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(54) **FABRIC TREATMENT**

TEXTILBEHANDLUNG

TRAITEMENT DE TISSUS

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WO-A1-03/020864 **WO-A1-2008/058833**
WO-A1-2010/028907 **WO-A1-2010/060677**
US-A- 5 007 478 **US-A1- 2004 121 072**

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DescriptionTechnical Field

5 **[0001]** The present invention relates to a use of an encapsulated phase change material to improve the beneficial effect of an additional encapsulated material, which is a volatile benefit agent.

Background and Prior Art

10 **[0002]** Encapsulated technologies, for example encapsulated perfume, are known for use in laundry products. Such technologies provide enhanced fragrance delivery over conventional free perfume oil by overcoming the issue of perfume loss during the drying process by protecting the perfume in the capsule. Encapsulation also ensures that perfume is released at the optimal time to enable the provision of a perceivable benefit to the wearer of laundered garments. Examples of the mode of action of encapsulates include: shear sensitive action, where the perfume core is released in response to mechanical rupture of the encapsulate, and diffusive action, wherein perfume is released by diffusion through the outer wall of the encapsulate. Some encaps are capable of both release mechanisms. One type of capsule that has been used in laundry compositions has a melamine formaldehyde shell and a perfume core. Release of perfume from melamine formaldehyde capsules is friction based, the benefit becoming apparent after a rubbing process is applied to the treated fabric. This benefit is provided by a boost in perfume intensity during wear.

20 **[0003]** WO 2010/060677 and WO 2010/028907 disclose the use of encapsulated perfume particles to improve the freshness longevity of laundry detergent compositions or fabric conditioners, which may also comprise further encaps. Phase change materials are also known in encapsulated form. Our related European patent number EP07821655 discloses the use of a fabric softening composition comprising a fabric softening compound and a material having a thermal phase transition temperature in the range 24 to 39°C to impart a cool feel to a fabric. WO2008/058833 also concerns fabric softening compound and a material having a thermal phase transition temperature in the range 26 to 39°C encapsulated in a polymer shell.

25 **[0004]** EP0371535 discloses fabric treatment compositions having antiwrinkling benefits which contain a mixture of hydrocarbons which has a thermal phase transition temperature of between 27 and 38°C. US2002/0061954 is directed to thermal energy storage compositions comprising microcapsules which contain a plurality of microencapsulated phase change materials to provide a cooling effect.

30 **[0005]** We have now found that the inclusion of a low level of encapsulated phase change material significantly increases the shear release effect associated with a fabric treatment formulation containing encapsulated volatile benefit agents, for example perfume.

Definition of the Invention

35 **[0006]** According to the invention there is provided the use of an encapsulated phase change active, having a phase transition temperature of from 24 to 39°C, to improve the beneficial effect of an additional encapsulated volatile benefit agent in the presence of an additional non-encapsulated volatile benefit agent, wherein the encapsulated volatile benefit agent is selected from perfume, insect repellent, aromatherapy oil, sensates such as menthol and an essential oil, and wherein the phase change active is a paraffin wax comprising n-octadecane.

40 **[0007]** A laundry composition provides a convenient mode of delivery for the encapsulated materials to provide the use according to the invention.

Detailed Description of the Invention**The Encapsulated Volatile Benefit Agent**

45 **[0008]** The encapsulated volatile benefit agent comprises a capsule and a volatile benefit agent. The capsule comprises a shell and a core.

50 **[0009]** The capsule comprising the volatile benefit agent comprises a shell that is comprised of materials including but not limited to polyurethane, polyamide, polyolefin, polysaccharide, protein, silicone, lipid, modified cellulose, gums, polyacrylate, polyphosphate, polystyrene, polyesters or combinations of these materials. Other encapsulating material which may be used effectively in the present invention, such as polymethylmethacrylate. Preferred encapsulating polymers include those formed from melamine formaldehyde or urea formaldehyde condensates, as well as similar types of aminoplasts. Most preferably the shell comprises melamine formaldehyde.

55 **[0010]** Additionally, microcapsules made via the simple or complex coacervation of gelatin are suitable for use in compositions of the invention.

5 [0011] A representative process used for aminoplast encapsulation is disclosed in 3516941 USAU.S. Patent No. 3,516,941 though it is recognised that many variations with regard to materials and process steps are possible. A representative process used for gelatin encapsulation is disclosed in 2800457USAU.S. Patent No. 2,800,457 though it is recognized that many variations with regard to materials and process steps are possible. Both of these processes are discussed in the context of fragrance encapsulation for use in consumer products in 4145184USAU.S. Patent Nos. 4,145,184 and 5112688USA5,112,688 respectively.

[0012] Encapsulation can provide pore vacancies or interstitial openings depending on the encapsulation techniques employed.

10 [0013] Fragrance capsules known in the art and suitable for use in the present invention comprise a wall or shell comprising a three-dimensional cross-linked network of an aminoplast resin, more specifically a substituted or unsubstituted acrylic acid polymer or co-polymer cross-linked with a urea-formaldehyde pre-condensate or a melamine-formaldehyde pre-condensate.

15 [0014] Microcapsule formation using mechanisms similar to the foregoing mechanism, using (i) melamine-formaldehyde or urea-formaldehyde pre-condensates and (ii) polymers containing substituted vinyl monomeric units having proton-donating functional group moieties (e.g. sulfonic acid groups or carboxylic acid anhydride groups) bonded thereto is disclosed in 44068162USBU.S. Patent 4,406,816 (2-acrylamido-2-methyl-propane sulfonic acid groups), 2062570GBAUK published Patent Application GB 2,062,570 A (styrene sulfonic acid groups) and 2006709GBAUK published Patent Application GB 2,006,709 A (carboxylic acid anhydride groups).

20 [0015] The capsules for use in the invention may further comprise a carrier oil in the core. The carrier oils are hydrophobic materials that are miscible in the volatile benefit agent materials used in the present invention. Suitable oils are those having reasonable affinity for the benefit agent. Where the benefit agent is a perfume, suitable materials include, but are not limited to triglyceride oil, mono and diglycerides, mineral oil, silicone oil, diethyl phthalate, polyalpha olefins, castor oil and isopropyl myristate. Preferably, the oil is a triglyceride oil, most preferably a capric/caprylic triglyceride oil.

25 [0016] For liquid laundry compositions, the capsules may be used in the form of a slurry, which preferably comprises about 40% solids.

30 [0017] Particle size and average diameter of the capsules can vary from about 10 nanometers to about 1000 microns, preferably from about 50 nanometers to about 100 microns, more preferably from about 2 to about 40 microns, even more preferably from about 4 to 15 microns. A particularly preferred range is from about 5 to 10 microns, for example 6 to 7 microns. The capsule distribution can be narrow, broad or multimodal. Multimodal distributions may be composed of different types of capsule chemistries.

[0018] The shell may further comprise a deposition aid, which is preferably covalently attached.

[0019] A preferred deposition aid is a polysaccharide. The polysaccharide preferably has a β -1,4-linked backbone.

35 [0020] 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, polyglucosaminan, polyxyloglucan and polygalactomannan or a mixture thereof. More preferably, the polysaccharide is selected from the group consisting of polyxyloglucan and polygalactomannan.

[0021] Highly preferred polysaccharides are selected from locust bean gum, tamarind gum, xyloglucan, non-ionic guar gum, cationic starch and mixtures thereof. Most preferably, the deposition aid is locust bean gum.

40 [0022] Preferably, the polysaccharide backbone has only β -1,4 linkages. Optionally, the polysaccharide has linkages in addition to the β -1,4 linkages, such as β -1,3 linkages. Thus, optionally some other linkages are present. Polysaccharide backbones which include some material which is not a saccharide ring are also within the ambit of the present invention (whether terminal or within the polysaccharide chain).

45 [0023] The polysaccharide may be straight or branched. Many naturally occurring polysaccharides have at least some degree of branching, or at any rate at least some saccharide rings are in the form of pendant side groups (which are therefore not in themselves counted in determining the degree of substitution) on a main polysaccharide backbone.

[0024] Preferably, the polysaccharide is present at levels of between 0.1 % to 10% w/w by weight of the total amount of the particle.

50 [0025] The deposition aid, which is preferably a polysaccharide, is attached to the particle by means of a covalent bond, entanglement or strong adsorption, preferably by a covalent bond or entanglement and most preferably by means of a covalent bond. By entanglement as used herein is meant that the deposition aid is adsorbed onto the particle as the polymerisation proceeds and the particle grows in size, part of the adsorbed deposition aid becomes buried within the interior of the particle. Hence at the end of the polymerisation, part of the deposition aid is entrapped and bound in the polymer matrix of the particle, whilst the remainder is free to extend into the aqueous phase.

55 [0026] By strong adsorption as used herein is meant strong adsorption of the deposition aid to the surface of the particle; such adsorption can, for example, occur due to hydrogen bonding, Van Der Waals or electrostatic attraction between the deposition aid and the particle.

