silicone wax emulsion using LBG-silicone as emulsifier, and enhancement of fabric properties from wash delivery.

Silicone wax emulsion preparation

Silicone wax was sourced from Clariant, namely, SilCare 41M65 (Stearyl Dimethicone, softening point near 30°C). LBG-silicone emulsifier is a polydimethylsiloxane substituted locust bean gum, prepared according to the method for silicone functionalisation of polysaccharides disclosed in WO 2006/117386.

Emulsions were prepared with deionised water and 1% LBG-silicone as the continuous phase and 5 % of silicone wax as the disperse phase. For the preparation of emulsions, the silicone wax was melted to about 10°C - 20°C above its melting point. The continuous phase was also heated to match the temperature (the same temperature) of the disperse phase ensuring that it was not boiling. The disperse phase was then added to the continuous phase and homogenised at 13500 rpm for 5 - 20 minutes using a rotor-stator system (Ultraturrax® T25 basic, IKA-WERKE GmbH & Co. KG, Staufen, Germany) to form a coarse emulsion. Homogenisation of the coarse emulsion was carried out in a

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double sealed beaker connected to a water bath to ensure the temperature was maintained above the melting points of the silicone wax.

After homogenisation of the coarse emulsion, it was immediately further

- 5 homogenised at 1200 bar for approximately 2 cycles using the Microfluidizer[®] M-110S (Microfluidics International Corporation, MA-Newton, USA). Samples were collected in sterile containers and left on the bench for cooling till samples reached room temperature (20°C).

10 **Wash evaluation**

The performance of LBG-silicone/silicone wax particles was evaluated on a mixed load of cotton containing fabric in a washing machine trial.

- 15 Two identical washloads (of total weight 1855g) were prepared comprising 675g of 100% cotton terry towelling, 345g of 100% cotton interlock, 530g of 100% cotton sheeting; and 305g of woven polycotton (65:35 polyester/cotton blend). Each washload was laundered on a 30°C express cycle in local medium/soft water (typically 6-18°F) using a Miele Honeycomb care W1714 machine, with
20 1400rpm spin. One washload was washed in the presence of Persil Small and Mighty liquid laundry detergent (35ml dose via dosing ball placed inside the wash drum). The other washload was washed in the presence of liquid detergent containing LBG-silicone/silicone wax particles (11.67g of 5% silicone wax/1% LBG-silicone emulsion stirred into 35ml of Persil Small and Mighty liquid laundry
25 detergent)

The washloads were tumble dried using a Miele Novotronic T430 tumble drier, using the 'extra dry' setting.

Mechanical testing of fabric

a) Fabric sample preparation

- 5 Eight 20cm by 20cm samples were cut from each fabric that underwent wash evaluation, and were allowed to condition for 24 hours in a room with controlled temperature and humidity (20°C; 65% RH) prior to mechanical testing.

b) Evaluation of fabric mechanical properties related to tactile performance.

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An indication of increased levels of deposition of silicone based materials to fabric substrates can be obtained by evaluating the change in fabric mechanical properties caused by lubrication of fibres and yarns due to the presence of silicone.

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Several methods exist to relate fabric mechanical properties to fabric handle. An internationally recognised method is the Kawabata Evaluation System (KES). In particular one fabric parameter that has been found to relate to tactile properties such as softness and flexibility is the fabric shear hysteresis. In this example, an automated apparatus based on the KES was used to determine fabric shear hysteresis.

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A lower value of shear hysteresis (2HG5 parameter) generally corresponds to increased lubrication and silicone deposition.

Table 7: Fabric shear hysteresis results:

Fabric type	Fabric treatment	Shear hysteresis (2HG5)
Cotton terry towelling	Liquid detergent only (no silicone emulsion)	4.29
Cotton terry towelling	SilCare 41M65/LBG-silicone	4.15
Cotton sheeting	Liquid detergent only (no silicone emulsion)	8.44
Cotton sheeting	SilCare 41M65/LBG-silicone	7.84
Cotton interlock	Liquid detergent only (no silicone emulsion)	2.16
Cotton interlock	SilCare 41M65/LBG-silicone	1.96
Polycotton	Liquid detergent only (no silicone emulsion)	7.08
Polycotton	SilCare 41M65/LBG-silicone	6.42

5 The addition of LBG-silicone/ silicone wax particles to the liquid detergent resulted in a reduction in shear hysteresis (related to increased flexibility/softening) on all fabrics.

Determination of amount of silicone deposited on cotton-based fabrics

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The amount of silicone deposited onto cotton-containing fabric as a result of the wash evaluation was determined by analysis of elemental silicon using X-ray Fluorescence (Phillips PW2400). The mean result from analysis of six fabric samples per treatment is shown in table 8.

Table 8: Silicone deposition results

Fabric type	Fabric treatment	Si (ppm)
Cotton terry towelling	Liquid detergent only (no silicone emulsion)	48
Cotton terry towelling	SilCare 41M65/LBG-silicone	55
Cotton sheeting	Liquid detergent only (no silicone emulsion)	379
Cotton sheeting	SilCare 41M65/LBG-silicone	411
Cotton interlock	Liquid detergent only (no silicone emulsion)	324
Cotton interlock	SilCare 41M65/LBG-silicone	325
Polycotton	Liquid detergent only (no silicone emulsion)	93
Polycotton	SilCare 41M65/LBG-silicone	116

Enhanced silicon levels were detected on the fabrics as a result of treatment with

- 5 SilCare41M65/LBG-silicone particles. This corresponds to at least 50% of the added silicone wax particles being deposited across the washload of cotton-based fabrics.

