

## PROCESS FOR PREPARING POLYESTER MICROCAPSULES

### Technical Field

5 The present invention relates to a new process for the preparation of polyester microcapsules. Microcapsules are also an object of the invention. Consumer products comprising said microcapsules, in particular perfumed consumer products or flavoured consumer products are also part of the invention.

### Background of the Invention

10 One of the problems faced by the perfume and flavour industry lies in the relatively rapid loss of olfactive benefit provided by active compounds due to their volatility. The encapsulation of those active substances provides at the same time a protection of the ingredients there-encapsulated against “aggressions” such as oxidation or moisture and allows, on the other hand, a certain control of the kinetics of flavour or fragrance release to induce sensory effects through  
15 sequential release.

Polyurea and polyurethane-based microcapsule slurry are widely used for example in perfumery industry for instance as they provide a long lasting pleasant olfactory effect after their applications on different substrates. Those microcapsules have been widely disclosed in the prior art (see for example WO2007/004166 or EP 2300146 from the Applicant).

20 In addition to the performance in terms of stability and olfactive performance, the consumer demand for eco-friendly delivery systems is more and more important and is driving the development of new delivery systems.

There is therefore still a need to provide new microcapsules using more eco-friendly materials, while not compromising on the performance of the microcapsules, in particular in terms  
25 of stability in a challenging medium such as a consumer product base, as well as in delivering a good performance in terms of hydrophobic material delivery, e.g. olfactive performance in the case of perfuming ingredients.

The present invention is proposing a solution to the above-mentioned problem, based on new polyester microcapsules.

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**Detailed description of the Invention**

Unless stated otherwise, percentages (%) are meant to designate a percentage by weight of a composition.

By “**hydrophobic material**”, it is meant any hydrophobic material – single material or a mixture of material - which forms a two-phase dispersion when mixed with water.

By “**ingredient**”, it is meant a single compound or a combination of ingredients.

By “**perfume or flavor oil**”, it is meant a single perfuming or flavoring compound or a mixture of several perfuming or flavoring compounds.

By “**consumer product**” or “**end-product**” it is meant a manufactured product ready to be distributed, sold and used by a consumer.

By “**polyester microcapsule**” it is meant that the polymer comprises ester linkages by the polyol capable of further reacting with a carboxylic acid derivative, for example with the acyl chloride.

By “**polyol**”, it is meant a compound having at least two hydroxyl functions.

By “**carboxylic acid derivative**”, it means a compound with an acyl group bounded to an electronegative atom or substituent, -X, that can act as a leaving group in substitution reactions. Electronegative atom may be for example alkyl amine, alkyl alcohol, amine or hydroxyl group, and -X may be chlorides, bromides, tosylates, mesylates, carboxylic acids (as anhydrides).

For the sake of clarity, by the expression “**dispersion**” in the present invention it is meant a system in which particles and/or oil droplets are dispersed in a continuous phase of a different composition and it specifically includes a suspension or an emulsion.

A “**core-shell microcapsule**”, or the similar, in the present invention it is meant that capsules have a particle size distribution in the micron range (e.g. a mean diameter (d(v, 0.5)) preferably comprised between about 1 and 3000 microns, preferably between 1 and 500 microns) and comprise a shell and an internal continuous oil phase enclosed by the shell. According to the invention, the wordings “mean diameter” or “mean size” are used indifferently.

By “**microcapsule slurry**”, it is meant microcapsule(s) that is (are) dispersed in a liquid. According to an embodiment, the microcapsule(s) is (are) dispersed in a hydrophilic phase.

By “**a carboxylic acid derivative**”, it should be understood a single carboxylic acid derivative or a mixture of carboxylic acid derivatives.

It has been found that performing polyester microcapsules could be obtained based on the reaction between a carboxylic acid derivative and a polyol.

A first object of the invention is therefore a process for preparing a core-shell polyester microcapsule slurry, said process comprising the steps of:

- (i) Dispersing an oil phase O1 comprising a hydrophobic material H1, into a continuous phase comprising a polyol to obtain a two-phases dispersion E1, wherein the oil phase O1 and/or the continuous phase comprise a stabilizer S1, and
  - (ii) applying conditions sufficient to induce interfacial polymerization and form polyester microcapsules in the form of a slurry,
- wherein at least one carboxylic acid derivative A1 is added into E1 after step i) and/or at least one carboxylic acid derivative A2 is added into the oil phase O1.

In a step of the process of the invention, an oil phase O1 comprising a hydrophobic material H1 is dispersed into a continuous phase comprising a polyol to obtain a two-phases dispersion E1, wherein the oil phase and/or the continuous phase comprise a stabilizer S1 and wherein at least one carboxylic acid derivative A1 is added into E1 after step i) and/or at least one carboxylic acid derivative A2 is added into the oil phase O1.

According to a particular embodiment, the process comprises the following steps:

- (i) Dispersing an oil phase O1 comprising a hydrophobic material H1, into a continuous phase comprising a polyol to obtain a two-phases dispersion E1, wherein the oil phase and/or the continuous phase comprise a stabilizer S1 and,
  - (ii) applying conditions sufficient to induce interfacial polymerization and form polyester microcapsules in the form of a slurry,
- wherein at least one carboxylic acid derivative A1 is added into E1 after step i).

According to a particular embodiment, the process comprises the following steps:

- (i) Dispersing an oil phase O1 comprising a hydrophobic material H1, into a continuous phase comprising a polyol to obtain a two-phases dispersion E1, wherein the oil phase and/or the continuous phase comprise a stabilizer S1 and,

- (ii) applying conditions sufficient to induce interfacial polymerization and form polyester microcapsules in the form of a slurry,

wherein at least one carboxylic acid derivative A2 is added into the oil phase O1.

5 According to a particular embodiment, the process comprises the following steps:

- (i) Dispersing an oil phase O1 comprising a hydrophobic material H1, into a continuous phase comprising a polyol to obtain a two-phases dispersion E1, wherein the oil phase and/or the continuous phase comprise a stabilizer S1 and,

- (ii) applying conditions sufficient to induce interfacial polymerization and form polyester microcapsules in the form of a slurry,

10 wherein at least one carboxylic acid derivative A1 is added into E1 after step i) and optionally at least a carboxylic acid derivative A2 is added into the oil phase O1.

According to a particular embodiment, at least one carboxylic acid derivative A1 is added into E1

15 after step i) and at least a carboxylic acid derivative A2 is added into the oil phase O1.

According to an embodiment, no amine, preferably no tertiary amine is added at any step of the process.

## 20 Oil phase O1

According to the invention, the oil phase O1 comprises a hydrophobic material H1.

According to a particular embodiment, the oil phase O1 consists of a hydrophobic material H1.

The hydrophobic material according to the invention can be “inert” material like solvents or active ingredients. A single hydrophobic material or mixture of hydrophobic materials can be used.

When the hydrophobic material is an active ingredient, it is preferably chosen from the group consisting of flavor, flavor ingredients, perfume, perfume ingredients, nutraceuticals, cosmetics, pest control agents, biocide actives, malodour counteracting ingredient, bactericide ingredient, fungicide ingredient, pharmaceutical or agrochemical ingredient, a sanitizing ingredient, an insect repellent or attractant, and mixtures thereof.

According to a particular embodiment, the hydrophobic material comprises a phase change material (PCM).

According to a particular embodiment, the hydrophobic material comprises a mixture of a perfume with another ingredient selected from the group consisting of nutraceuticals, cosmetics, pest control agents and biocide actives.

According to a particular embodiment, the hydrophobic material comprises a mixture of biocide actives with another ingredient selected from the group consisting of perfume, nutraceuticals, cosmetics, pest control agents.

According to a particular embodiment, the hydrophobic material comprises a mixture of pest control agents with another ingredient selected from the group consisting of perfume, nutraceuticals, cosmetics, biocide actives.

According to a particular embodiment, the hydrophobic material comprises a perfume.

According to a particular embodiment, the hydrophobic material consists of a perfume.

According to a particular embodiment, the hydrophobic material consists of biocide actives.

According to a particular embodiment, the hydrophobic material consists of pest control agents.

By “perfume” (or also “perfume oil”) what is meant here is an ingredient or a composition that is a liquid at about 20°C. According to any one of the above embodiments said perfume oil can be a perfuming ingredient alone or a mixture of ingredients in the form of a perfuming composition. As a “perfuming ingredient” it is meant here a compound, which is used for the primary purpose of conferring or modulating an odor. In other words such an ingredient, to be considered as being a perfuming one, must be recognized by a person skilled in the art as being able to at least impart or modify in a positive or pleasant way the odor of a composition, and not just as having an odor. For the purpose of the present invention, perfume oil also includes a combination of perfuming ingredients with substances which together improve, enhance or modify the delivery of the perfuming ingredients, such as perfume precursors, modulators, emulsions or dispersions, as well as combinations which impart an additional benefit beyond that of modifying or imparting an odor, such as long-lastingness, blooming, malodor counteraction, antimicrobial effect, microbial stability, pest control.

The nature and type of the perfuming ingredients present in the oil phase do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled person being able to select them on the basis of its general knowledge and according to intended use or application and the desired organoleptic effect. In general terms, these perfuming ingredients  
 5 belong to chemical classes as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitriles, terpenoids, nitrogenous or sulfurous heterocyclic compounds and essential oils (for example thyme oil), and said perfuming co-ingredients can be of natural or synthetic origin. Many of these co-ingredients are in any case listed in reference texts such as the book by S. Arctander, Perfume and Flavor Chemicals, 1969, Montclair, New Jersey, USA, or its more recent versions,  
 10 or in other works of a similar nature, as well as in the abundant patent literature in the field of perfumery.