[0027] The deposition aid is thus mainly attached to the particle surface and is not, to any significant extent, distributed throughout the internal bulk of the particle. This is distinct from graft copolymers in which e.g. a polysaccharide may be

grafted along the length of a polymer chain. A particle which is formed from a graft copolymer would, therefore, contain polysaccharide throughout the internal bulk of the particle as well as on the particle surface and the present invention is not intended to cover such a particle. Thus the particle which is produced when using a polysaccharide as the deposition aid according to the process of the invention can be thought of as a "hairy particle", which is different from a graft copolymer. This feature of the invention provides significant cost reduction opportunities for the manufacturer as much less deposition aid is required to achieve the same level of activity as systems which utilise polysaccharide copolymers.

[0028] The deposition aid is present in the outermost portion of the shell, which is made of melamine formaldehyde polymer having a thickness of from 5 to 20 nm.

[0029] Polyesters of terephthalic and other aromatic dicarboxylic acids having soil release properties, in particular, the so-called PET/POET (polyethylene terephthalate/polyoxyethylene terephthalate) and PET/PEG (polyethylene terephthalate/polyethylene glycol) polyesters may be employed as deposition aids.

[0030] The polymer must have at least one mole free OH group per mole polymer, to allow covalent binding to the reactive dye(s). Most preferably the polymer comprises at least two free OH groups. Preferably the OH groups are the terminal groups of the polymer.

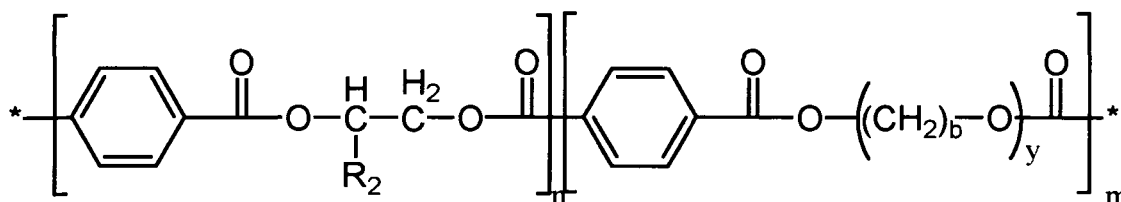
[0031] Preferably, the oxyalkyleneoxy $[-O(CH_2)_tO-]$ is selected from: oxy-1,2-propyleneoxy $[-OCH_2CH(Me)O-]$; oxy-1,3-propyleneoxy $[O-CH_2CH_2CH_2O-]$; and, oxy-1,2-ethyleneoxy $[-OCH_2CH_2O-]$ (t is an interger). As is evident one or more of the CH_2 groups of the oxyalkyleneoxy may be substituted by C1 to C4 alkyl group(s).

[0032] The polyoxyalkyleneoxy facilitates water solubility of the polymer. Preferably, the polyoxyalkyleneoxy $[-O(CH_2)_w-]_sO-$ is selected from: polyoxy-1,2-propyleneoxy $[-O(CH_2CH(Me)-)]_sO-$; polyoxy-1,3-propyleneoxy $[O-CH_2CH_2CH_2-]_sO-$; and, polyoxy-1,2-ethyleneoxy $[O-CH_2CH_2-]_sO-$; The polyoxyalkyleneoxy may be a mixture of different oxyalkyleneoxy. Different polyoxyalkyleneoxy types may present in the polymer. (s and w are intergers).

[0033] Preferably the phenyl dicarboxylate is a 1,4-phenyl dicarboxylate. Preferably the phenyl dicarboxylate is of the form: $-OC(O)C_6H_4C(O)-$.

[0034] Examples of preferred polymers are a PET/POET (Polyethylene terephthalate/polyoxyethylene terephthalate), PEG/POET (Polyethyleneglycol/ polyoxyethylene terephthalate) or PET/PEG (Polyethylene terephthalate/ Polyethyleneglycol) polymer. Most preferable a PET/POET.

[0035] The structure of a preferred polymer is found below.



wherein

R_2 is selected from H or CH_3 , preferably H;

b is 2 or 3, preferably 2;

y is 2 to 100, preferably 5 to 50;

n and m are independently 1 to 100, preferably 2 to 30; and, the terminal (end) groups of the polymer are $(CH_2)_bOH$.

[0036] The polymers may be synthesised by a variety of routes, for example an esterification reaction of dimethyl terephthalate with ethyleneglycol and polyethyleneglycol, this reaction is discussed in Polymer Bulletin 28, 451-458 (1992). Another example would be the direct esterification of terephthalic acid with ethylene glycol and/or propylene glycol and polypropylene glycol.

[0037] A further example would be a transesterification of a polyethyleneterephthalate with a polyethyleneglycol or polypropylene glycol.

[0038] It is preferred that the number average molecular weight of the polymer is in the range from 1000 to 50,000, preferably the average molecular weight of the polymer is in the range of from 1000 to 15000, more preferably from 2000 to 10000.

The Volatile Benefit Agent

[0039] The volatile benefit agent is an agent which is volatile and which confers a benefit to fabric.

[0040] Suitable volatile benefit agents include but are not limited to perfumes, insect repellents, essential oils, sensates

such as menthol and aromatherapy actives, preferably perfumes. Mixtures of volatile benefit agents may be used.

[0041] The total amount of volatile benefit agent is preferably from 0.01 to 10 % by weight, more preferably from 0.05 to 5 % by weight, even more preferably from 0.1 to 4.0 %, most preferably from 0.15 to 4.0 % by weight, based on the total weight of the composition.

[0042] The preferred volatile benefit agent is a perfume. The compositions of the invention also comprise an unconfined (also called non-encapsulated) volatile benefit agent. Where the volatile benefit agent is a perfume, the perfumes described below are suitable for use as the encapsulated volatile benefit agent and also as the unconfined perfume component.

[0043] 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 Flavor Ingredients, 1975, CRC Press; Synthetic Food Adjuncts, 1947 by M. B. Jacobs, edited by Van Nostrand; or Perfume and Flavor Chemicals by 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.

[0044] 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'.

[0045] 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 envisaged that at least 20%wt would be present within the encapsulate.

[0046] Some or all of the perfume or pro-fragrance may be encapsulated, typical perfume components which it is advantageous to encapsulate, include those with a relatively low boiling point, preferably those with a boiling point of less than 300, preferably 100-250 Celsius and pro-fragrances which can produce such components.

[0047] It is also advantageous to encapsulate perfume components which have a low Clog P (i.e. those which will be partitioned into water), preferably with a Clog P of less than 3.0. These materials, of relatively low boiling point and relatively low Clog P have been called the "delayed blooming" perfume ingredients and include the following materials:

[0048] 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, Laevo-Carvone, d-Carvone, Cinnamic Alcohol, Cinamyl Formate, Cis-Jasmone, cis-3-Hexenyl Acetate, Cuminal Alcohol, Cycloal 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), 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 Benzyl Acetate, Methyl Eugenol, Methyl Heptenone, Methyl Heptene Carbonate, Methyl Heptyl Ketone, Methyl Hexyl Ketone, Methyl Phenyl Carbonyl 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, Phenyl Ethyl Alcohol, Phenyl Ethyl Dimethyl Carbinol, Prenyl Acetate, Propyl Bornate, Pulegone, Rose Oxide, Safrole, 4-Terpinenol, Alpha-Terpinenol, and/or Viridine.

[0049] Preferred non-encapsulated perfume ingredients are those hydrophobic perfume components with a ClogP above 3. As used herein, the term "ClogP" means the calculated logarithm to base 10 of the octanol/water partition coefficient (P). The octanol/water partition coefficient of a perfume raw material (PRM) is the ratio between its equilibrium concentrations in octanol and water. Given that this measure is a ratio of the equilibrium concentration of a PRM in a non-polar solvent (octanol) with its concentration in a polar solvent (water), ClogP is also a measure of the hydrophobicity of a material—the higher the ClogP value, the more hydrophobic the material. ClogP values can be readily calculated from a program called "CLOGP" which is available from Daylight Chemical Information Systems Inc., Irvine Calif., USA. Octanol/water partition coefficients are described in more detail in U.S. Pat. No. 5,578,563.