CLAIMS

1. Particles comprising a waxy solid and a polymeric deposition aid having no overall cationic charge, wherein the polymeric deposition aid is partially embedded in the waxy solid.
5
2. Particles according to claim 1, further comprising a benefit agent dispersed in, or dissolved in, the waxy solid.
- 10 3. Particles according to claim 1 or 2, wherein the waxy solid comprises at least one of: silicone waxes, hydrocarbon waxes or sucrose polyester materials.
- 15 4. Particles according to any of claims 1-3, wherein the deposition aid comprises at least one of: non-hydrolysable cotton-substantive polymer, hydrolysable cotton-substantive polymer or polyester-substantive polymer.
- 20 5. Particles according to claim 4, wherein the polymeric deposition aid comprises at least one of:
 - a) tamarind gum, guar gum, locust bean gum, or,
 - b) gums derived from Tara, Fenugreek, Aloe, Chia, Flaxseed, Psyllium seed, quince seed, or,
 - 25 c) xanthan, gellan, welan, rhamsan, dextran, curdlan, pullulan, scleroglucan, schizophyllan, chitin, hydroxyalkyl cellulose, arabinan, de- branched arabinan, arabinoxylan, galactan, pectic galactan, galactomannan, glucomannan, lichenan, mannan, pachyman, 30 rhamnogalacturonan, acacia gum, agar, alginates, carrageenan,

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chitosan, clavan, hyaluronic acid, heparin, inulin, cellodextrins, cellulose and derivatives thereof.

- 5 6. Particles according to claim 4, wherein the deposition aid comprises a polysaccharide which has been modified to render it more water soluble by means of groups covalently attached to the polysaccharide by means of hydrolysable bond independently selected from one or more of acetate, propanoate, trifluoroacetate, 2- (2-hydroxy-1-oxopropoxy) propanoate, lactate, glycolate, pyruvate, crotonate, isovalerate cinnamate, formate, 10 salicylate, carbamate, methylcarbamate, benzoate, gluconate, methanesulphonate, toluene, sulphonate, groups and hemiester groups of fumaric, malonic, itaconic, oxalic, maleic, succinic, tartaric, aspartic, glutamic, and malic acids.
- 15 7. Particles according to claim 4, wherein the deposition aid is a phthalate containing polymer.
- 20 8. Particles according to claim 7 wherein the deposition aid is a polymer having one or more nonionic hydrophilic components comprising oxyethylene, polyoxyethylene, oxypropylene or polyoxypropylene segments, and, one or more hydrophobic components comprising terephthalate segments.
- 25 9. Particles according to any of claims 1-8 wherein the deposition aid is a block copolymer comprising at least two regions which differ in their hydrophobic/hydrophilic nature such that a portion of the molecule facilitates adsorption onto the waxy-solid particle and a further portion is deposition promoting.

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10. Particles according to any of claims 1-9 wherein the molecular weight of the polymeric deposition aid is in the range of from about 5kD to about 500kD, preferably 10kD-500kD, more preferably 20kD-300kD.
- 5 11. Particles according to any of claims 1-10 wherein the polymeric deposition aid is present at levels such that the ratio polymer:waxy solid is in the range 1:50-3:1, preferably 1:20-1:3.
- 10 12. Particles according to any of claims 1-11 wherein the particle size is between 50 microns and 20nm.
- 15 13. Particles according to any of claims 1-12 wherein the particle further comprises at least one a benefit agent selected from: silicone oils, resins, and modifications thereof, perfume components, organic sunscreen actives, antimicrobial agents, ester solvents, lipids and lipid like substance, hydrocarbon oils, fish and vegetable oils, hydrophobic plant extracts and pigments.
- 20 14. A process for the preparation of particles according to any of claims 1-13 wherein the polymeric deposition aid is contacted with an emulsion of which the waxy solid, in molten form, comprises the dispersed phase.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2010/052815

A. CLASSIFICATION OF SUBJECT MATTER

INV. C11D17/00 C11D3/37 C11D3/22 C11D3/50 C11D1/66
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	US 2003/203829 A1 (SHEFER ADI [US] ET AL) 30 October 2003 (2003-10-30) paragraphs [0054], [108], [118]; claims 1, 22, 23, 30-32, 36; examples 1, 3	1-6, 10-14
X	WO 03/020819 A1 (UNILEVER PLC [GB]; UNILEVER NV [NL]; LEVER HINDUSTAN LTD [IN]) 13 March 2003 (2003-03-13) claims 1, 2, 8, 13, 14; examples 1, 3, 3A	1-6, 9-13
A,P	WO 2009/037060 A1 (UNILEVER PLC [GB]; UNILEVER NV [NL]; UNILEVER HINDUSTAN [IN]; ALONSO C) 26 March 2009 (2009-03-26) page 12, line 21 - line 28 page 16, line 21 - line 23; claims 1-4	1, 2, 4-10
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Further documents are listed in the continuation of Box C.



See patent family annex.

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"&" document member of the same patent family

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INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2010/052815

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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