In particular one may cite perfuming ingredients which are commonly used in perfume formulations, such as:

- Aldehydic ingredients: decanal, dodecanal, 2-methyl-undecanal, 10-undecenal, octanal,  
 15 nonanal and/or nonenal;
- Aromatic-herbal ingredients: eucalyptus oil, camphor, eucalyptol, 5-methyltricyclo[6.2.1.0<sup>2,7</sup>]undecan-4-one, 1-methoxy-3-hexanethiol, 2-ethyl-4,4-dimethyl-1,3-oxathiane, 2,2,7/8,9/10-tetramethylspiro[5.5]undec-8-en-1-one, menthol and/or alpha-pinene;
- 20 - Balsamic ingredients: coumarin, ethylvanillin and/or vanillin;
- Citrus ingredients: dihydromyrcenol, citral, orange oil, linalyl acetate, citronellyl nitrile, orange terpenes, limonene, 1-p-menthen-8-yl acetate and/or 1,4(8)-p-menthadiene;
- Floral ingredients: methyl dihydrojasmonate, linalool, citronellol, phenylethanol, 3-(4-tert-butylphenyl)-2-methylpropanal, hexylcinnamic aldehyde, benzyl acetate, benzyl  
 25 salicylate, tetrahydro-2-isobutyl-4-methyl-4(2H)-pyranol, beta ionone, methyl 2-(methylamino)benzoate, (E)-3-methyl-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-3-buten-2-one, (1E)-1-(2,6,6-trimethyl-2-cyclohexen-1-yl)-1-penten-3-one, 1-(2,6,6-trimethyl-1,3-cyclohexadien-1-yl)-2-buten-1-one, (2E)-1-(2,6,6-trimethyl-2-cyclohexen-1-yl)-2-buten-1-one, (2E)-1-[2,6,6-trimethyl-3-cyclohexen-1-yl]-2-buten-1-one, (2E)-1-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-buten-1-one,  
 30 3-(3,3/1,1-dimethyl-5-indanyl)propanal, 2,5-dimethyl-2-indanmethanol, 2,6,6-trimethyl-3-cyclohexene-1-carboxylate, 3-(4,4-

- dimethyl-1-cyclohexen-1-yl)propanal, hexyl salicylate, 3,7-dimethyl-1,6-nonadien-3-ol, 3-(4-isopropylphenyl)-2-methylpropanal, verdyl acetate, geraniol, p-menth-1-en-8-ol, 4-(1,1-dimethylethyl)-1-cyclohexyle acetate, 1,1-dimethyl-2-phenylethyl acetate, 4-cyclohexyl-2-methyl-2-butanol, amyl salicylate, high cis methyl dihydrojasmonate, 3-methyl-5-phenyl-1-pentanol, verdyl propionate, geranyl acetate, tetrahydro linalool, cis-7-p-menthanol, propyl (S)-2-(1,1-dimethylpropoxy)propanoate, 2-methoxynaphthalene, 2,2,2-trichloro-1-phenylethyl acetate, 4/3-(4-hydroxy-4-methylpentyl)-3-cyclohexene-1-carbaldehyde, amylcinnamic aldehyde, 8-decen-5-olide, 4-phenyl-2-butanone, isononyl acetate, 4-(1,1-dimethylethyl)-1-cyclohexyl acetate, verdyl isobutyrate and/or mixture of methylionones isomers;
- 10 - Fruity ingredients: gamma-undecalactone, 2,2,5-trimethyl-5-pentylcyclopentanone, 2-methyl-4-propyl-1,3-oxathiane, 4-decanolide, ethyl 2-methyl-pentanoate, hexyl acetate, ethyl 2-methylbutanoate, gamma-nonolactone, allyl heptanoate, 2-phenoxyethyl isobutyrate, ethyl 2-methyl-1,3-dioxolane-2-acetate, diethyl 1,4-cyclohexanedicarboxylate, 3-methyl-2-hexen-1-yl acetate, 1-[3,3-dimethylcyclohexyl]ethyl [3-ethyl-2-oxiranyl]acetate and/or diethyl 1,4-cyclohexane dicarboxylate;
- 15 - Green ingredients: 2-methyl-3-hexanone (E)-oxime, 2,4-dimethyl-3-cyclohexene-1-carbaldehyde, 2-tert-butyl-1-cyclohexyl acetate, styrallyl acetate, allyl (2-methylbutoxy)acetate, 4-methyl-3-decen-5-ol, diphenyl ether, (Z)-3-hexen-1-ol and/or 1-(5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one;
- 20 - Musk ingredients: 1,4-dioxo-5,17-cycloheptadecanedione, (Z)-4-cyclopentadecen-1-one, 3-methylcyclopentadecanone, 1-oxa-12-cyclohexadecen-2-one, 1-oxa-13-cyclohexadecen-2-one, (9Z)-9-cycloheptadecen-1-one, 2-[(1S)-1-[(1R)-3,3-dimethylcyclohexyl]ethoxy]-2-oxoethyl propionate, 3-methyl-5-cyclopentadecen-1-one, 4,6,6,7,8-hexamethyl-1,3,4,6,7,8-hexahydrocyclopenta[g]isochromene, (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl propanoate, oxacyclohexadecan-2-one and/or (1S,1'R)-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxycarbonyl]methyl propanoate;
- 25 - Woody ingredients: 1-[(1RS,6SR)-2,2,6-trimethylcyclohexyl]-3-hexanol, 3,3-dimethyl-5-[(1R)-2,2,3-trimethyl-3-cyclopenten-1-yl]-4-penten-2-ol, 3,4'-dimethylspiro[oxirane-2,9'-tricyclo[6.2.1.0<sup>2,7</sup>]undec[4]ene, (1-ethoxyethoxy)cyclododecane, 2,2,9,11-
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- tetramethylspiro[5.5]undec-8-en-1-yl acetate, 1-(octahydro-2,3,8,8-tetramethyl-2-naphthalenyl)-1-ethanone, patchouli oil, terpenes fractions of patchouli oil, Clearwood®, (1'R,E)-2-ethyl-4-(2',2',3'-trimethyl-3'-cyclopenten-1'-yl)-2-buten-1-ol, 2-ethyl-4-(2,2,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol, methyl cedryl ketone, 5-(2,2,3-trimethyl-3-cyclopentenyl)-3-methylpentan-2-ol, 1-(2,3,8,8-tetramethyl-1,2,3,4,6,7,8,8a-octahydronaphthalen-2-yl)ethan-1-one and/or isobornyl acetate;
- Other ingredients (e.g. amber, powdery spicy or watery): dodecahydro-3a,6,6,9a-tetramethyl-naphtho[2,1-b]furan and any of its stereoisomers, heliotropin, anisic aldehyde, eugenol, cinnamic aldehyde, clove oil, 3-(1,3-benzodioxol-5-yl)-2-methylpropanal, 7-methyl-2H-1,5-benzodioxepin-3(4H)-one, 2,5,5-trimethyl-1,2,3,4,4a,5,6,7-octahydro-2-naphthalenol, 1-phenylvinyl acetate, 6-methyl-7-oxa-1-thia-4-azaspiro[4.4]nonane and/or 3-(3-isopropyl-1-phenyl)butanal.
- It is also understood that said ingredients may also be compounds known to release in a controlled manner various types of perfuming compounds also known as properfume or profragrance. Non-limiting examples of suitable properfumes may include 4-(dodecylthio)-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-2-butanone, 4-(dodecylthio)-4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-butanone, 3-(dodecylthio)-1-(2,6,6-trimethyl-3-cyclohexen-1-yl)-1-butanone, 2-(dodecylthio)octan-4-one, 2-phenylethyl oxo(phenyl)acetate, 3,7-dimethylocta-2,6-dien-1-yl oxo(phenyl)acetate, (Z)-hex-3-en-1-yl oxo(phenyl)acetate, 3,7-dimethyl-2,6-octadien-1-yl hexadecanoate, bis(3,7-dimethylocta-2,6-dien-1-yl) succinate, (2-((2-methylundec-1-en-1-yl)oxy)ethyl)benzene, 1-methoxy-4-(3-methyl-4-phenethoxybut-3-en-1-yl)benzene, (3-methyl-4-phenethoxybut-3-en-1-yl)benzene, 1-(((Z)-hex-3-en-1-yl)oxy)-2-methylundec-1-ene, (2-((2-methylundec-1-en-1-yl)oxy)ethoxy)benzene, 2-methyl-1-(octan-3-yloxy)undec-1-ene, 1-methoxy-4-(1-phenethoxyprop-1-en-2-yl)benzene, 1-methyl-4-(1-phenethoxyprop-1-en-2-yl)benzene, 2-(1-phenethoxyprop-1-en-2-yl)naphthalene, (2-phenethoxyvinyl)benzene, 2-(1-((3,7-dimethyloct-6-en-1-yl)oxy)prop-1-en-2-yl)naphthalene, (2-((2-pentylcyclopentylidene)methoxy)ethyl)benzene, 4-allyl-2-methoxy-1-((2-methoxy-2-phenylvinyl)oxy)benzene, (2-((2-heptylcyclopentylidene)methoxy)ethyl)benzene, 1-isopropyl-4-methyl-2-((2-pentylcyclopentylidene)methoxy)benzene, 2-methoxy-1-((2-pentylcyclopentylidene)methoxy)-4-propylbenzene, 3-methoxy-4-((2-methoxy-2-

phenylvinyl)oxy)benzaldehyde, 4-((2-(hexyloxy)-2-phenylvinyl)oxy)-3-methoxybenzaldehyde or a mixture thereof.

The perfuming ingredients may be dissolved in a solvent of current use in the perfume industry. The solvent is preferably not an alcohol. Examples of such solvents are diethyl phthalate, isopropyl myristate, Abalyn<sup>®</sup> (rosin resins, available from Eastman), benzyl benzoate, ethyl citrate, triethyl citrate, limonene or other terpenes, or isoparaffins. Preferably, the solvent is very hydrophobic and highly sterically hindered, like for example Abalyn<sup>®</sup> or benzyl benzoate. Preferably the perfume comprises less than 30% of solvent. More preferably the perfume comprises less than 20% and even more preferably less than 10% of solvent, all these percentages being defined by weight relative to the total weight of the perfume. Most preferably, the perfume is essentially free of solvent.

Preferred perfuming ingredients are those having a high steric hindrance (bulky materials) and in particular those from one of the following groups:

- Group 1: perfuming ingredients comprising a cyclohexane, cyclohexene, cyclohexanone or cyclohexenone ring substituted with at least one linear or branched C<sub>1</sub> to C<sub>4</sub> alkyl or alkenyl substituent;
- Group 2: perfuming ingredients comprising a cyclopentane, cyclopentene, cyclopentanone or cyclopentenone ring substituted with at least one linear or branched C<sub>4</sub> to C<sub>8</sub> alkyl or alkenyl substituent;
- Group 3: perfuming ingredients comprising a phenyl ring or perfuming ingredients comprising a cyclohexane, cyclohexene, cyclohexanone or cyclohexenone ring substituted with at least one linear or branched C<sub>5</sub> to C<sub>8</sub> alkyl or alkenyl substituent or with at least one phenyl substituent and optionally one or more linear or branched C<sub>1</sub> to C<sub>3</sub> alkyl or alkenyl substituents;
- Group 4: perfuming ingredients comprising at least two fused or linked C<sub>5</sub> and/or C<sub>6</sub> rings;
- Group 5: perfuming ingredients comprising a camphor-like ring structure;
- Group 6: perfuming ingredients comprising at least one C<sub>7</sub> to C<sub>20</sub> ring structure;
- Group 7: perfuming ingredients having a logP value above 3.5 and comprising at least one tert-butyl or at least one trichloromethyl substituent;

Examples of ingredients from each of these groups are:

- Group 1: 2,4-dimethyl-3-cyclohexene-1-carbaldehyde (origin: Firmenich SA, Geneva, Switzerland), isocyclocitral, menthone, isomenthone, methyl 2,2-dimethyl-6-methylene-1-cyclohexanecarboxylate (origin: Firmenich SA, Geneva, Switzerland), nerone, terpineol, dihydroterpineol, terpenyl acetate, dihydroterpenyl acetate, dipentene, eucalyptol, hexylate, rose oxide, (S)-1,8-p-menthadiene-7-ol (origin: Firmenich SA, Geneva, Switzerland), 1-p-menthene-4-ol, (1RS,3RS,4SR)-3-p-mentanyl acetate, (1R,2S,4R)-4,6,6-trimethyl-bicyclo[3,1,1]heptan-2-ol, tetrahydro-4-methyl-2-phenyl-2H-pyran (origin: Firmenich SA, Geneva, Switzerland), cyclohexyl acetate, cyclanol acetate, 1,4-cyclohexane diethyldicarboxylate (origin: Firmenich SA, Geneva, Switzerland), (3RS,3aRS,6SR,7ASR)-perhydro-3,6-dimethyl-benzo[B]furan-2-one (origin: Firmenich SA, Geneva, Switzerland), ((6R)-perhydro-3,6-dimethyl-benzo[B]furan-2-one (origin: Firmenich SA, Geneva, Switzerland), 2,4,6-trimethyl-4-phenyl-1,3-dioxane, 2,4,6-trimethyl-3-cyclohexene-1-carbaldehyde;
- Group 2: (E)-3-methyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-4-penten-2-ol (origin: Givaudan SA, Vernier, Switzerland), (1'R,E)-2-ethyl-4-(2',2',3'-trimethyl-3'-cyclopenten-1'-yl)-2-buten-1-ol (origin: Firmenich SA, Geneva, Switzerland), (1'R,E)-3,3-dimethyl-5-(2',2',3'-trimethyl-3'-cyclopenten-1'-yl)-4-penten-2-ol (origin: Firmenich SA, Geneva, Switzerland), 2-heptylcyclopentanone, methyl-cis-3-oxo-2-pentyl-1-cyclopentane acetate (origin: Firmenich SA, Geneva, Switzerland), 2,2,5-trimethyl-5-pentyl-1-cyclopentanone (origin: Firmenich SA, Geneva, Switzerland), 3,3-dimethyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-4-penten-2-ol (origin: Firmenich SA, Geneva, Switzerland), 3-methyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-2-pentanol (origin, Givaudan SA, Vernier, Switzerland);
- Group 3: damascones, 1-(5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one (origin: Firmenich SA, Geneva, Switzerland), (1'R)-2-[2-(4'-methyl-3'-cyclohexen-1'-yl)propyl]cyclopentanone, alpha-ionone, beta-ionone, damascenone, mixture of 1-(5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one and 1-(3,3-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one (origin: Firmenich SA, Geneva, Switzerland), 1-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-buten-1-one (origin: Firmenich SA, Geneva, Switzerland), (1S,1'R)-[1-(3',3'-Dimethyl-1'-cyclohexyl)ethoxycarbonyl]methyl propanoate (origin: Firmenich SA, Geneva, Switzerland), 2-tert-butyl-1-cyclohexyl acetate (origin: International Flavors and Fragrances, USA), 1-(2,2,3,6-tetramethyl-cyclohexyl)-3-hexanol (origin: Firmenich SA, Geneva, Switzerland),

- trans-1-(2,2,6-trimethyl-1-cyclohexyl)-3-hexanol (origin: Firmenich SA, Geneva, Switzerland), (E)-3-methyl-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-3-buten-2-one, terpenyl isobutyrate, 4-(1,1-dimethylethyl)-1-cyclohexyl acetate (origin: Firmenich SA, Geneva, Switzerland), 8-methoxy-1-p-menthene, (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl propanoate (origin: Firmenich SA, Geneva, Switzerland), para tert-butylcyclohexanone, menthenethiol, 1-methyl-4-(4-methyl-3-pentenyl)-3-cyclohexene-1-carbaldehyde, allyl cyclohexylpropionate, cyclohexyl salicylate, 2-methoxy-4-methylphenyl methyl carbonate, ethyl 2-methoxy-4-methylphenyl carbonate, 4-ethyl-2-methoxyphenyl methyl carbonate;
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- 10 - Group 4: Methyl cedryl ketone (origin: International Flavors and Fragrances, USA), a mixture of (1RS,2SR,6RS,7RS,8SR)-tricyclo[5.2.1.0<sup>2,6</sup>]dec-3-en-8-yl 2-methylpropanoate and (1RS,2SR,6RS,7RS,8SR)-tricyclo[5.2.1.0<sup>2,6</sup>]dec-4-en-8-yl 2-methylpropanoate, vetyverol, vetyverone, 1-(octahydro-2,3,8,8-tetramethyl-2-naphtalenyl)-1-ethanone (origin: International Flavors and Fragrances, USA), (5RS,9RS,10SR)-2,6,9,10-tetramethyl-1-
- 15 oxaspiro[4.5]deca-3,6-diene and the (5RS,9SR,10RS) isomer, 6-ethyl-2,10,10-trimethyl-1-oxaspiro[4.5]deca-3,6-diene, 1,2,3,5,6,7-hexahydro-1,1,2,3,3-pentamethyl-4-indenone (origin: International Flavors and Fragrances, USA), a mixture of 3-(3,3-dimethyl-5-indanyl)propanal and 3-(1,1-dimethyl-5-indanyl)propanal (origin: Firmenich SA, Geneva, Switzerland), 3',4-dimethyl-tricyclo[6.2.1.0(2,7)]undec-4-ene-9-spiro-2'-oxirane (origin:
- 20 Firmenich SA, Geneva, Switzerland), 9/10-ethyldiene-3-oxatricyclo[6.2.1.0(2,7)]undecane, (perhydro-5,5,8A-trimethyl-2-naphthalenyl acetate (origin: Firmenich SA, Geneva, Switzerland), octalynol, (dodecahydro-3a,6,6,9a-tetramethyl-naphtho[2,1-b]furan, origin: Firmenich SA, Geneva, Switzerland), tricyclo[5.2.1.0(2,6)]dec-3-en-8-yl acetate and tricyclo[5.2.1.0(2,6)]dec-4-en-8-yl acetate as well as tricyclo[5.2.1.0(2,6)]dec-3-en-8-yl
- 25 propanoate and tricyclo[5.2.1.0(2,6)]dec-4-en-8-yl propanoate, (+)-(1S,2S,3S)-2,6,6-trimethyl-bicyclo[3.1.1]heptane-3-spiro-2'-cyclohexen-4'-one;
- Group 5: camphor, borneol, isobornyl acetate, 8-isopropyl-6-methyl-bicyclo[2.2.2]oct-5-ene-2-carbaldehyde, pinene, camphene, 8-methoxycedrane, (8-methoxy-2,6,6,8-tetramethyl-tricyclo[5.3.1.0(1,5)]undecane (origin: Firmenich SA, Geneva, Switzerland), cedrene,
- 30 cedrenol, cedrol, mixture of 9-ethylidene-3-oxatricyclo[6.2.1.0(2,7)]undecan-4-one and 10-ethylidene-3-oxatricyclo[6.2.1.0<sup>2,7</sup>]undecan-4-one (origin: Firmenich SA, Geneva,

Switzerland), 3-methoxy-7,7-dimethyl-10-methylene-bicyclo[4.3.1]decane (origin: Firmenich SA, Geneva, Switzerland);

- Group 6: (trimethyl-13-oxabicyclo-[10.1.0]-trideca-4,8-diene (origin: Firmenich SA, Geneva, Switzerland), 9-hexadecen-16-olide (origin: Firmenich SA, Geneva, Switzerland),  
 5 pentadecenolide (origin: Firmenich SA, Geneva, Switzerland), 3-methyl-(4/5)-cyclopentadecenone ,(origin: Firmenich SA, Geneva, Switzerland), 3-methylcyclopentadecanone (origin: Firmenich SA, Geneva, Switzerland), pentadecanolide (origin: Firmenich SA, Geneva, Switzerland), cyclopentadecanone (origin: Firmenich SA, Geneva, Switzerland), 1-ethoxyethoxy)cyclododecane (origin: Firmenich SA, Geneva,  
 10 Switzerland), 1,4-dioxacycloheptadecane-5,17-dione, 4,8-cyclododecadien-1-one;
- Group 7: (+-)-2-methyl-3-[4-(2-methyl-2-propanyl)phenyl]propanal (origin: Givaudan SA, Vernier, Switzerland), 2,2,2-trichloro-1-phenylethyl acetate.

Preferably, the perfume comprises at least 30%, preferably at least 50%, more preferably at least 60% of ingredients selected from Groups 1 to 7, as defined above. More preferably said  
 15 perfume comprises at least 30%, preferably at least 50% of ingredients from Groups 3 to 7, as defined above. Most preferably said perfume comprises at least 30%, preferably at least 50% of ingredients from Groups 3, 4, 6 or 7, as defined above.

According to another preferred embodiment, the perfume comprises at least 30%, preferably at least 50%, more preferably at least 60% of ingredients having a logP above 3,  
 20 preferably above 3.5 and even more preferably above 3.75.

According to a particular embodiment, the perfume used in the invention contains less than 10% of its own weight of primary alcohols, less than 15% of its own weight of secondary alcohols and less than 20% of its own weight of tertiary alcohols. Advantageously, the perfume used in the  
 25 invention does not contain any primary alcohols and contains less than 15% of secondary and tertiary alcohols.

According to an embodiment, the oil phase (or the oil-based core) comprises:

- 25-100wt% of a perfume oil comprising at least 15wt% of high impact perfume raw materials having a Log T<-4, and
  - 0-75wt% of a density balancing material having a density greater than 1.07 g/cm<sup>3</sup>.
- 30 “*High impact perfume raw materials*” should be understood as perfume raw materials having a LogT<-4. The odor threshold concentration of a chemical compound is determined in part by its

shape, polarity, partial charges and molecular mass. For convenience, the odor threshold concentration is presented as the common logarithm of the threshold concentration, i.e., Log [Threshold] ("LogT").

5 A "*density balancing material*" should be understood as a material having a density greater than 1.07 g/cm<sup>3</sup> and having preferably low or no odor.

The odor threshold concentration of a perfuming compound is determined by using a gas chromatograph ("GC"). Specifically, the gas chromatograph is calibrated to determine the exact volume of the perfume oil ingredient injected by the syringe, the precise split ratio, and the hydrocarbon response using a hydrocarbon standard of known concentration and chain-length distribution. The air flow rate is accurately measured and, assuming the duration of a human inhalation to last 12 seconds, the sampled volume is calculated. Since the precise concentration at the detector at any point in time is known, the mass per volume inhaled is known and hence the concentration of the perfuming compound. To determine the threshold concentration, solutions are delivered to the sniff port at the back-calculated concentration. A panelist sniffs the GC effluent and identifies the retention time when odor is noticed. The average across all panelists determines the odor threshold concentration of the perfuming compound. The determination of odor threshold is described in more detail in C. Vuilleumier et al., *Multidimensional Visualization of Physical and Perceptual Data Leading to a Creative Approach in Fragrance Development*, Perfume & Flavorist, Vol. 33, September, 2008, pages 54-61.

20 The nature of high impact perfume raw materials having a Log T<-4 and density balancing material having a density greater than 1.07 g/cm<sup>3</sup> are described in WO2018115250, the content of which are included by reference.