[0050] Perfume components with a ClogP above 3 comprise: Iso E super, citronellol, Ethyl cinnamate, Bangalol, 2,4,6-Trimethylbenzaldehyde, Hexyl cinnamic aldehyde, 2,6-Dimethyl-2-heptanol, Diisobutylcarbinol, Ethyl salicylate, Phenethyl isobutyrate, Ethyl hexyl ketone, Propyl amyl ketone, Dibutyl ketone, Heptyl methyl ketone, 4,5-Dihydrotoluene, Caprylic aldehyde, Citral, Geraniol, Isopropyl benzoate, Cyclohexanepropionic acid, Campholene aldehyde, Caprylic acid, Caprylic alcohol, Cuminaldehyde, 1-Ethyl-4-nitrobenzene, Heptyl formate, 4-Isopropylphenol, 2-Isopropylphenol, 3-Isopropylphenol, Allyl disulfide, 4-Methyl-1-phenyl-2-pentanone, 2-Propylfuran, Allyl caproate, Styrene, Isoeugenyl methyl ether, Indonaphthene, Diethyl suberate, L-Menthone, Menthone racemic, p-Cresyl isobutyrate, Butyl butyrate, Ethyl hexanoate, Propyl valerate, n-Pentyl propanoate, Hexyl acetate, Methyl heptanoate, trans-3,3,5-Trimethylcyclohexanol, 3,3,5-Trimethylcyclohexanol, Ethyl p-anisate, 2-Ethyl-1-hexanol, Benzyl isobutyrate, 2,5-Dimethylthiophene, Iso-

butyl 2-butenolate, Caprylnitrile, gamma-Nonalactone, Nerol, trans-Geraniol, 1-Vinylheptanol, Eucalyptol, 4-Terpinenol, Dihydrocarveol, Ethyl 2-methoxybenzoate, Ethyl cyclohexanecarboxylate, 2-Ethylhexanal, Ethyl amyl carbinol, 2-Octanol, 2-Octanol, Ethyl methylphenylglycidate, Diisobutyl ketone, Coumarone, Propyl isovalerate, Isobutyl butanoate, Isopentyl propanoate, 2-Ethylbutyl acetate, 6-Methyl-tetrahydroquinoline, Eugenyl methyl ether, Ethyl dihydrocinnamate, 3,5-Dimethoxytoluene, Toluene, Ethyl benzoate, n-Butyrophenone, alpha-Terpineol, Methyl 2-methylbenzoate, Methyl 4-methylbenzoate, Methyl 3-methylbenzoate, sec. Butyl n-butyrate, 1,4-Cineole, Fenchyl alcohol, Pinanol, cis-2-Pinanol, 2,4-Dimethylacetophenone, Isoeugenol, Safrole, Methyl 2-octynoate, o-Methylanisole, p-Cresyl methyl ether, Ethyl anthranilate, Linalool, Phenyl butyrate, Ethylene glycol dibutyrate, Diethyl phthalate, Phenyl mercaptan, Cumic alcohol, m-Toluquinoline, 6-Methylquinoline, Lepidine, 2-Ethylbenzaldehyde, 4-Ethylbenzaldehyde, o-Ethylphenol, p-Ethylphenol, m-Ethylphenol, (+)-Pulegone, 2,4-Dimethylbenzaldehyde, Isoxylaldehyde, Ethyl sorbate, Benzyl propionate, 1,3-Dimethylbutyl acetate, Isobutyl isobutanoate, 2,6-Xylenol, 2,4-Xylenol, 2,5-Xylenol, 3,5-Xylenol, Methyl cinnamate, Hexyl methyl ether, Benzyl ethyl ether, Methyl salicylate, Butyl propyl ketone, Ethyl amyl ketone, Hexyl methyl ketone, 2,3-Xylenol, 3,4-Xylenol, Cyclopentadenanolid and Phenyl ethyl 2 phenylacetate 2.

[0051] It is commonplace for a plurality of perfume components to be present in a formulation. In the compositions 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 and/or the list of perfume components with a ClogP above 3 present in the perfume.

[0052] The volatile benefit agent may be an insect repellent. In chemical terms, most repellent actives belong to one of four groups: amides, alcohols, esters or ethers. Those suitable for use in the present invention are liquids or solids with a relatively low melting point and a boiling point above 150 °C, preferably liquids. They evaporate slowly at room temperature. Where the volatile benefit agent is an insect repellent, the repellents described below are suitable for use as the encapsulated volatile benefit agent and also as the unconfined repellent component.

[0053] Many suitable insect repellents are related to perfume species (many fall into both classes). The most commonly used insect repellents include: DEET (N,N-diethyl-m-toluamide), essential oil of the lemon eucalyptus (*Corymbia citriodora*) and its active compound p-menthane-3,8-diol (PMD), Icaridin, also known as Picaridin, D-Limonene, Bayrepel, and KBR 3023, Nepetalactone, also known as "catnip oil", Citronella oil, Permethrin, Neem oil and Bog Myrtle.

[0054] Known insect repellents derived from natural sources include: *Achillea alpina*, alpha-terpinene, Basil oil (*Ocimum basilicum*), *Callicarpa americana* (Beautyberry), Camphor, Carvacrol, Castor oil (*Ricinus communis*), Catnip oil (*Nepeta* species), Cedar oil (*Cedrus atlantica*), Celery extract (*Apium graveolens*), Cinnamon (*Cinnamomum Zeylanicum*, leaf oil), Citronella oil (*Cymbopogon fleusus*), Clove oil (*Eugenic caryophyllata*), Eucalyptus oil (70%+ eucalyptol, also known as cineol), Fennel oil (*Foeniculum vulgare*), Garlic Oil (*Allium sativum*), Geranium oil (also known as *Pelargonium graveolens*), Lavender oil (*Lavandula officinalis*), Lemon eucalyptus (*Corymbia citriodora*) essential oil and its active ingredient p-menthane-3,8-diol (PMD), Lemongrass oil (*Cymbopogon flexuosus*), Marigolds (*Tagetes* species), Marjoram (*Tetranychus urticae* and *Eutetranychus orientalis*), Neem oil (*Azadirachta indica*), Oleic acid, Peppermint (*Mentha x piperita*), Pennyroyal (*Mentha pulegium*), Pyrethrum (from *Chrysanthemum* species, particularly *C. cinerariifolium* and *C. coccineum*), Rosemary oil (*Rosmarinus officinalis*), Spanish Flag Lantana camara (*Helopeltis theivora*), *Solanum villosum* berry juice, Tea tree oil (*Melaleuca alternifolia*) and Thyme (*Thymus* species) and mixtures thereof.

[0055] Preferred encapsulated insect repellents are mosquito repellents available from Celessence, Rochester, England. Celessence Repel, containing the active ingredient Saltidin™ and Celessence Repel Natural, containing the active Citrepel™ 75. Saltidin is a man made molecule developed originally by the Bayer Corporation. Citrepel is produced from eucalyptus oils and is high in p-menthane-3,8-diol (PMD). A preferred non-encapsulated repellent is Citriodiol™ supplied by Citrefine.

[0056] Another group of volatile benefit agents with which the present invention can be applied are the so-called 'aromatherapy' materials. These include components of essential oils such as Clary Sage, Eucalyptus, Geranium, Lavender, Mace Extract, Neroli, Nutmeg, Spearmint, Sweet Violet Leaf and Valerian.

The Encapsulated Phase Change Active

[0057] Phase change actives are materials that can absorb, store and release heat whilst the material changes its physical form. This is known as a phase change. Water changing from solid (ice) to liquid is an example of this phenomenon. During these phase changes large amounts of heat are absorbed or released.

[0058] The phase change active has a thermal phase transition temperature (TPTT) in the range 24 to 39°C. The TPTT may conveniently be measured by the Perkin & Elmer thermal analysis system.

[0059] The Perkin & Elmer thermal analysis system measures the heat flow into a material to be heated as a function of the temperature of the material. By investigating a material at various temperatures, a temperature profile is obtained. Such a temperature profile usually has one or more peaks, each peak corresponding to a maximum for the heat flow into the material at a specific temperature. The temperature corresponding to the major peak in the temperature profile is referred to as the thermal phase transition temperature. Generally a high TPTT corresponds to a high softening

temperature of the material. The material has a TPTT in the range 24 to 39°C, preferably from 25 to 39°C, more preferably from 26 to 38°C and most preferably from 26 to 30°C.

5 [0060] Phase change actives possess a latent heat and show a phase transition phenomena between phases at a phase transition temperature. The phase transition of the present invention incorporated solid to liquid, liquid to vapor, solid to vapor, gel to liquid-crystalline phase changes. In the present invention, preferable phase transitions are solid to liquid phase or liquid to solid phase changes. At these phase changes, PTMs reversibly absorb or release heat from the environment at around the phase transition temperature, which is accompanied with a corresponding change in the ambient temperature.

10 [0061] The phase change active may be in the form of a composition (or mixture) provided that the total composition has a TPTT in the range 24 to 39°C, preferably from 25 to 39°C, more preferably from 26 to 38°C and most preferably from 26 to 30°C.

15 [0062] Suitable compositions may comprise hydrocarbon materials comprising a linear or branched alkyl chain and preferably comprising an average of from 12 to 50 carbon atoms per molecule, preferably from 12 to 30 carbon atoms. Preferably, the hydrocarbon materials are either alkanes or alkenes. Relatively small amounts of non-alkyl substituent groups may be present provided the hydrocarbon nature of the product is not substantially affected. Mixtures of these materials may be used.

20 [0063] Examples of suitable hydrocarbon materials for use in the hydrocarbon composition are the liquid hydrocarbon materials of natural source. Other liquid hydrocarbon materials including the liquid fractions derived from crude oil, such as mineral oil, liquid paraffins, cracked hydrocarbons and mixtures thereof. In the present invention the phase change active includes paraffin wax (comprising n-Octadecane).

[0064] Examples of solid or semi-solid hydrocarbon materials are the paraffinic materials of longer chain length, and hydrogenated versions of some of the liquid materials mentioned above.

25 [0065] A particularly useful combination of hydrocarbon materials is a mixture of mineral oil (for example, M85 ex Daltons Company) and petroleum jelly (for example, Silkolene 910 ex Daltons), wherein the weight ratio of mineral oil to petroleum jelly is chosen such that the TPTT of the mixture is in the range of from 24 to 39 °C. In our experiments this result was obtained by using a ratio of mineral oil to petroleum jelly of less than 3:1, preferably from 2:1 to 1:3. The mineral oil was a liquid mixture of linear and branched hydrocarbons having an average number of carbon atoms per molecule of 26. Petroleum jelly was a semi-solid mixture of linear and branched hydrocarbons having an average number of carbon atoms per molecule of 26, and having a softening temperature of about 50°C.

30 [0066] The encapsulated phase change active comprises a capsule and a phase change active. The capsule comprises a shell and a core. The capsule for the phase change material preferably has a shell that is permeable to the unconfined volatile benefit agent in the composition. A mixture of encapsulated phase change actives may be present.

35 [0067] The phase change active is encapsulated in a polymer shell to form encapsulated particles having a preferred particle size of from 10 nm to 1000 μm, preferably 50 nm to 100 μm, more preferably 0.2 to 30 μm. The use of encapsulated materials has the advantage that the materials may be readily dispersed without interference or interaction with the fabric softener compound. An additional advantage in that the encapsulated material does not cause a "messiness" feeling when deposited on the fabric which may be present with materials of a semi-liquid nature.

40 [0068] Suitable encapsulating polymers include those formed from melamine-formaldehyde or urea formaldehyde condensates, as well as similar types of aminoplasts. Additionally, capsules made via the simple or complex coacervation of gelatin are also preferred for use with the coating. Capsules having shell walls comprised of polyurethane, polyamide, polyolefin, polysaccharide, protein, silicone, lipid, modified cellulose, gums, polyacrylate, polyphosphate, polystyrene, and polyesters or combinations of these materials are also functional.

45 [0069] Further examples of suitable phase change actives are those materials disclosed in WO 03/014460 having a phase transition temperature of from 24 to 39 °C, referred to therein as "Phase Transition Materials" or "PTM's" at page 6, final paragraph to the penultimate line on page 8.

[0070] A preferred material is Lurapret TX PMC 28 commercially available from BASF which is a material, specifically paraffin wax (comprising n-Octadecane), encapsulated in polymethylmethacrylate having a particle size in the range 0.2 to 20μm. This material has a phase transition temperature of about 28°C.

50 [0071] The phase change actives are generally deposited to apply from 0.2 to 1 %, preferably 0.2 to 0.5 % by weight of the fabric after drying. The encapsulated phase change actives are preferably present in an amount of from 0.01 to 15 wt %, more preferably 0.01 to 10 wt %, even more preferably from 0.05 to 5 wt %, still more preferably from 0.05 to 2 wt %, more preferably still from 0.05 to 1 wt % and most preferably from 0.05 to 0.5 wt % by weight of the fabric softening composition.

55 [0072] The encapsulated phase change material comprises a shell that is permeable to the unconfined volatile benefit agent in the composition. Suitable encapsulating polymers include those formed from melamine-formaldehyde or urea formaldehyde condensates, as well as similar types of aminoplasts. Additionally, capsules made via the simple or complex coacervation of gelatin are also preferred for use with the coating. Capsules having shell walls comprised of polyurethane, polyamide, polyolefin, polysaccharide, protein, silicone, lipid, modified cellulose, gums, polyacrylate, polyphosphate,

polystyrene, and polyesters or combinations of these materials are also suitable. A preferred material is polymethylmethacrylate.

Compositions

[0073] The use of the invention is preferably as a treatment applied by consumers in the home. The treatment may be applied directly to the fabric, e.g. as a spray, or via a laundry product, such as a detergent composition and a fabric conditioner composition. The composition for use in the present invention is preferably a liquid. The compositions are preferably aqueous.

[0074] The laundry product will contain an active ingredient, which 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.

[0075] The detergent compositions for use in 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. The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and non-ionic compounds.

[0076] The detergent compositions for use in the invention may contain linear alkylbenzene sulphonate, particularly linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅. It is preferred if the level of linear alkylbenzene sulphonate is from 0 wt% to 30 wt%, more preferably 1 wt% to 25 wt%, most preferably from 2 wt% to 15 wt%.

[0077] The compositions for use 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 sulphates, particularly C₈-C₁₅ primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

[0078] The compositions for use in the invention may also contain non-ionic surfactant. Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C₈-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-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 alkyl-polyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

[0079] It is preferred if the level of nonionic surfactant is from 0 wt% to 30 wt%, preferably from 1 wt% to 25 wt%, most preferably from 2 wt% to 15 wt%.

Fabric Softening Compound

[0080] The particle for use in the present invention is preferably a component of a laundry composition. The laundry composition is preferably a fabric conditioner. The fabric conditioner preferably comprises a fabric softening active.

[0081] The fabric softening active is preferably different from the phase change active. Suitable fabric softening compounds are described below.

[0082] The fabric conditioning agents (also referred to herein as a fabric softening active or compound) may be cationic or non-ionic.

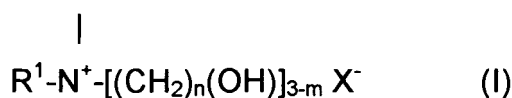
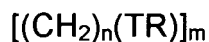
[0083] Fabric conditioning compositions for use in accordance with the invention may be dilute or concentrated. Dilute products typically contain up to about 8 %, generally about 2 to 8 % by weight of softening active, whereas concentrated products may contain up to about 50 wt %, preferably from about 8 to about 50 %, more preferably from 8 to 25 % by weight active. Overall, the products of the invention may contain from 2 to 50 wt %, preferably from 3 to 25 wt % of softening active.

[0084] The preferred softening active for use in rinse conditioner compositions of the invention is a quaternary ammonium compound (QAC). The preferred quaternary ammonium fabric conditioner for use in compositions of the present invention are the so called "ester quats".

[0085] Particularly preferred materials are the ester-linked triethanolamine (TEA) quaternary ammonium compounds comprising a mixture of mono-, di- and tri-ester linked components.

[0086] Typically, TEA-based fabric softening compounds comprise a mixture of mono, di- and tri-ester forms of the compound where the di-ester linked component comprises no more than 70 % by weight of the fabric softening compound, preferably no more than 60 wt % of the fabric softening compound and at least 10 % of the monoester linked component.

[0087] A first group of quaternary ammonium compounds (QACs) suitable for use in the present invention is represented by formula (I):

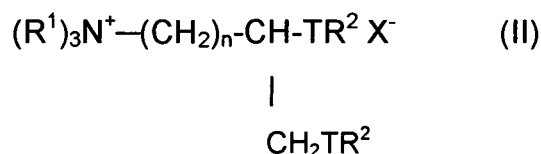


wherein each R is independently selected from a C₅₋₃₅ alkyl or alkenyl group; R¹ represents a C₁₋₄ alkyl, C₂₋₄ alkenyl or a C₁₋₄ hydroxyalkyl group; T is generally O-CO. (i.e. an ester group bound to R *via* its carbon atom), but may alternatively be CO-O (i.e. an ester group bound to R *via* its oxygen atom); n is a number selected from 1 to 4; m is a number selected from 1, 2, or 3; and X⁻ is an anionic counterion, such as a halide or alkyl sulphate, e.g. chloride or methylsulphate. Di-esters variants of formula I (i.e. m = 2) are preferred and typically have mono- and tri-ester analogues associated with them. Such materials are particularly suitable for use in the present invention.

[0088] Especially preferred agents are preparations which are rich in the di-esters of triethanolammonium methylsulphate, otherwise referred to as "TEA ester quats". Commercial examples include Stepantex™ UL85, ex Stepan, Prapagen™ TQL, ex Clariant, and Tetranyl™ AHT-1, ex Kao, (both di-[hardened tallow ester] of triethanolammonium methylsulphate), AT-1 (di-[tallow ester] of triethanolammonium methylsulphate), and L5/90 (di-[palm ester] of triethanolammonium methylsulphate), both ex Kao, and Rewoquat™ WE15 (a di-ester of triethanolammonium methylsulphate having fatty acyl residues deriving from C₁₀-C₂₀ and C₁₆-C₁₈ unsaturated fatty acids), ex Witco Corporation.

[0089] Also, soft quaternary ammonium actives such as Stepantex VK90, Stepantex VT90, SP88 (ex-Stepan), Prapagen TQ (ex-Clariant), Dehyquart AU-57 (ex-Cognis), Rewoquat WE18 (ex-Degussa) and Tetranyl L190 P, Tetranyl L190 SP and Tetranyl L190 S (all ex-Kao) are suitable.

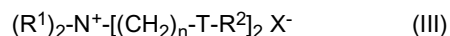
[0090] A second group of QACs suitable for use in the invention is represented by formula (II):



wherein each R¹ group is independently selected from C₁₋₄ alkyl, hydroxyalkyl or C₂₋₄ alkenyl groups; and wherein each R² group is independently selected from C₈₋₂₈ alkyl or alkenyl groups; and wherein n, T, and X⁻ are as defined above.