According to an embodiment, the high impact perfume raw materials having a Log T<-4 are selected from the group consisting of (+-)-1-methoxy-3-hexanethiol, 4-(4-hydroxy-1-phenyl)-2-butanone, 2-methoxy-4-(1-propenyl)-1-phenyl acetate, pyrazobutyle, 3-propylphenol, 1-(3-methyl-1-benzofuran-2-yl)ethanone, 2-(3-phenylpropyl)pyridine, 1-(3,3/5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one, 1-(5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one, a mixture comprising (3RS,3aRS,6SR,7ASR)-perhydro-3,6-dimethyl-benzo[b]furan-2-one and (3SR,3aRS,6SR,7ASR)-perhydro-3,6-dimethyl-benzo[b]furan-2-one, (+-)-1-(5-ethyl-5-methyl-1-cyclohexen-1-yl)-4-penten-1-one, (1'S,3'R)-1-methyl-2-[(1',2',2'-trimethylbicyclo[3.1.0]hex-3'-yl)methyl]cyclopropyl}methanol, (+-)-3-mercaptohexyl acetate, (2E)-1-(2,6,6-trimethyl-1,3-

cyclohexadien-1-yl)-2-buten-1-one, H-methyl-2h-1,5-benzodioxepin-3(4H)-one, (2E,6Z)-2,6-nonadien-1-ol, (4Z)-4-dodecenal, (+)-4-hydroxy-2,5-dimethyl-3(2H)-furanone, methyl 2,4-dihydroxy-3,6-dimethylbenzoate, 3-methylindole, (+)-perhydro-4alpha,8abeta-dimethyl-4a-naphthalenol, patchoulol, 2-methoxy-4-(1-propenyl)phenol, mixture comprising (+)-5,6-dihydro-4-methyl-2-phenyl-2H-pyran and tetrahydro-4-methylene-2-phenyl-2H-pyran, mixture comprising 4-methylene-2-phenyltetrahydro-2H-pyran and (+)-4-methyl-2-phenyl-3,6-dihydro-2H-pyran, 4-hydroxy-3-methoxybenzaldehyde, nonylenic aldehyde, 2-methoxy-4-propylphenol, 3-methyl-5-phenyl-2-pentenitrile, 1-(spiro[4.5]dec-6/7-en-7-yl)-4-penten-1-one, 2-methoxynaphthalene, (-)-(3aR,5AS,9AS,9BR)-3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-b]furan, 5-nonanolide, (3aR,5AS,9AS,9BR)-3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-b]furan, 7-isopropyl-2H,4H-1,5-benzodioxepin-3-one, coumarin, 4-methylphenyl isobutyrate, (2E)-1-(2,6,6-trimethyl-1,3-cyclohexadien-1-yl)-2-buten-1-one, beta,2,2,3-tetramethyl-delta-methylene-3-cyclopentene-1-butanol, delta damascone ((2E)-1-[(1R,2SR)-2,6,6-trimethyl-3-cyclohexen-1-yl]-2-buten-1-one), (+)-3,6-dihydro-4,6-dimethyl-2-phenyl-2h-pyran, anisaldehyde, paracresol, 3-ethoxy-4-hydroxybenzaldehyde, methyl 2-aminobenzoate, ethyl methylphenylglycidate, octalactone gamma, ethyl 3-phenyl-2-propenoate, (-)-(2E)-2-ethyl-4-[(1R)-2,2,3-trimethyl-3-cyclopenten-1-yl]-2-buten-1-ol, paracresyl acetate, dodecalactone, tricyclone, (+)-(3R,5Z)-3-methyl-5-cyclopentadecen-1-one, undecalactone, (1R,4R)-8-mercapto-3-p-menthanone, (3S,3AS,6R,7AR)-3,6-dimethylhexahydro-1-benzofuran-2(3H)-one, beta ionone, (+)-6-pentyltetrahydro-2H-pyran-2-one, (3E,5Z)-1,3,5-undecatriene, 10-undecenal, (9E)-9-undecenal (9Z)-9-undecenal, (Z)-4-decenal, (+)-ethyl 2-methylpentanoate, 1,2-diallyldisulfane, 2-tridecenenitrile, 3-tridecenenitrile, , (+)-2-ethyl-4,4-dimethyl-1,3-oxathiane, (+)-(3R,5Z)-3-methyl-5-cyclopentadecen-1-one, 3-(4-tert-butylphenyl)propanal, allyl (cyclohexyloxy)acetate, methylnaphthylketone, (+)-(4E)-3-methyl-4-cyclopentadecen-1-one, (+)-5E3-methyl-5-cyclopentadecen-1-one, cyclopropylmethyl 3-hexenoate, (4E)-4-methyl-5-(4-methylphenyl)-4-pentenal, (+)-1-(5-propyl-1,3-benzodioxol-2-yl)ethanone, 4-methyl-2-pentylpyridine, (+)-(E)-3-methyl-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-3-buten-2-one, (3aRS,5aSR,9aSR,9bRS)-3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-b]furan, (2S,5R)-5-methyl-2-(2-propanyl)cyclohexanone oxime, 6-hexyltetrahydro-2H-pyran-2-one, (+)-3-(3-isopropyl-1-phenyl)butanal, methyl 2-(3-oxo-2-pentylcyclopentyl)acetate, 1-(2,6,6-trimethyl-1-cyclohex-2-enyl)pent-1-en-3-one, indol, 7-propyl-2H,4H-1,5-benzodioxepin-3-one, ethyl praline,

(4-methylphenoxy)acetaldehyde, ethyl tricyclo[5.2.1.0.<sup>2,6</sup>]decane-2-carboxylate, (+)-(1'S,2S,E)-3,3-dimethyl-5-(2',2',3'-trimethyl-3'-cyclopenten-1'-yl)-4-penten-2-ol, (4E)-3,3-dimethyl-5-[(1R)-2,2,3-trimethyl-3-cyclopenten-1-yl]-4-penten-2-ol, 8-isopropyl-6-methyl-bicyclo[2.2.2]oct-5-ene-2-carbaldehyde, methylnonylacetaldehyde, 4-formyl-2-methoxyphenyl 2-methylpropanoate, (E)-4-decenal, (+)-2-ethyl-4-(2,2,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol, (1R,5R)-4,7,7-trimethyl-6-thiabicyclo[3.2.1]oct-3-ene, (1R,4R,5R)-4,7,7-trimethyl-6-thiabicyclo[3.2.1]octane, (-)-(3R)-3,7-dimethyl-1,6-octadien-3-ol, (E)-3-phenyl-2-propenenitrile, 4-methoxybenzyl acetate, (E)-3-methyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-4-penten-2-ol, allyl (2/3-methylbutoxy)acetate, (+)-(2E)-1-(2,6,6-trimethyl-2-cyclohexen-1-yl)-2-buten-1-one, (1E)-1-(2,6,6-trimethyl-1-cyclohexen-1-yl)-1-penten-3-one, and mixtures thereof.

According to an embodiment, perfume raw materials having a Log T<-4 are chosen in the group consisting of aldehydes, ketones, alcohols, phenols, esters lactones, ethers, epoxides, nitriles and mixtures thereof.

According to an embodiment, perfume raw materials having a Log T<-4 comprise at least one compound chosen in the group consisting of alcohols, phenols, esters lactones, ethers, epoxides, nitriles and mixtures thereof, preferably in amount comprised between 20 and 70% by weight based on the total weight of the perfume raw materials having a Log T<-4.

According to an embodiment, perfume raw materials having a Log T<-4 comprise between 20 and 70% by weight of aldehydes, ketones, and mixtures thereof based on the total weight of the perfume raw materials having a Log T<-4.

The remaining perfume raw materials contained in the oil-based core may have therefore a Log T>-4.

According to an embodiment, the perfume raw materials having a Log T>-4 are chosen in the group consisting of ethyl 2-methylbutyrate, (E)-3-phenyl-2-propenyl acetate, (+)-6/8-sec-butylquinoline, (+)-3-(1,3-benzodioxol-5-yl)-2-methylpropanal, verdyl propionate, 1-(octahydro-2,3,8,8-tetramethyl-2-naphtalenyl)-1-ethanone, methyl 2-((1RS,2RS)-3-oxo-2-pentylcyclopentyl)acetate, (+)-(E)-4-methyl-3-decen-5-ol, 2,4-dimethyl-3-cyclohexene-1-carbaldehyde, 1,3,3-trimethyl-2-oxabicyclo[2.2.2]octane, tetrahydro-4-methyl-2-(2-methyl-1-propenyl)-2H-pyran, dodecanal, 1-oxa-12/13-cyclohexadecen-2-one, (+)-3-(4-isopropylphenyl)-2-methylpropanal, aldehyde C11, (+)-2,6-dimethyl-7-octen-2-ol, allyl 3-cyclohexylpropanoate, (Z)-3-hexenyl acetate, 5-methyl-2-(2-propenyl)cyclohexanone, allyl heptanoate, 2-(2-methyl-2-

propanyl)cyclohexyl acetate, 1,1-dimethyl-2-phenylethyl butyrate, geranyl acetate, neryl acetate, (+)-1-phenylethyl acetate, 1,1-dimethyl-2-phenylethyl acetate, 3-methyl-2-butenyl acetate, ethyl 3-oxobutanoate, (2Z)-ethyl 3-hydroxy-2-butenate, 8-p-menthanol, 8-p-menthanyl acetate, 1-p-menthanyl acetate, (+)-2-(4-methyl-3-cyclohexen-1-yl)-2-propanyl acetate, (+)-2-methylbutyl  
5 butanoate, 2-[(1S)-1-[(1R)-3,3-dimethylcyclohexyl]ethoxy]-2-oxoethyl propionate, 3,5,6-trimethyl-3-cyclohexene-1-carbaldehyde, 2,4,6-trimethyl-3-cyclohexene-1-carbaldehyde, 2-cyclohexylethyl acetate, octanal, ethyl butanoate, (+)-(3E)-4-(2,6,6-trimethyl-1/2-cyclohexen-1-yl)-3-buten-2-one, 1-[(1R,6SR)-2,2,6-trimethylcyclohexyl]-3-hexanol, 1,3,3-trimethyl-2-oxabicyclo[2.2.2]octane, 1,3,3-trimethyl-2-oxabicyclo[2.2.2]octane, ethyl hexanoate, undecanal,  
10 decanal, 2-phenylethyl acetate, (1S,2S,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol, (1S,2R,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol, (+)-3,7-dimethyl-3-octanol, 1-methyl-4-(2-propanylidene)cyclohexene, (+)-(R)-4-(2-methoxypropan-2-yl)-1-methylcyclohex-1-ene, verdyl acetate, (3R)-1-[(1R,6S)-2,2,6-trimethylcyclohexyl]-3-hexanol, (3S)-1-[(1R,6S)-2,2,6-trimethylcyclohexyl]-3-hexanol, (3R)-1-[(1S,6S)-2,2,6-trimethylcyclohexyl]-3-hexanol, (+)-  
15 (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl propanoate, and mixtures thereof.

According to an embodiment, the perfume formulation comprises

- 0 to 60 wt.% of a hydrophobic solvent (based on the total weight of the perfume formulation),
- 20 - 40 to 100 wt.% of a perfume oil (based on the total weight of the perfume formulation), wherein the perfume oil has at least two, preferably all of the following characteristics:
  - at least 35%, preferably at least 40%, preferably at least 50%, more preferably at least 60% of perfuming ingredients having a log P above 3, preferably above 3.5,
  - 25 ○ at least 20%, preferably at least 25%, preferably at least 30%, more preferably at least 40% of bulky materials of groups 1 to 6, preferably 3 to 6 as previously defined and
  - at least 15%, preferably at least 20%, more preferably at least 25%, even more preferably at least 30% of high impact perfume materials having a Log T < -4,
  - 30 optionally, further hydrophobic active ingredients.

According to a particular embodiment, the perfume comprises 0 to 60 wt.% of a hydrophobic solvent.

According to a particular embodiment, the hydrophobic solvent is a density balancing material preferably chosen in the group consisting of benzyl salicylate, benzyl benzoate, cyclohexyl salicylate, benzyl phenylacetate, phenylethyl phenylacetate, triacetin, ethyl citrate, methyl and ethyl salicylate, benzyl cinnamate, and mixtures thereof.