[0091] Preferred materials of this second group include 1,2 *bis*[tallowoyloxy]-3-trimethylammonium propane chloride, 1,2 *bis*[hardened tallowoyloxy]-3-trimethylammonium propane chloride, 1,2-*bis*[oleoyloxy]-3-trimethylammonium propane chloride, and 1,2 *bis*[stearoyloxy]-3-trimethylammonium propane chloride. Such materials are described in US 4,137,180 (Lever Brothers). Preferably, these materials also comprise an amount of the corresponding monoester.

[0092] A third group of QACs suitable for use in the invention is represented by formula (III):



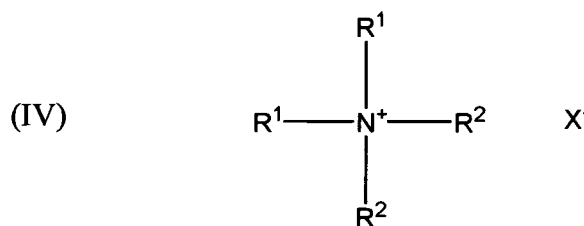
wherein each R¹ group is independently selected from C₁₋₄ alkyl, or C₂₋₄ alkenyl groups; and wherein each R² group is independently selected from C₈₋₂₈ alkyl or alkenyl groups; and n, T, and X⁻ are as defined above. Preferred materials of this third group include bis(2-tallowoyloxyethyl)dimethyl ammonium chloride, partially hardened and hardened versions thereof.

[0093] The iodine value of the quaternary ammonium fabric conditioning material is preferably from 0 to 80, more preferably from 0 to 60, and most preferably from 0 to 45. The iodine value may be chosen as appropriate. Essentially saturated material having an iodine value of from 0 to 5, preferably from 0 to 1 may be used in the compositions of the invention. Such materials are known as "hardened" quaternary ammonium compounds.

[0094] A further preferred range of iodine values is from 20 to 60, preferably 25 to 50, more preferably from 30 to 45. A material of this type is a "soft" triethanolamine quaternary ammonium compound, preferably triethanolamine di-alkylester methylsulphate. Such ester-linked triethanolamine quaternary ammonium compound comprise unsaturated fatty chains.

[0095] Iodine value as used in the context of the present invention refers to the measurement of the degree of unsaturation present in a material by a method of nmr spectroscopy as described in Anal. Chem., 34, 1136 (1962) Johnson and Shoolery.

[0096] A further type of softening compound is a non-ester quaternary ammonium material represented by formula (IV):-



10 wherein each R¹ group is independently selected from C₁₋₄ alkyl, hydroxyalkyl or C₂₋₄ alkenyl groups; R² group is independently selected from C₈₋₂₈ alkyl or alkenyl groups, and X⁻ is as defined above.

Oily sugar derivatives

15 **[0097]** The compositions for use in the invention may contain a non-cationic softening material, which is preferably an oily sugar derivative. An oily sugar derivative is a liquid or soft solid derivative of a cyclic polyol (CPE) or of a reduced saccharide (RSE), said derivative resulting from 35 to 100 % of the hydroxyl groups in said polyol or in said saccharide being esterified or etherified. The derivative has two or more ester or ether groups independently attached to a C₈-C₂₂ alkyl or alkenyl chain.

20 **[0098]** Advantageously, the CPE or RSE does not have any substantial crystalline character at 20°C. Instead it is preferably in a liquid or soft solid state as herein defined at 20°C.

[0099] The liquid or soft solid (as hereinafter defined) CPEs or RSEs suitable for use in the present invention result from 35 to 100% of the hydroxyl groups of the starting cyclic polyol or reduced saccharide being esterified or etherified with groups such that the CPEs or RSEs are in the required liquid or soft solid state. These groups typically contain

25 unsaturation, branching or mixed chain lengths.
[0100] Typically the CPEs or RSEs have 3 or more ester or ether groups or mixtures thereof, for example 3 to 8, especially 3 to 5. It is preferred if two or more of the ester or ether groups of the CPE or RSE are independently of one another attached to a C₈ to C₂₂ alkyl or alkenyl chain. The C₈ to C₂₂ alkyl or alkenyl groups may be branched or linear carbon chains.

30 **[0101]** Preferably 35 to 85 % of the hydroxyl groups, most preferably 40-80 %, even more preferably 45-75 %, such as 45-70 % are esterified or etherified.

[0102] Preferably the CPE or RSE contains at least 35 % tri or higher esters, eg at least 40%.

35 **[0103]** The CPE or RSE has at least one of the chains independently attached to the ester or ether groups having at least one unsaturated bond. This provides a cost effective way of making the CPE or RSE a liquid or a soft solid. It is preferred if predominantly unsaturated fatty chains, derived from, for example, rape oil, cotton seed oil, soybean oil, oleic, tallow, palmitoleic, linoleic, erucic or other sources of unsaturated vegetable fatty acids, are attached to the ester/ether groups.

[0104] These chains are referred to below as the ester or ether chains (of the CPE or RSE).

40 **[0105]** The ester or ether chains of the CPE or RSE are preferably predominantly unsaturated. Preferred CPEs or RSEs include sucrose tetratallowate, sucrose tetrarapeate, sucrose tetraoleate, sucrose tetraesters of soybean oil or cotton seed oil, cellobiose tetraoleate, sucrose trioleate, sucrose triapeate, sucrose pentaoleate, sucrose pentarapeate, sucrose hexaoleate, sucrose hexarapeate, sucrose triesters, pentaesters and hexaesters of soybean oil or cotton seed oil, glucose tiroleate, glucose tetraoleate, xylose trioleate, or sucrose tetra-, tri-, penta- or hexa- esters with any mixture of predominantly unsaturated fatty acid chains. The most preferred CPEs or RSEs are those with monounsaturated fatty acid chains, i.e. where any polyunsaturation has been removed by partial hydrogenation. However some CPEs or RSEs based on polyunsaturated fatty acid chains, e.g. sucrose tetralinoleate, may be used provided most of the polyunsaturation has been removed by partial hydrogenation.

45 **[0106]** The most highly preferred liquid CPEs or RSEs are any of the above but where the polyunsaturation has been removed through partial hydrogenation. Preferably 40 % or more of the fatty acid chains contain an unsaturated bond, more preferably 50 % or more, most preferably 60% or more. In most cases 65 % to 100 %, e.g. 65 % to 95 % contain an unsaturated bond.

[0107] CPEs are preferred for use with the present invention. Inositol is a preferred example of a cyclic polyol. Inositol derivatives are especially preferred.

55 **[0108]** In the context of the present invention, the term cyclic polyol encompasses all forms of saccharides. Indeed saccharides are especially preferred for use with this invention. Examples of preferred saccharides for the CPEs or RSEs to be derived from are monosaccharides and disaccharides.

[0109] Examples of monosaccharides include xylose, arabinose, galactose, fructose, sorbose and glucose. Glucose is especially preferred. Examples of disaccharides include maltose, lactose, cellobiose and sucrose. Sucrose is especially

preferred. An example of a reduced saccharide is sorbitan.

The liquid or soft solid CPEs can be prepared by methods well known to those skilled in the art. These include acylation of the cyclic polyol or reduced saccharide with an acid chloride; trans-esterification of the cyclic polyol or reduced saccharide fatty acid esters using a variety of catalysts; acylation of the cyclic polyol or reduced saccharide with an acid anhydride and acylation of the cyclic polyol or reduced saccharide with a fatty acid. See for instance US 4 386 213 and AU 14416/88 (both P&G).

[0110] It is preferred if the CPE or RSE has 3 or more, preferably 4 or more ester or ether groups. If the CPE is a disaccharide it is preferred if the disaccharide has 3 or more ester or ether groups. Particularly preferred CPEs are esters with a degree of esterification of 3 to 5, for example, sucrose tri, tetra and penta esters.

[0111] Where the cyclic polyol is a reducing sugar it is advantageous if each ring of the CPE has one ether or ester group, preferably at the C₁ position. Suitable examples of such compounds include methyl glucose derivatives.

[0112] Examples of suitable CPEs include esters of alkyl(poly)glucosides, in particular alkyl glucoside esters having a degree of polymerisation of 2.

[0113] The length of the unsaturated (and saturated if present) chains in the CPE or RSE is C₈-C₂₂, preferably C₁₂-C₂₂. It is possible to include one or more chains of C₁-C₈, however these are less preferred.

[0114] The liquid or soft solid CPEs or RSEs which are suitable for use in the present invention are characterised as materials having a solid: liquid ratio of between 50:50 and 0:100 at 20°C as determined by T₂ relaxation time NMR, preferably between 43:57 and 0:100, most preferably between 40:60 and 0:100, such as, 20:80 and 0:100. The T₂ NMR relaxation time is commonly used for characterising solid:liquid ratios in soft solid products such as fats and margarines. For the purpose of the present invention, any component of the signal with a T₂ of less than 100 μs is considered to be a solid component and any component with T₂ ≥ 100 μs is considered to be a liquid component.

[0115] For the CPEs and RSEs, the prefixes (e.g. tetra and penta) only indicate the average degrees of esterification. The compounds exist as a mixture of materials ranging from the monoester to the fully esterified ester. It is the average degree of esterification which is used herein to define the CPEs and RSEs.