In a particular embodiment, the hydrophobic solvent has Hansen Solubility Parameters compatible with entrapped perfume oil.

The term "Hansen solubility parameter" is understood to a solubility parameter approach proposed by Charles Hansen used to predict polymer solubility and was developed around the basis that the total energy of vaporization of a liquid consists of several individual parts. To calculate the "weighted Hansen solubility parameter" one must combine the effects of (atomic) dispersion forces, (molecular) permanent dipole-permanent dipole forces, and (molecular) hydrogen bonding (electron exchange). The weighted Hansen solubility parameter" is calculated as  $(\delta D^2 + \delta P^2 + \delta H^2)^{0.5}$ , wherein  $\delta D$  is the Hansen dispersion value (also referred to in the following as the atomic dispersion fore),  $\delta P$  is the Hansen polarizability value (also referred to in the following as the dipole moment), and  $\delta H$  is the Hansen Hydrogen-bonding ("h-bonding") value (also referred to in the following as hydrogen bonding). For a more detailed description of the parameters and values, see Charles Hansen, The Three Dimensional Solubility Parameter and Solvent Diffusion Coefficient, Danish Technical Press (Copenhagen, 1967).

Euclidean difference in solubility parameter between a fragrance and a solvent is calculated as  $(4 * (\delta D_{\text{solvent}} - \delta D_{\text{fragrance}})^2 + (\delta P_{\text{solvent}} - \delta P_{\text{fragrance}})^2 + (\delta H_{\text{solvent}} - \delta H_{\text{fragrance}})^2)^{0.5}$ , in which  $\delta D_{\text{solvent}}$ ,  $\delta P_{\text{solvent}}$ , and  $\delta H_{\text{solvent}}$ , are the Hansen dispersion value, Hansen polarizability value, and Hansen h-bonding values of the solvent, respectively; and  $\delta D_{\text{fragrance}}$ ,  $\delta P_{\text{fragrance}}$ , and  $\delta H_{\text{fragrance}}$  are the Hansen dispersion value, Hansen polarizability value, and Hansen h-bonding values of the fragrance, respectively.

In a particular embodiment, the perfume oil and the hydrophobic solvent have at least two Hansen solubility parameters selected from a first group consisting of: an atomic dispersion force ( $\delta D$ ) from 12 to 20, a dipole moment ( $\delta P$ ) from 1 to 8, and a hydrogen bonding ( $\delta H$ ) from 2.5 to 11.

In a particular embodiment, the perfume oil and the hydrophobic solvent have at least two Hansen solubility parameters selected from a second group consisting of: an atomic dispersion force ( $\delta D$ ) from 12 to 20, preferably from 14 to 20, a dipole moment ( $\delta P$ ) from 1 to 8, preferably from 1 to 7, and a hydrogen bonding ( $\delta H$ ) from 2.5 to 11, preferably from 4 to 11.

5 In a particular embodiment, at least 90% of the perfume oil, preferably at least 95% of the perfume oil, most preferably at least of 98% of the perfume oil has at least two Hansen solubility parameters selected from a first group consisting of: an atomic dispersion force ( $\delta D$ ) from 12 to 20, a dipole moment ( $\delta P$ ) from 1 to 8, and a hydrogen bonding ( $\delta H$ ) from 2.5 to 11.

In a particular embodiment, the perfume oil and the hydrophobic solvent have at least two  
10 Hansen solubility parameters selected from a second group consisting of: an atomic dispersion force ( $\delta D$ ) from 12 to 20, preferably from 14 to 20, a dipole moment ( $\delta P$ ) from 1 to 8, preferably from 1 to 7, and a hydrogen bonding ( $\delta H$ ) from 2.5 to 11, preferably from 4 to 11.

According to an embodiment, the perfuming formulation comprises a fragrance modulator (that can be used in addition to the hydrophobic solvent when present or as substitution of the  
15 hydrophobic solvent when there is no hydrophobic solvent).

Preferably, the fragrance modulator is defined as a fragrance material with

- i. a vapor pressure of less than 0.0008 Torr at 22°C;
- ii. a clogP of 3.5 and higher, preferably 4.0 and higher and more preferably 4.5
- iii. at least two Hansen solubility parameters selected from a first group consisting  
20 of: an atomic dispersion force from 12 to 20, a dipole moment from 1 to 7, and a hydrogen bonding from 2.5 to 11,
- iv. at least two Hansen solubility parameters selected from a second group consisting of: an atomic dispersion force from 14 to 20, a dipole moment from 1 to 8, and a hydrogen bonding from 4 to 11, when in solution with a compound having a  
25 vapor pressure range of 0.0008 to 0.08 Torr at 22°C.

Preferably, as examples the following ingredients can be listed as modulators but the list is not limited to the following materials: alcohol C12, oxacyclohexadec-12/13-en-2-one, 3-[(2',2',3'-trimethyl-3'-cyclopenten-1'-yl)methoxy]-2-butanol, cyclohexadecanone, (Z)-4-cyclopentadecen-1-one, cyclopentadecanone, (8Z)-oxacycloheptadec-8-en-2-one, 2-[5-(tetrahydro-5-methyl-5-vinyl-2-furyl)-tetrahydro-5-methyl-2-furyl]-2-propanol, muguet aldehyde, 1,5,8-trimethyl-13-  
30 oxabicyclo[10.1.0]trideca-4,8-diene, (+-)-4,6,6,7,8,8-hexamethyl-1,3,4,6,7,8-

hexahydrocyclopenta[g]isochromene, (+)-(1S,2S,3S,5R)-2,6,6-trimethylspiro[bicyclo[3.1.1]heptane-3,1'-cyclohexane]-2'-en-4'-one, oxacyclohexadecan-2-one, 2-[(1S)-1-[(1R)-3,3-dimethylcyclohexyl]ethoxy]-2-oxoethyl propionate, (+)-(4R,4aS,6R)-4,4a-dimethyl-6-(1-propen-2-yl)-4,4a,5,6,7,8-hexahydro-2(3H)-naphthalenone, amylcinnamic  
 5 aldehyde, hexylcinnamic aldehyde, hexyl salicylate, (1E)-1-(2,6,6-trimethyl-1-cyclohexen-1-yl)-1,6-heptadien-3-one, (9Z)-9-cycloheptadecen-1-one, and mixtures thereof.

According to a particular embodiment, the hydrophobic material is free of any active ingredient (such as perfume). According to this particular embodiment, it comprises, preferably consists of hydrophobic solvents, preferably chosen in the group consisting of isopropyl  
 10 myristate, triglycerides (e.g. Neobee® MCT oil, vegetable oils), D-limonene, silicone oil, mineral oil, and mixtures thereof with optionally hydrophilic solvents preferably chosen in the group consisting of 1,4-butanediol, benzyl alcohol, triethyl citrate, triacetin, benzyl acetate, ethyl acetate, propylene glycol (1,2-propanediol), 1,3-propanediol, dipropylene glycol, glycerol, glycol ethers and mixtures thereof.

15 The term "biocide" refers to a chemical substance capable of killing living organisms (e.g. microorganisms) or reducing or preventing their growth and/or accumulation. Biocides are commonly used in medicine, agriculture, forestry, and in industry where they prevent the fouling of, for example, water, agricultural products including seed, and oil pipelines. A biocide can be a pesticide, including a fungicide, herbicide, insecticide, algicide, molluscicide, miticide and  
 20 rodenticide; and/or an antimicrobial such as a germicide, antibiotic, antibacterial, antiviral, antifungal, antiprotozoal and/or antiparasite.

As used herein, a "pest control agent" indicates a substance that serves to repel or attract pests, to decrease, inhibit or promote their growth, development or their activity. Pests refer to any living organism, whether animal, plant or fungus, which is invasive or troublesome to plants  
 25 or animals, pests include insects notably arthropods, mites, spiders, fungi, weeds, bacteria and other microorganisms.

According to any one of the invention's embodiments, the hydrophobic material H1 represents between 10% and 60% w/w, or even between 15% and 45% w/w, by weight, relative to the total weight of the two-phases dispersion E1 as obtained after step i).

30 According to a particular embodiment, the process comprises an additional step consisting of adding a hydrophobic material H2 in the slurry. The hydrophobic material H2 is preferably

added in an amount comprised between 20% and 80%, preferably between 30% and 75% by weight based on the total weight of the slurry as obtained after step iv).

The hydrophobic material H2 is defined as the hydrophobic material H1 defined previously and is preferably a perfume.

5

#### Continuous phase

According to the invention, the continuous phase in step a) comprises a polyol.

According to a particular embodiment, the content of water in the continuous phase is below or equal to 10% by weight based on the total weight of the continuous phase.

10

According to a particular embodiment, the continuous phase is free from water.

According to a particular embodiment, no water is added at any stage of the process.

Any polyol that is not soluble with the hydrophobic material H1 can be used in the present invention.

A polyol or a mixture of polyols can be used.

15

The polyol can be chosen in the group consisting of glycerol, 1,4-butanediol, ethylene glycol, 1,2-propylene glycol, 1,3-propylene glycol, triethanolamine, di(trimethylolpropane), 1,2-hexanediol, 1,6-hexanediol, 2-ethyl-2-(hydroxymethyl)propane-1,3-diol (trimethylolpropane, TMP), 2,2-bis(hydroxymethyl)propane-1,3-diol (pentaerythritol), 2-amino-2-ethylpropane-1,3-diol,—2-amino-2-(hydroxymethyl)propane-1,3-diol,—2,2'-azanediyldis(ethan-1-ol)—2-amino-2-methylpropane-1,3-diol, polyphenols and mixtures thereof.

20

According to an embodiment, the polyol is added in an amount comprised between 10% and 90%, preferably between 25% and 60% by weight based on the total weight of the two-phases dispersion E1.

According to a particular embodiment, the polyol is a mixture of glycerol and propylene glycol, preferably 1,2 propylene glycol.

25

According to a particular embodiment, the polyol is a mixture of glycerol and 1,4-butanediol.

#### Stabilizer S1

30

According to the invention, the oil phase O1 and/or the continuous phase comprise a stabilizer S1. The stabilizer S1 is preferably added in the continuous phase.

A single stabilizer or mixture of stabilizers can be used.

Said stabilizer can be an ionic or non-ionic emulsifier or a colloidal stabilizer. According to an embodiment, the stabilizer is a colloid stabilizer.

The stabilizer can be a molecular emulsifier (standard emulsion) or a colloidal particle stabilizer (Pickering emulsion). As colloidal particle stabilizer that can be used, one may cite for example calcium phosphate, silica, silicates, titanium dioxide, aluminium oxide, zinc oxide, iron oxide, mica, kaolin, montmorillonite, laponite, bentonite, perlite, dolomite, diatomite, vermiculite, hectorite, gibbsite, illite, kaolinite, aluminosilicates, gypsum, bauxite, magnesite, talc, magnesium carbonate, calcium carbonate, diatomaceous earth and mixtures thereof.

10       “**Stabilizer**” and “**emulsifier**” are used indifferently in the present invention.

Among the stabilizer S1 that can be used in the present invention, one may cite for example Tergitol 15-S-12 (secondary alcohol ethoxylate), Tween 80 (polyethylene glycol sorbitan monooleate), Tween 20 (polyethylene glycol sorbitan monooleate), Silica HKD H15 (hydrophobized silica particles); silk protein; Span® 60 (sorbitan stearate), hydroxyapatite particles, PGPR90 (polyglycerol polyricinoleate), silica HKD H15 (hydrophobized silica particles), Span 85 (sorbitane trioleate), Makibase SEB (polyglyceryl-3 diisostearate), cellulose derivatives such as hydroxypropylcellulose, and mixtures thereof.