[0116] The HLB of the CPE or RSE is typically between 1 and 3.

[0117] Where present, the CPE or RSE is preferably present in the composition in an amount of 0.5-50% by weight, based upon the total weight of the composition, more preferably 1-30% by weight, such as 2-25%, e.g. 2-20%.

[0118] The CPEs and RSEs for use in the compositions of the invention include sucrose tetraoleate, sucrose pentaerucate, sucrose tetraerucate and sucrose pentaoleate.

Co-softeners and fatty complexing agents

[0119] Co-softeners may be used. When employed, they are typically present at from 0.1 to 20% and particularly at from 0.5 to 10%, based on the total weight of the composition. Preferred co-softeners include fatty esters, and fatty N-oxides. Fatty esters that may be employed include fatty monoesters, such as glycerol monostearate, fatty sugar esters, such as those disclosed WO 01/46361 (Unilever).

[0120] The compositions for use in the present invention may comprise a fatty complexing agent.

[0121] Especially suitable fatty complexing agents include fatty alcohols and fatty acids. Of these, fatty alcohols are most preferred.

[0122] Fatty complexing material may be used to improve the viscosity profile of the composition.

[0123] Preferred fatty acids include hardened tallow fatty acid (available under the tradename Pristerene™, ex Uniqema). Preferred fatty alcohols include hardened tallow alcohol (available under the tradenames Stenol™ and Hydrenol™, ex Cognis and Laurex™ CS, ex Albright and Wilson).

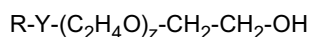
[0124] The fatty complexing agent is preferably present in an amount greater than 0.3 to 5% by weight based on the total weight of the composition. More preferably, the fatty component is present in an amount of from 0.4 to 4%. The weight ratio of the mono-ester component of the quaternary ammonium fabric softening material to the fatty complexing agent is preferably from 5:1 to 1:5, more preferably 4:1 to 1:4, most preferably 3:1 to 1:3, e.g. 2:1 to 1:2.

Non-ionic surfactant

[0125] The compositions for use in the present invention may further comprise a nonionic surfactant. Typically these can be included for the purpose of stabilising the compositions. These are particularly suitable for compositions comprising hardened quaternary ammonium compounds.

[0126] Suitable nonionic surfactants include addition products of ethylene oxide and/or propylene oxide with fatty alcohols, fatty acids and fatty amines. Any of the alkoxyated materials of the particular type described hereinafter can be used as the nonionic surfactant.

[0127] Suitable surfactants are substantially water soluble surfactants of the general formula:



where R is selected from the group consisting of primary, secondary and branched chain alkyl and/or acyl hydrocarbyl groups (when Y = -C(O)O, R ≠ an acyl hydrocarbyl group); primary, secondary and branched chain alkenyl hydrocarbyl groups; and primary, secondary and branched chain alkenyl-substituted phenolic hydrocarbyl groups; the hydrocarbyl groups having a chain length of from 8 to about 25, preferably 10 to 20, e.g. 14 to 18 carbon atoms.

[0128] In the general formula for the ethoxylated nonionic surfactant, Y is typically:



in which R has the meaning given above or can be hydrogen; and Z is at least about 8, preferably at least about 10 or 11.

[0129] Preferably the nonionic surfactant has an HLB of from about 7 to about 20, more preferably from 10 to 18, e.g. 12 to 16. Genapol™ C200 (Clariant) based on coco chain and 20 EO groups is an example of a suitable nonionic surfactant.

[0130] If present, the nonionic surfactant is present in an amount from 0.01 to 10%, more preferably 0.1 to 5 by weight, based on the total weight of the composition.

Shading Dyes

[0131] Optional shading dyes can be used. Preferred dyes are violet or blue. Suitable and preferred classes of dyes are discussed below. Moreover the unsaturated quaternary ammonium compounds are subject to some degree of UV light and/or transition metal ion catalysed radical auto-oxidation, with an attendant risk of yellowing of fabric. The presence of a shading dye also reduces the risk of yellowing from this source.

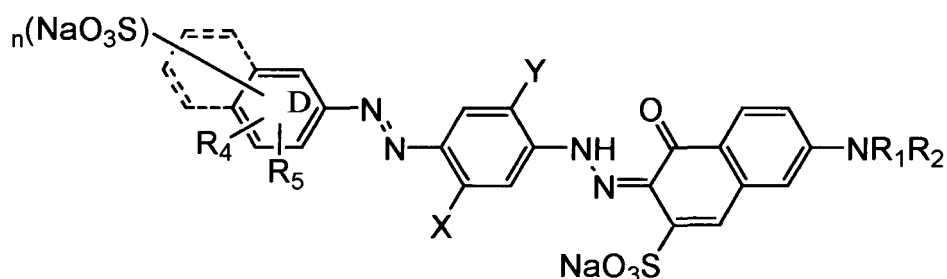
[0132] Different shading dyes give different levels of colouring. The level of shading dye present in the compositions of the present invention depend, therefore, on the type of shading dye. Preferred overall ranges, suitable for the present invention are from 0.00001 to 0.1 wt %, more preferably 0.0001 to 0.01 wt %, most preferably 0.0005 to 0.005 wt % by weight of the total composition.

Direct Dyes

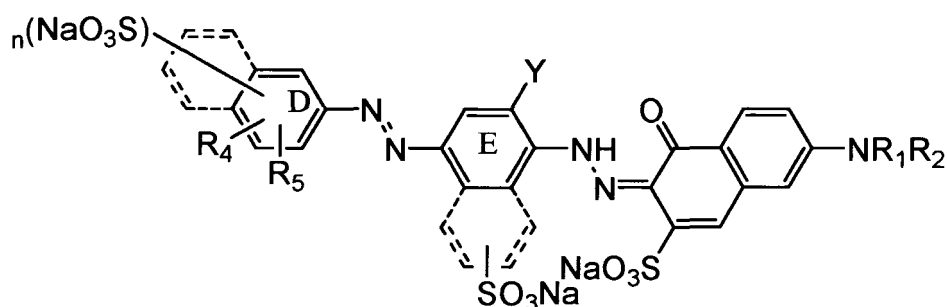
[0133] Direct dyes (otherwise known as substantive dyes) are the class of water soluble dyes which have an affinity for fibres and are taken up directly. Direct violet and direct blue dyes are preferred.

[0134] Preferably the dye are *bis-azo* or *tris-azo* dyes are used.

[0135] Most preferably, the direct dye is a direct violet of the following structures:



or



wherein:

ring D and E may be independently naphthyl or phenyl as shown;

R_1 is selected from: hydrogen and C1-C4-alkyl, preferably hydrogen;

R_2 is selected from: hydrogen, C1-C4-alkyl, substituted or unsubstituted phenyl and substituted or unsubstituted naphthyl, preferably phenyl;

R_3 and R_4 are independently selected from: hydrogen and C1-C4-alkyl, preferably hydrogen or methyl;

X and Y are independently selected from: hydrogen, C1-C4-alkyl and C1-C4-alkoxy; preferably the dye has X= methyl; and, Y = methoxy and n is 0, 1 or 2, preferably 1 or 2.

[0136] Preferred dyes are direct violet 7, direct violet 9, direct violet 11, direct violet 26, direct violet 31, direct violet 35, direct violet 40, direct violet 41, direct violet 51, and direct violet 99. Bis-azo copper containing dyes such as direct violet 66 may be used.

[0137] The benzidine based dyes are less preferred.

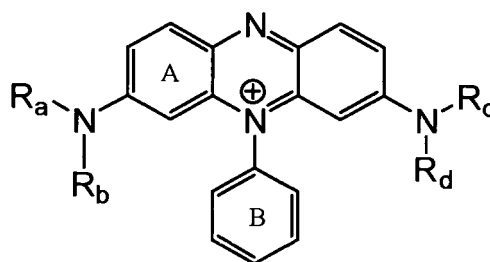
[0138] Preferably the direct dye is present at 0.00001 wt% to 0.0010 wt% of the formulation.

[0139] In another embodiment the direct dye may be covalently linked to the photo-bleach, for example as described in WO2006/024612.

Acid dyes

[0140] Cotton substantive acid dyes give benefits to cotton containing garments. Preferred dyes and mixes of dyes are blue or violet. Preferred acid dyes are:

(i) azine dyes, wherein the dye is of the following core structure:



wherein R_a , R_b , R_c and R_d are selected from: H, a branched or linear C1 to C7-alkyl chain, benzyl a phenyl, and a naphthyl;

the dye is substituted with at least one SO_3^- or $-COO^-$ group;

the B ring does not carry a negatively charged group or salt thereof;

and the A ring may further substituted to form a naphthyl;

the dye is optionally substituted by groups selected from: amine, methyl, ethyl, hydroxyl, methoxy, ethoxy, phenoxy, Cl, Br, I, F, and NO_2 .

[0141] Preferred azine dyes are: acid blue 98, acid violet 50, and acid blue 59, more preferably acid violet 50 and acid blue 98.

[0142] Other preferred non-azine acid dyes are acid violet 17, acid black 1 and acid blue 29.