According to an embodiment, the stabilizer S1 is chosen in the group consisting of secondary alcohol ethoxylate, polyethylene glycol sorbitan monooleate, hydrophobized silica particles (hydrophobic silica particles); silk protein; sorbitan stearate, hydroxyapatite particles, polyglycerol polyricinoleate, sorbitane trioleate, polyglyceryl-3 diisostearate, cellulose derivatives such as hydroxypropylcellulose, and mixtures thereof.

The stabilizer S1 is typically used in an amount comprised between 0.1 and 50%, preferably between 0.2 and 30%, most preferably 0.5 to 20%, even more preferably 1 to 10% by weight based on the total weight of the oil phase O1.

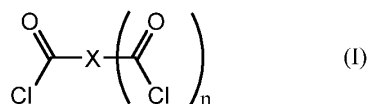
According to the invention, at least one carboxylic acid derivative A1 is added into E1 after step i) and/or at least one carboxylic acid derivative A2 is added into the oil phase O1.

According to an embodiment, the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is chosen in the group consisting of acid, amide, ester, anhydride, acid chloride and mixtures thereof.

According to a particular embodiment, the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is an acyl chloride.

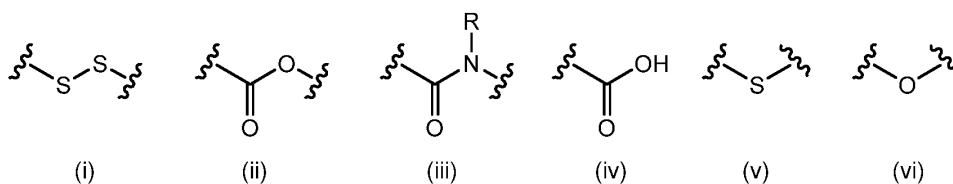
According to a particular embodiment, the acyl chloride has the following formula (I)

5



wherein n is an integer varying between 1 and 8, preferably between 1 and 6, more preferably between 1 and 4, and

10 wherein X is an (n+1)-valent C<sub>2</sub> to C<sub>45</sub> hydrocarbon group optionally comprising at least one group selected from (i) to (vi),



15

wherein R is a hydrogen atom or a methyl or ethyl group, preferably a hydrogen atom.

It is understood that by "... **hydrocarbon group** ..." it is meant that said group consists of hydrogen and carbon atoms and can be in the form of an aliphatic hydrocarbon, i.e. linear or branched saturated hydrocarbon (e.g. alkyl group), a linear or branched unsaturated hydrocarbon (e.g. alkenyl or alkynyl group), a saturated cyclic hydrocarbon (e.g. cycloalkyl) or an unsaturated cyclic hydrocarbon (e.g. cycloalkenyl or cycloalkynyl), or can be in the form of an aromatic hydrocarbon, i.e. aryl group, or can also be in the form of a mixture of said type of groups, e.g. a specific group may comprise a linear alkyl, a branched alkenyl (e.g. having one or more carbon-carbon double bonds), a (poly)cycloalkyl and an aryl moiety, unless a specific limitation to only one type is mentioned. Similarly, in all the embodiments of the invention, when a group is mentioned as being in the form of more than one type of topology (e.g. linear, cyclic or branched)

and/or being saturated or unsaturated (e.g. alkyl, aromatic or alkenyl), it is also meant a group which may comprise moieties having any one of said topologies or being saturated or unsaturated, as explained above. Similarly, in all the embodiments of the invention, when a group is mentioned as being in the form of one type of saturation or unsaturation, (e.g. alkyl), it is meant that said group can be in any type of topology (e.g. linear, cyclic or branched) or having several moieties with various topologies.

It is understood that with the term “... **a hydrocarbon group, optionally comprising ...**” it is meant that said hydrocarbon group optionally comprises heteroatoms to form ether, thioether, amine, nitrile or carboxylic acid groups. These groups can either substitute a hydrogen atom of the hydrocarbon group and thus be laterally attached to said hydrocarbon, or substitute a carbon atom (if chemically possible) of the hydrocarbon group and thus be inserted into the hydrocarbon chain or ring.

According to a particular embodiment, the acyl chloride is chosen from the group consisting of benzene-1,3,5-tricarbonyl trichloride (trimesoyl trichloride), benzene-1,2,4-tricarboxyl trichloride, benzene-1,2,4,5-tetracarboxyl tetrachloride, cyclohexane-1,3,5-tricarboxyl trichloride, isophthaloyl dichloride, diglycolyl dichloride, terephthaloyl chloride, fumaryl dichloride, adipoyl dichloride, succinic acid dichloride (succinic dichloride or succinyl chloride or succinyl dichloride), propane-1,2,3-tricarboxyl trichloride, cyclohexane-1,2,4,5-tetracarboxyl tetrachloride, 2,2'-disulfanediylsuccinyl dichloride, 2-(2-chloro-2-oxo-ethyl)sulfanylbutanediyl dichloride, (4-chloro-4-oxobutanoyl)-L-glutamoyl dichloride, (S)-4-((1,5-dichloro-1,5-dioxopentan-2-yl)amino)-4-oxobutanoic acid, 2,2-bis[(4-chloro-4-oxobutanoyl)oxymethyl]butyl 4-chloro-4-oxo-butanoate, [2-[2,2-bis[(4-chloro-4-oxobutanoyl)oxymethyl]butoxymethyl]-2-[(4-chloro-4-oxo-butanoyl)oxymethyl]butyl] 4-chloro-4-oxo-butanoate, 2,2-bis[(2-chlorocarbonylbenzoyl)oxymethyl]butyl 2-chlorocarbonyl-benzoate, [2-[2,2-bis[(2-chlorocarbonylbenzoyl)oxymethyl]butoxymethyl]-2-[(2-chlorocarbonylbenzoyl)oxymethyl]butyl] 2-chlorocarbonylbenzoate, 4-(2,4,5-trichlorocarbonylbenzoyl)oxybutyl 2,4,5-trichlorocarbonyl-benzoate, propane-1,2,3-triyl tris(4-chloro-4-oxobutanoate), propane-1,2-diyl bis(4-chloro-4-oxobutanoate) and mixtures thereof.

According to a particular embodiment, the acyl chloride is chosen from the group consisting of benzene-1,2,4-tricarboxyl trichloride, benzene-1,2,4,5-tetracarboxyl tetrachloride, cyclohexane-1,3,5-tricarboxyl trichloride, isophthaloyl dichloride, diglycolyl dichloride,

terephthaloyl chloride, fumaryl dichloride, adipoyl dichloride, succinic dichloride, propane-1,2,3-tricarboxyl trichloride, cyclohexane-1,2,4,5-tetracarboxyl tetrachloride, 2,2'-disulfanediyldisuccinyl dichloride, 2-(2-chloro-2-oxo-ethyl)sulfanylbutanedioyl dichloride, (4-chloro-4-oxobutanoyl)-L-glutamoyl dichloride, (S)-4-((1,5-dichloro-1,5-dioxopentan-2-yl)amino)-4-oxobutanoic acid, 2,2-bis[(4-chloro-4-oxo-butanoyl)oxymethyl]butyl 4-chloro-4-oxo-butanoate, [2-[2,2-bis[(4-chloro-4-oxo-butanoyl)oxymethyl]butoxymethyl]-2-[(4-chloro-4-oxo-butanoyl)oxymethyl]butyl] 4-chloro-4-oxo-butanoate, 2,2-bis[(2-chlorocarbonylbenzoyl)oxymethyl]butyl 2-chlorocarbonyl-benzoate, [2-[2,2-bis[(2-chlorocarbonylbenzoyl)oxymethyl]butoxymethyl]-2-[(2-chlorocarbonylbenzoyl)oxymethyl]butyl] 2-chlorocarbonylbenzoate, 4-(2,4,5-trichlorocarbonylbenzoyl)oxybutyl 2,4,5-trichlorocarbonyl-benzoate, propane-1,2,3-triyl tris(4-chloro-4-oxobutanoate), propane-1,2-diyl bis(4-chloro-4-oxobutanoate), and mixtures thereof.

According to a particular embodiment, the acyl chloride is chosen from the group consisting of fumaryl dichloride, adipoyl dichloride, succinic dichloride (succinyl chloride), propane-1,2,3-triyl tris(4-chloro-4-oxobutanoate), propane-1,2-diyl bis(4-chloro-4-oxobutanoate), and mixtures thereof.

According to a particular embodiment, the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is an acyl chloride.

According to a particular embodiment, the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 can be the same or can be different.

According to a particular embodiment, the carboxylic acid derivative A1 is adipoyl chloride.

According to a particular embodiment, the carboxylic acid derivative A1 is succinyl chloride.

According to a particular embodiment, the carboxylic acid derivative A1 is a mixture of adipoyl chloride and succinyl chloride.

According to a particular embodiment, the carboxylic acid derivative A2 is trimesoyl trichloride.

The carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is preferably used in an amount from 0.1 to 50% by weight based on the total weight of the dispersion E1.

According to a particular embodiment:

- the polyol is a mixture of glycerol and 1,4-butanediol.
- the carboxylic acid derivative A1 is succinyl chloride or adipoyl chloride, and
- the stabilizer S1 is hydrophobic silica particles.

5 In another step of the process, conditions sufficient are applied to induce interfacial polymerization and form polyester microcapsules in the form of a slurry.

This step allows ending up with microcapsules in the form of a slurry. According to a preferred embodiment, to enhance the kinetics, said step is performed at a temperature comprised between 5 and 90°C, possibly under pressure, for 1 to 30 hours. More preferably it is performed at between 10 and 80°C for between 30 minutes and 5 hours. However, this step (called curing step)  
10 can be carried out at room temperature.

According to a particular embodiment of the invention, at the end of step iv) or during step iv) one may also add to the invention's slurry a polymer selected from the group consisting of a non-ionic polysaccharide, a cationic polymer, a polysuccinimide derivative (as described for instance in WO2021185724) and mixtures thereof to form an outer coating to the microcapsule.

15 Non-ionic polysaccharide polymers are well known to a person skilled in the art and are described for instance in WO2012/007438 page 29, lines 1 to 25 and in WO2013/026657 page 2, lines 12 to 19 and page 4, lines 3 to 12. Preferred non-ionic polysaccharides are selected from the group consisting of locust bean gum, xyloglucan, guar gum, hydroxypropyl guar, hydroxypropyl cellulose and hydroxypropyl methyl cellulose.