[0143] Preferably the acid dye is present at 0.0005 wt% to 0.01 wt% of the formulation.

Hydrophobic dyes

[0144] The composition for use in the invention may comprise one or more hydrophobic dyes selected from benzodifuranes, methine, triphenylmethanes, naphthalimides, pyrazole, naphthoquinone, anthraquinone and mono-azo or di-azo dye chromophores. Hydrophobic dyes are dyes which do not contain any charged water solubilising group. Hydrophobic dyes may be selected from the groups of disperse and solvent dyes. Blue and violet anthraquinone and mono-azo dye are preferred.

[0145] Preferred dyes include solvent violet 13, disperse violet 27 disperse violet 26, disperse violet 28, disperse violet 63 and disperse violet 77.

[0146] Preferably the hydrophobic dye is present at 0.0001 wt% to 0.005 wt% of the formulation.

Basic dyes

[0147] Basic dyes are organic dyes which carry a net positive charge. They deposit onto cotton. They are of particular utility for used in composition that contain predominantly cationic surfactants. Dyes may be selected from the basic violet and basic blue dyes listed in the Colour Index International.

[0148] Preferred examples include triarylmethane basic dyes, methane basic dye, anthraquinone basic dyes, basic blue 16, basic blue 65, basic blue 66, basic blue 67, basic blue 71, basic blue 159, basic violet 19, basic violet 35, basic violet 38, basic violet 48; basic blue 3, basic blue 75, basic blue 95, basic blue 122, basic blue 124, basic blue 141.

Reactive dyes

[0149] Reactive dyes are dyes which contain an organic group capable of reacting with cellulose and linking the dye to cellulose with a covalent bond. They deposit onto cotton.

[0150] Preferably the reactive group is hydrolysed or reactive group of the dyes has been reacted with an organic species such as a polymer, so as to the link the dye to this species. Dyes may be selected from the reactive violet and reactive blue dyes listed in the Colour Index International.

[0151] Preferred examples include reactive blue 19, reactive blue 163, reactive blue 182 and reactive blue 96.

Dye conjugates

[0152] Dye conjugates are formed by binding direct, acid or basic dyes to polymers or particles via physical forces.

[0153] Dependent on the choice of polymer or particle they deposit on cotton or synthetics. A description is given in WO2006/055787. They are not preferred.

[0154] Particularly preferred dyes are: direct violet 7, direct violet 9, direct violet 11, direct violet 26, direct violet 31, direct violet 35, direct violet 40, direct violet 41, direct violet 51, direct violet 99, acid blue 98, acid violet 50, acid blue 59, acid violet 17, acid black 1, acid blue 29, solvent violet 13, disperse violet 27 disperse violet 26, disperse violet 28, disperse violet 63, disperse violet 77 and mixtures thereof.

Further Optional Ingredients

[0155] The compositions for use in the invention may contain one or more other ingredients. Such ingredients include further preservatives (e.g. bactericides), pH buffering agents, perfume carriers, hydrotropes, anti-redeposition agents, soil-release agents, polyelectrolytes, anti-shrinking agents, anti-wrinkle agents, antioxidants, sunscreens, anti-corrosion agents, drape imparting agents, anti-static agents, ironing aids, silicones, antifoams, colourants, pearlisers and/or opacifiers, natural oils/extracts, processing aids, e.g. electrolytes, hygiene agents, e.g. antibacterials and antifungals, thickeners and skin benefit agents.

[0156] The Fabric softening compositions may also comprise viscosity modifiers. Suitable viscosity modifiers are disclosed, for example, in WO 02/081611, US 2004/0214736, US 6827795, EP 0501714, US 2003/0104964, EP 0385749 and EP 331237.

Product Form

[0157] The compositions for use in the present invention are preferably rinse-added softening compositions.

[0158] The compositions have a pH ranging from about 2.5 to 6, preferably from about 2.5 to 4.5, most preferably about 2.5 to 2.8. The compositions for use in the invention may also contain pH modifiers such as hydrochloric acid or lactic acid.

[0159] A composition for use in the invention is preferably in liquid form. The composition may be a concentrate to be diluted in a solvent, including water, before use. The composition may also be a ready-to-use (in-use) composition. Preferably the composition is provided as a ready to use liquid comprising an aqueous phase. The aqueous phase may comprise water-soluble species, such as mineral salts or short chain (C₁₋₄) alcohols.

[0160] The composition is preferably for use in the rinse cycle of a home textile laundering operation, where, it may be added directly in an undiluted state to a washing machine, e.g. through a dispenser drawer or, for a top-loading washing machine, directly into the drum. Alternatively, it can be diluted prior to use. The compositions may also be used in a domestic hand-washing laundry operation. It is also possible for the compositions of the present invention to be used in industrial laundry operations, e.g. as a finishing agent for softening new clothes prior to sale to consumers.

Preparation

[0161] Compositions used in the invention can be prepared by any method suitable for preparing dispersed, emulsified systems. One method involves the forming of a molten premixture of the active materials in water at an elevated temperature, adding additional water to obtain the desired active concentration, and then cooling to ambient temperature. When desired, some minor ingredients such as electrolytes, colouring agents, etc. may be post-dosed. A second method involves the forming of the product by phase inversion of a water in hydrocarbon emulsion, wherein the cationic material is either part of the hydrocarbon phase or added as a separate predispersion. This method is advantageous, because this provides very finely divided hydrocarbon particles in the final product. In an alternative method the encapsulated phase change active may be post dosed in the form of an aqueous slurry.

Examples

[0162] Embodiments of the invention will now be illustrated by the following non-limiting examples. Further modifications will be apparent to the person skilled in the art.

[0163] Examples of the invention are represented by a number. Comparative examples are represented by a letter.

[0164] Unless otherwise stated, amounts of components are expressed as a percentage of the total weight of the composition.

Example 1:- Preparation and composition of Fabric Conditioner 1, for use in accordance with the invention, and Comparative Example A.

[0165] Composition 1 and Comparative Example A were dilute liquid fabric conditioners, comprising 4.5 % of softening active. The compositions are shown in Table 1.

Table 1: Composition of Fabric Conditioner 1 (wt %, based on 100 % active ingredients)

Ingredient	Tradename	Supplier	1	A
Softener active	TEP-88L ¹	FXG	5.8	5.8
Fatty alcohol	Cetostearyl alcohol	-	0.2	0.2
Acid	1M HCl	-	0.009	0.009
Antifoam ³	Wacker SRE	Wacker	0.005	0.005
Dye	Liquitint dye	Milliken	0.002	0.002
Salt	CaCl ₂ (10% Soln.)	-	0.00125	0.00125
Perfume	-	-	0.8	0.8
Encapsulated perfume	-	-	0.25	0.25
Encapsulated phase change material	Lurapret TXPMC 28	-	5.0	-
Water and minors ²	-	-	To 100 %	To 100 %
¹ Palm based soft TEA Quat ² Pearlescer, preservative, sequestrant, etc ³ Based on 100 % activity				

Example 2:- Treatment of Garments using Composition 1 and Comparative Example ATest garments

[0166] Black trousers and white tops were used as test garments. Garments for the study were tailor-made for each participant to ensure consistency of garment design and fit across all participants.

[0167] All test garments were first washed in commercially available washing powder in an automatic washing machine and line dried. The wash conditions were as follows;

Treatment with Composition 1 and Comparative Example A

[0168] Garments were added to an automatic washing machine and the rinse cycle selected. 42 g of the fabric conditioner was added to the washing machine drawer. The garments were then line dried.

Panel Test Method and Conditions

[0169] A blind, unbranded test regime was adopted. The test took place during a hot and humid period in Bangkok and involved 30 participants wearing the test garments all day. Each participant wore garments treated with the comparative example and composition 1 on separate days. Control garments (wash only) were also included. Participants were not asked to compare one set of garments to another. They went about their normal routine of travelling to and from work, having their lunch outdoors, and working indoors in air-conditioned offices at other times. The weather was monitored during the experiment and was found to be relatively consistent, averaging around 32°C/50% RH at midday.

[0170] Participants recorded fresh smell as well as perfume intensity levels at the event of onset of sweating.

[0171] Statistical analysis was carried out using a repeated measurement model fitted to the data; "repeated" meaning that each participant in the study was measured several times. Such a model incorporates the time and also reflects that there are several layers of random influences present in the way the data was collected. These layers are: the "within participant" variation over time, the "between participant" variation due to different features of the participant and treatment and the overall external random influences and measurement errors.

[0172] A linear effects model was used to describe the three sources of variability. This model consists of a classical linear model (some function of the controlled and observed influences) and a random effects model. Only after fitting both is the error variance determined. Therefore, the output of the model is a functional description of the population trend and, for each individual, a fit of the deviation from this trend.

[0173] The statistics package R (version 3.1-94) was used for the repeated measurement and linear mixed effects models.

[0174] The p values shown below indicate Confidence at 95% Significance. The confidence for the perfume intensity was generated from the on-set of sweating event data.

Example 3:- Fresh smell and perfume intensity on sweating imparted by garments treated with Composition 1 and Comparative Example A

[0175]

Table 2: Fresh smell and perfume intensity levels at the onset of sweating for garments treated with comparative example A and Composition 1.