20 Cationic polymers are well known to a person skilled in the art. Preferred cationic polymers have cationic charge densities of at least 0.5 meq/g, more preferably at least about 1.5 meq/g, but also preferably less than about 7 meq/g, more preferably less than about 6.2 meq/g. The cationic charge density of the cationic polymers may be determined by the Kjeldahl method as described in the US Pharmacopoeia under chemical tests for Nitrogen determination. The preferred cationic  
25 polymers are chosen from those that contain units comprising primary, secondary, tertiary and/or quaternary amine groups that can either form part of the main polymer chain or can be borne by a side substituent directly connected thereto. The weight average (Mw) molecular weight of the cationic polymer is preferably between 10,000 and 3.5M Dalton, more preferably between 50,000 and 1.5M Dalton. According to a particular embodiment, one will use cationic polymers based on  
30 acrylamide, methacrylamide, N-vinylpyrrolidone, quaternized N,N-dimethylaminomethacrylate, diallyldimethylammonium chloride, quaternized vinylimidazole (3-methyl-1-vinyl-1H-imidazol-

3-ium chloride), vinylpyrrolidone, acrylamidopropyltrimonium chloride, cassia hydroxypropyltrimonium chloride, guar hydroxypropyltrimonium chloride or polygalactomannan 2-hydroxypropyltrimethylammonium chloride ether, starch hydroxypropyltrimonium chloride and cellulose hydroxypropyltrimonium chloride. Preferably copolymers shall be selected from the group consisting of polyquaternium-5, polyquaternium-6, polyquaternium-7, polyquaternium-10, polyquaternium-11, polyquaternium-16, polyquaternium-22, polyquaternium-28, polyquaternium-43, polyquaternium-44, polyquaternium-46, cassia hydroxypropyltrimonium chloride, guar hydroxypropyltrimonium chloride or polygalactomannan 2-hydroxypropyltrimethylammonium chloride ether, starch hydroxypropyltrimonium chloride and cellulose hydroxypropyltrimonium chloride. As specific examples of commercially available products, one may cite Salcare® SC60 (cationic copolymer of acrylamidopropyltrimonium chloride and acrylamide, origin: BASF) or Luviquat®, such as the PQ 11N, FC 550 or Style (polyquaternium-11 to 68 or quaternized copolymers of vinylpyrrolidone origin: BASF), or also the Jaguar® (C13S or C17, origin Rhodia).

According to any one of the above embodiments of the invention, there is added an amount of polymer described above comprised between about 0% and 5% w/w, or even between about 0.1% and 2% w/w, percentage being expressed on a w/w basis relative to the total weight of the slurry as obtained after step iv). It is clearly understood by a person skilled in the art that only part of said added polymers will be incorporated into/deposited on the microcapsule shell.

According to a particular embodiment, the process comprises an additional step consisting of removing the hydrogen chloride (HCl) from the slurry. Indeed, HCl might be generated during the process and can be removed for example by using a gas flow or by using a base.

Another object of the invention is a process for preparing a microcapsule powder comprising the steps as defined above and an additional step consisting of submitting the slurry obtained in step iv) or v) to a drying, like spray-drying, to provide the microcapsules as such, i.e. in a powdery form. It is understood that any standard method known by a person skilled in the art to perform such drying is also applicable. In particular the slurry may be spray-dried preferably in the presence of a polymeric carrier material such as polyvinyl acetate, polyvinyl alcohol, dextrans, natural or modified starch, vegetable gums, pectins, xanthans, alginates, carragenans or cellulose derivatives to provide microcapsules in a powder form.

However, one may cite also other drying method such as the extrusion, plating, spray granulation, the fluidized bed, or even a drying at room temperature using materials (carrier, desiccant) that meet specific criteria as disclosed in WO2017/134179.

According to a particular embodiment, the carrier material contains free perfume oil which  
5 can be the same or different from the perfume from the core of the microcapsules.

### Multiple capsule system

According to an embodiment, the microcapsules of the invention (first type of microcapsule) can be used in combination with a second type of microcapsules.

10 Another object of the invention is a microcapsule delivery system comprising:

- the microcapsules of the present invention as a first type of microcapsules, and
- a second type of microcapsules, wherein the first type of microcapsules and the second type of microcapsules differ in their hydrophobic material and/or their wall material and/or in their coating material.

15

### Polyester microcapsule

The embodiments disclosed previously for defining components in the process also apply for the microcapsule.

A second object of the invention is a polyester microcapsule slurry obtainable by the process as  
20 defined above.

A third object of the invention is a core-shell polyester microcapsule comprising:

- a core comprising an oil phase, wherein the oil phase comprises a hydrophobic material H1, and
- a polyester shell made from the reaction between at least one carboxylic acid derivative A1 and/or at least one carboxylic acid derivative A2 with a polyol.

25 According to an embodiment, the shell is made of polyester such as poly(glycerol-co-succinate), poly(propylene glycol-co-succinate), polyester like poly(glycerol-co-propylene glycol-co-succinate), poly(glycerol-co-adipate), poly(propylene glycol-co-adipate), polyester like poly(glycerol-co-propylene glycol-co-adipate), and mixtures thereof.

According to a particular embodiment, stabilizer S1 is within the polyester shell.

30 In a fourth aspect, the present invention relates to a core-shell polyester microcapsule slurry comprising at least one core-shell polyester microcapsule made of:

- an oil phase comprising a hydrophobic material H1, and
- a shell comprising the reaction product between at least one carboxylic acid derivative A1 and/or at least one carboxylic acid derivative A2 with a polyol.

Embodiments disclosed for the microcapsules previously also apply for the microcapsule  
5 slurry.

According to an embodiment, the microcapsule slurry comprises a hydrophobic material H2, preferably a perfume freely dispersed in the slurry.

In a particular embodiment, the shell material comprises a biodegradable material.

In a particular embodiment, the shell has a biodegradability of at least 40%, preferably at  
10 least 45%, 50%, 60%, 65%, 70%, 75%, 80%, 85%, 90%, 95% or 98%, within 60 days according to OECD301F.

In a particular embodiment, the core-shell microcapsule has a biodegradability of at least 40 %, preferably at least 60 %, preferably at least 65%, 70%, 75%, 80%, 85%, 90%, 95% or 98% within 60 days according to OECD301F.

15 Thereby it is understood that the core-shell microcapsule including all components, such as the core, shell and optionally coating may have a biodegradability of at least 40 %, preferably at least 60 %, preferably at least 65%, 70%, 75%, 80%, 85%, 90%, 95% or 98% within 60 days according to OECD301F.

OECD301F is a standard test method on the biodegradability from the Organization of  
20 Economic Co-operation and Development.

A typical method for extracting the shell for measuring the biodegradability is disclosed in *Gasparini and all in Molecules 2020, 25,718*.

### **Perfuming composition/consumer products**

25 The microcapsules of the invention can be used in combination with active ingredients. An object of the invention is therefore a composition comprising:

- (i) microcapsules or microcapsule slurry as defined above;
- (ii) an active ingredient, preferably chosen in the group consisting of a cosmetic ingredient, skin caring ingredient, perfume ingredient, flavor ingredient, malodour counteracting ingredient,  
30 bactericide ingredient, fungicide ingredient, pharmaceutical or agrochemical ingredient, a sanitizing ingredient, an insect repellent or attractant, and mixtures thereof.

The microcapsules of the invention can be used for the preparation of perfuming or flavouring compositions which are also an object of the invention.

5 The capsules of the invention show very good performance in terms of stability in challenging medium.

Another object of the present invention is a perfuming composition comprising:

- (i) microcapsules or microcapsule slurry as defined above, wherein the oil comprises a perfume;
- (ii) at least one ingredient selected from the group consisting of a perfumery carrier, a  
10 perfumery co-ingredient and mixtures thereof;
- (iii) optionally at least one perfumery adjuvant.

As liquid perfumery carrier one may cite, as non-limiting examples, an emulsifying system, i.e. a solvent and a surfactant system, or a solvent commonly used in perfumery. A detailed description of the nature and type of solvents commonly used in perfumery cannot be exhaustive.  
15 However, one can cite as non-limiting examples solvents such as dipropyleneglycol, diethyl phthalate, isopropyl myristate, benzyl benzoate, 2-(2-ethoxyethoxy)-1-ethanol or ethyl citrate, which are the most commonly used. For the compositions which comprise both a perfumery carrier and a perfumery co-ingredient, other suitable perfumery carriers than those previously specified, can be also ethanol, water/ethanol mixtures, limonene or other terpenes, isoparaffins such as those  
20 known under the trademark Isopar® (origin: Exxon Chemical) or glycol ethers and glycol ether esters such as those known under the trademark Dowanol® (origin: Dow Chemical Company). By “perfumery co-ingredient” it is meant here a compound, which is used in a perfuming preparation or a composition to impart a hedonic effect and which is not a microcapsule as defined above. In other words such a co-ingredient, to be considered as being a perfuming one, must be recognized  
25 by a person skilled in the art as being able to at least impart or modify in a positive or pleasant way the odor of a composition, and not just as having an odor.

The nature and type of the perfuming co-ingredients present in the perfuming composition do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled person being able to select them on the basis of his general knowledge and according to the  
30 intended use or application and the desired organoleptic effect. In general terms, these perfuming co-ingredients belong to chemical classes as varied as alcohols, lactones, aldehydes, ketones,

esters, ethers, acetates, nitriles, terpenoids, nitrogenous or sulphurous heterocyclic compounds and essential oils, and said perfuming co-ingredients can be of natural or synthetic origin. Many of these co-ingredients are in any case listed in reference texts such as the book by S. Arctander, Perfume and Flavor Chemicals, 1969, Montclair, New Jersey, USA, or its more recent versions, or in other works of a similar nature, as well as in the abundant patent literature in the field of perfumery. It is also understood that said co-ingredients may also be compounds known to release in a controlled manner various types of perfuming compounds also known as properfume or profragrance. Non-limiting examples of suitable properfumes may include

4-(dodecylthio)-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-2-butanone, 4-(dodecylthio)-4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-butanone, trans-3-(dodecylthio)-1-(2,6,6-trimethyl-3-cyclohexen-1-yl)-1-butanone, 2-(dodecylthio)octan-4-one, 2-phenylethyl oxo(phenyl)acetate, 3,7-dimethylocta-2,6-dien-1-yl oxo(phenyl)acetate, (Z)-hex-3-en-1-yl oxo(phenyl)acetate, 3,7-dimethyl-2,6-octadien-1-yl hexadecanoate, bis(3,7-dimethylocta-2,6-dien-1-yl) succinate, (2-((2-methylundec-1-en-1-yl)oxy)ethyl)benzene, 1-methoxy-4-(3-methyl-4-phenethoxybut-3-en-1-yl)benzene, (3-methyl-4-phenethoxybut-3-en-1-yl)benzene, 1-(((Z)-hex-3-en-1-yl)oxy)-2-methylundec-1-ene, (2-((2-methylundec-1-en-1-yl)oxy)ethoxy)benzene, 2-methyl-1-(octan-3-yloxy)undec-1-ene, 1-methoxy-4-(1-phenethoxyprop-1-en-2-yl)benzene, 1-methyl-4-(1-phenethoxyprop-1-en-2-yl)benzene, 2-(1-phenethoxyprop-1-en-2-yl)naphthalene, (2-phenethoxyvinyl)benzene, 2-(1-((3,7-dimethyloct-6-en-1-yl)oxy)prop-1-en-2-yl)naphthalene, (2-((2-pentylcyclopentylidene)methoxy)ethyl)benzene, 4-allyl-2-methoxy-1-((2-methoxy-2-phenylvinyl)oxy)benzene, (2-((2-heptylcyclopentylidene)methoxy)ethyl)benzene, 1-isopropyl-4-methyl-2-((2-pentylcyclopentylidene)methoxy)benzene, 2-methoxy-1-((2-pentylcyclopentylidene)methoxy)-4-propylbenzene, 3-methoxy-4-((2-methoxy-2-phenylvinyl)oxy)benzaldehyde, 4-((2-(hexyloxy)-2-phenylvinyl)oxy)-3-methoxybenzaldehyde or a mixture thereof.