Treatment	Intensity Rating	
	Fresh Smell	Perfume
A	2.53	-2.20
1	14.35	14.72
Confidence	P <0.05	P <0.005

[0176] It will be seen that garments treated in accordance with the invention conferred an enhanced effect from the perfume encaps.

Claims

1. Use of an encapsulated phase change active, having a phase transition temperature of from 24 to 39°C, to improve the beneficial effect of an additional encapsulated volatile benefit agent in the presence of an additional non-encapsulated volatile benefit agent, wherein the encapsulated volatile benefit agent is selected from perfume, insect repellent, aromatherapy oil, sensates such as menthol and an essential oil, and wherein the phase change active is a paraffin wax comprising n-octadecane.
2. Use as claimed in claim 1, wherein the non-encapsulated volatile benefit agent is selected from a perfume, insect repellent, aromatherapy oil, sensates such as menthol and an essential oil.

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3. Use according to any preceding claim, wherein the phase change active comprises hydrocarbon materials comprising a linear or branched alkyl chain comprising an average of from 12 to 50 carbon atoms per molecule.
- 5 4. Use according to claim any preceding claim, wherein the phase change active comprises a mixture of mineral oil and petroleum jelly.
5. Use as claimed in any preceding claim, wherein the encapsulated phase change material has a particle size of from 10 nm to 1000 microns.
- 10 6. Use as claimed in any preceding claim, wherein the encapsulated phase change material and the additional encapsulated volatile benefit agent are components of a laundry composition which further comprises an additional non-encapsulated volatile benefit agent.
- 15 7. Use as claimed in any preceding claim in which the laundry composition is a fabric conditioner which comprises a fabric softening agent.
8. Use as claimed in claim 7 wherein the fabric softening agent is selected from an oily sugar derivative, a cationic fabric softening compound and mixture thereof.
- 20 9. Use as claimed in claim 8 in which the cationic fabric softening agent is a quaternary ammonium compound having at least two C_{12-28} groups connected to the nitrogen head group that may independently be alkyl or alkenyl groups, preferably being connected to the nitrogen head group by an ester link.
- 25 10. Use as claimed in any one of claims 6 to 9, wherein the additional non-encapsulated volatile benefit agent is the same as the encapsulated volatile benefit agent.
11. Use as claimed in any one of claims 6 to 10 in which the phase change material is present in an amount of from 0.05 to 50 wt % by weight of the composition.
- 30 12. Use as claimed in any one of claims 6 to 11, wherein the total amount of volatile benefit agent is from 0.01 to 10 % by weight, based on the total weight of the composition.

Patentansprüche

- 35 1. Verwendung eines eingekapselten Phasenumwandlungsmittels mit einer Phasenübergangstemperatur von 24 bis 39°C zur Verbesserung des Nutzeffekts eines zusätzlichen eingekapselten flüchtigen Nutzmittels in der Gegenwart eines zusätzlichen nicht-eingekapselten flüchtigen Nutzmittels, wobei das eingekapselte flüchtige Nutzmittel unter Duftstoff, Insektenschutzmittel, Aromatherapieöl, empfindungswirksamen Mitteln, wie Menthol und ein ätherisches Öl, ausgewählt ist und wobei das Phasenumwandlungsmittel ein n-Octadecan umfassendes Paraffinwachs ist.
- 40 2. Verwendung nach Anspruch 1, wobei das nicht-eingekapselte flüchtige Nutzmittel unter Duftstoff, Insektenschutzmittel, Aromatherapieöl, empfindungswirksamen Mitteln, wie Menthol und ein ätherisches Öl, ausgewählt ist.
- 45 3. Verwendung nach irgendeinem vorhergehenden Anspruch, wobei das Phasenumwandlungsmittel Kohlenwasserstoffmaterialien, umfassend eine lineare oder verzweigte Alkylkette, umfassend im Durchschnitt 12 bis 50 Kohlenstoffatome pro Molekül, einbezieht.
- 50 4. Verwendung nach irgendeinem vorhergehenden Anspruch, wobei das Phasenumwandlungsmittel eine Mischung von Mineralöl und Petrolat umfasst.
5. Verwendung nach irgendeinem vorhergehenden Anspruch, wobei das eingekapselte Phasenumwandlungsmaterial eine Teilchengröße von 10 nm bis 1000 Mikrometer aufweist.
- 55 6. Verwendung nach irgendeinem vorhergehenden Anspruch, wobei das eingekapselte Phasenumwandlungsmaterial und das zusätzliche eingekapselte flüchtige Nutzmittel Bestandteile einer Waschmittelzusammensetzung sind, die des Weiteren ein zusätzliches nicht-eingekapseltes flüchtiges Nutzmittel umfasst.

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7. Verwendung nach irgendeinem vorhergehenden Anspruch, wobei die Waschmittelzusammensetzung ein Textil-
konditioniermittel ist, das einen Textilweichspüler umfasst.
- 5 8. Verwendung nach Anspruch 7, wobei der Textilweichspüler unter einem ölartigen Zuckerderivat, einem kationischen
Textilweichspüler und einer Mischung davon ausgewählt ist.
9. Verwendung nach Anspruch 8, in der der kationische Textilweichspüler eine quaternäre Ammoniumverbindung mit
mindestens zwei C₁₂₋₂₈-Gruppen ist, verbunden mit der Stickstoffkopfguppe, die unabhängig voneinander Alkyl-
oder Alkenylgruppen sein können, vorzugsweise mit der Stickstoffkopfguppe über eine Esterbindung verbunden.
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10. Verwendung nach irgendeinem der Ansprüche 6 bis 9, wobei das zusätzliche nicht-eingekapselte flüchtige Nutzmittel
das Gleiche wie das eingekapselte flüchtige Nutzmittel ist.
11. Verwendung nach irgendeinem der Ansprüche 6 bis 10, wobei das Phasenumwandlungsmaterial in einer Menge
von 0,05 bis 50 Gewichts-% der Zusammensetzung vorliegt.
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12. Verwendung nach irgendeinem der Ansprüche 6 bis 11, wobei die gesamte Menge des flüchtigen Nutzmittels 0,01
bis 10 Gewichts-%, bezogen auf das Gesamtgewicht der Zusammensetzung, beträgt.

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Revendications

1. Utilisation d'un actif de changement de phase encapsulé, présentant une température de transition de phase de 24
à 39 °C, pour améliorer l'effet bénéfique d'un agent bénéfique volatil encapsulé supplémentaire en présence d'un
25 agent bénéfique volatil non-encapsulé supplémentaire, dans laquelle l'agent bénéfique volatil encapsulé est choisi
parmi un parfum, un répulsif d'insecte, une huile d'aromathérapie, des agents communiquant une sensation, tels
que du menthol et une huile essentielle, et dans laquelle l'actif de changement de phase est une cire de paraffine
comprenant du n-octadécane.
- 30 2. Utilisation selon la revendication 1, dans laquelle l'agent bénéfique volatil non-encapsulé est choisi parmi un parfum,
un répulsif d'insecte, une huile d'aromathérapie, des agents communiquant une sensation, tels que le menthol et
une huile essentielle.
3. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle l'actif de changement de phase
35 comprend des matériaux hydrocarbonés comprenant une chaîne alkyle linéaire ou ramifiée comprenant une moyenne
de 12 à 50 atomes de carbone par molécule.
4. Utilisation selon l'une quelconque des revendications précédentes, dans lequel l'actif de changement de phase
comprend un mélange d'huile minérale et de gelée de pétrole.
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5. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle le matériau de changement de
phase encapsulé présente une taille de particule de 10 nm à 1 000 microns.
6. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle le matériau de changement de
45 phase encapsulé et l'agent bénéfique volatil encapsulé supplémentaire sont des constituants d'une composition de
lavage qui comprend de plus un agent bénéfique volatil non-encapsulé supplémentaire.
7. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle la composition de lavage est un
conditionneur de textile qui comprend un agent adoucissant de textile.
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8. Utilisation selon la revendication 7, dans laquelle l'agent adoucissant de textile est choisi parmi un dérivé de sucre
huileux, un composé adoucissant de textile cationique et un mélange de ceux-ci.
9. Utilisation selon la revendication 8, dans laquelle l'agent adoucissant de textile cationique est un composé d'am-
55 monium quaternaire présentant au moins deux groupes en C₁₂₋₂₈ connectés au groupe de tête d'azote qui peuvent
être indépendamment des groupes alkyle ou alcényle, de préférence connectés au groupe de tête d'azote par une
liaison ester.

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10. Utilisation selon l'une quelconque des revendications 6 à 9, dans laquelle l'agent bénéfique volatil non-encapsulé supplémentaire est identique à l'agent bénéfique volatil encapsulé.
11. Utilisation selon l'une quelconque des revendications 6 à 10, dans laquelle le matériau de changement de phase est présent dans une quantité de 0,05 à 50 % en masse en masse de la composition.
12. Utilisation selon l'une quelconque des revendications 6 à 11, dans laquelle la quantité totale d'agent bénéfique volatil est de 0,01 à 10 % en masse, rapportée à la masse totale de la composition.

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