By “**perfumery adjuvant**” we mean here an ingredient capable of imparting additional added benefit such as a color, a particular light resistance, chemical stability, etc. A detailed description of the nature and type of adjuvant commonly used in perfuming bases cannot be exhaustive, but it has to be mentioned that said ingredients are well known to a person skilled in the art.

Preferably, the perfuming composition according to the invention comprises between 0.01 and 30 % by weight of microcapsules as defined above.

The invention's microcapsules can advantageously be used in many application fields and used in consumer products. Microcapsules can be used in liquid form applicable to liquid consumer products as well as in powder form, applicable to powder consumer products.

According to a particular embodiment, the consumer product as defined above is liquid and comprises:

- a) from 2 to 65% by weight, relative to the total weight of the consumer product, of at least one surfactant;
- b) water or a water-miscible hydrophilic organic solvent; and
- c) microcapsule slurry or microcapsules as defined above,
- d) optionally non-encapsulated perfume.

According to a particular embodiment, the consumer product as defined above is in a powder form and comprises:

- a) from 2 to 65% by weight, relative to the total weight of the consumer product, of at least one surfactant;
- b) microcapsule powder as defined above.
- c) optionally perfume powder that is different from the microcapsules defined above.

In the case of microcapsules including a perfume oil-based core, the products of the invention, can in particular be of used in perfumed consumer products such as product belonging to fine fragrance or "functional" perfumery. Functional perfumery includes in particular personal-care products including hair-care, body cleansing, skin care, hygiene-care as well as home-care products including laundry care and air care. Consequently, another object of the present invention consists of a perfumed consumer product comprising as a perfuming ingredient, the microcapsules defined above or a perfuming composition as defined above. The perfume element of said consumer product can be a combination of perfume microcapsules as defined above and free or non-encapsulated perfume, as well as other types of perfume microcapsule than those here-disclosed.

In particular a liquid consumer product comprising:

- a) from 2 to 65% by weight, relative to the total weight of the consumer product, of at least one surfactant;

- b) water or a water-miscible hydrophilic organic solvent; and
- c) a perfuming composition as defined above is another object of the invention.

Also a powder consumer product comprising

- (a) from 2 to 65% by weight, relative to the total weight of the consumer product, of at least one  
5 surfactant; and
- (b) a perfuming composition as defined above is part of the invention.

The invention's microcapsules can therefore be added as such or as part of an invention's perfuming composition in a perfumed consumer product.

For the sake of clarity, it has to be mentioned that, by "perfumed consumer product" it is  
10 meant a consumer product which is expected to deliver among different benefits a perfuming effect to the surface to which it is applied (e.g. skin, hair, textile, paper, or home surface) or in the air (air-freshener, deodorizer etc). In other words, a perfumed consumer product according to the invention is a manufactured product which comprises a functional formulation also referred to as "base", together with benefit agents, among which an effective amount of microcapsules according  
15 to the invention.

The nature and type of the other constituents of the perfumed consumer product do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled person being able to select them on the basis of his general knowledge and according to the nature and the desired effect of said product. Base formulations of consumer products in which the  
20 microcapsules of the invention can be incorporated can be found in the abundant literature relative to such products. These formulations do not warrant a detailed description here which would in any case not be exhaustive. The person skilled in the art of formulating such consumer products is perfectly able to select the suitable components on the basis of his general knowledge and of the available literature.

25 Non-limiting examples of suitable perfumed consumer product can be a perfume, such as a fine perfume, a cologne, an after-shave lotion, a body-splash; a fabric care product, such as a liquid or solid detergent, tablets and unit dose (single chamber or multi chambers), a fabric conditioner, a dryer sheet, a fabric refresher, an ironing water, or a bleach; a personal-care product, such as a hair-care product (e.g. a shampoo, hair conditioner, a colouring preparation or a hair  
30 spray), a cosmetic preparation (e.g. a vanishing cream, body lotion or a deodorant or antiperspirant), or a skin-care product (e.g. a perfumed soap, shower or bath mousse, body wash,

oil or gel, bath salts, or a hygiene product); an air care product, such as an air freshener or a “ready to use” powdered air freshener; or a home care product, such all-purpose cleaners, liquid or power or tablet dishwashing products, toilet cleaners or products for cleaning various surfaces, for example sprays & wipes intended for the treatment / refreshment of textiles or hard surfaces  
5 (floors, tiles, stone-floors etc.); a hygiene product such as sanitary napkins, diapers, toilet paper.

Fabric softener or fabric conditioner are used indifferently in the present invention.

Another object of the invention is a consumer product comprising:

- a personal care active base, and
- microcapsules or microcapsule slurry as defined above or the perfuming composition as defined  
10 above,

wherein the consumer product is in the form of a personal care composition.

Personal care active base in which the microcapsules of the invention can be incorporated can be found in the abundant literature relative to such products. These formulations do not warrant a detailed description here which would in any case not be exhaustive. The person skilled in the  
15 art of formulating such consumer products is perfectly able to select the suitable components on the basis of his general knowledge and of the available literature.

The personal care composition is preferably chosen in the group consisting of a hair-care product (e.g. a shampoo, hair conditioner, a colouring preparation or a hair spray), a cosmetic preparation (e.g. a vanishing cream, body lotion or a deodorant or antiperspirant), or a skin-care product (e.g.  
20 a perfumed soap, shower or bath mousse, body wash, oil or gel, bath salts, or a hygiene product);

Another object of the invention is a consumer product comprising:

- a home care or a fabric care active base, and
  - microcapsules or microcapsule slurry as defined above or the perfuming composition as defined  
above,
- 25 wherein the consumer product is in the form of a home care or a fabric care composition.

Home care or fabric care active base in which the microcapsules of the invention can be incorporated can be found in the abundant literature relative to such products. These formulations do not warrant a detailed description here which would in any case not be exhaustive. The person skilled in the art of formulating such consumer products is perfectly able to select the suitable  
30 components on the basis of his general knowledge and of the available literature.

Preferably, the consumer product comprises from 0.1 to 15 wt%, more preferably between 0.2 and 5 wt% of the microcapsules of the present invention, these percentages being defined by weight relative to the total weight of the consumer product. Of course the above concentrations may be adapted according to the benefit effect desired in each product.

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For liquid consumer product mentioned below, by “active base”, it should be understood that the active base includes active materials (typically including surfactants) and water.

For solid consumer product mention below, by “active base”, it should be understood that the active base includes active materials (typically including surfactants) and auxiliary agents (such as bleaching agents, buffering agent; builders; soil release or soil suspension polymers; granulated enzyme particles, corrosion inhibitors, antifoaming, sud suppressing agents; dyes, fillers, and mixtures thereof).

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#### **Fabric softener**

An object of the invention is a consumer product in the form of a fabric softener composition comprising:

- a fabric softener active base; preferably comprising at least one active material chosen in the group consisting of dialkyl quaternary ammonium salts, dialkyl ester quaternary ammonium salts (esterquats), Hamburg esterquat (HEQ), TEAQ (triethanolamine quat),  
silicones and mixtures thereof, the active base being used preferably in an amount comprised between 85 and 99.95% by weight based on the total weight of the composition,
- a microcapsule slurry as defined above, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
- optionally free perfume oil.

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#### **Liquid detergent**

An object of the invention is a consumer product in the form of a liquid detergent composition comprising:

- a liquid detergent active base; preferably comprising at least one active material chosen in the group consisting of anionic surfactant such as alkylbenzenesulfonate (ABS), secondary

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- alkyl sulfonate (SAS), primary alcohol sulfate (PAS), lauryl ether sulfate (LES), methyl ester sulfonate (MES) and nonionic surfactant such as alkyl amines, alkanolamide, fatty alcohol poly(ethylene glycol) ether, fatty alcohol ethoxylate (FAE), ethylene oxide (EO) and propylene oxide (PO) copolymers, amine oxydes, alkyl polyglucosides, alkyl polyglucosamides, the active base being used preferably in an amount comprised between 85 and 99.95% by weight based on the total weight of the composition,
- a microcapsule slurry as defined above, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
  - optionally free perfume oil.

### **Solid detergent**

An object of the invention is a consumer product in the form of a solid detergent composition comprising:

- a solid detergent active base; preferably comprising at least one active material chosen in the group consisting of anionic surfactant such as alkylbenzenesulfonate (ABS), secondary alkyl sulfonate (SAS), primary alcohol sulfate (PAS), lauryl ether sulfate (LES), methyl ester sulfonate (MES) and nonionic surfactant such as alkyl amines, alkanolamide, fatty alcohol poly(ethylene glycol) ether, fatty alcohol ethoxylate (FAE), ethylene oxide (EO) and propylene oxide (PO) copolymers, amine oxydes, alkyl polyglucosides, alkyl polyglucosamides, the active base being used preferably in an amount comprised between 85 and 99.95% by weight based on the total weight of the composition,
- a microcapsule powder or microcapsule slurry as defined above, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
- optionally free perfume oil.

### **Shampoo/shower gel**

An object of the invention is a consumer product in the form of a shampoo or a shower gel composition comprising:

- a shampoo or a shower gel active base; preferably comprising at least one active material chosen in the group consisting of sodium alkylether sulfate, ammonium alkylether sulfates, alkylamphoacetate, cocamidopropyl betaine, cocamide MEA, alkylglucosides and aminoacid based surfactants and mixtures thereof, the active base being used preferably in an amount comprised between 85 and 99.95% by weight based on the total weight of the composition,
- a microcapsule slurry as defined above, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
- optionally free perfume oil.

### **Rinse-Off Conditioner**

An object of the invention is a consumer product in the form of a rinse-off conditioner composition comprising:

- a rinse-off conditioner active base; preferably comprising at least one active material chosen in the group consisting of cetyltrimonium chloride, stearyl trimonium chloride, benzalkonium chloride, behentrimonium chloride and mixture thereof, the active base being used preferably in an amount comprised between 85 and 99.95% by weight based on the total weight of the composition,
- a microcapsule slurry as defined above, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
- optionally free perfume oil.

### **Solid scent booster**

An object of the invention is a consumer product in the form of a solid scent booster composition comprising:

- a solid carrier, preferably chosen in the group consisting of urea, sodium chloride, sodium sulphate, sodium acetate, zeolite, sodium carbonate, sodium bicarbonate, clay, talc, calcium carbonate, magnesium sulfate, gypsum, calcium sulfate, magnesium oxide, zinc oxide, titanium dioxide, calcium chloride, potassium chloride, magnesium chloride, zinc

chloride, saccharides such as sucrose, mono-, di-, and polysaccharides and derivatives such as starch, cellulose, methyl cellulose, ethyl cellulose, propyl cellulose, polyols/sugar alcohols such as sorbitol, maltitol, xylitol, erythritol, and isomalt, PEG, PVP, citric acid or any water soluble solid acid, fatty alcohols or fatty acids and mixtures thereof,

- 5       - a microcapsule slurry as defined above, in a powdered form, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
- optionally free perfume oil.

#### 10   **Liquid scent booster**

An object of the invention is a consumer product in the form of a liquid scent booster composition comprising:

an aqueous phase,

- a surfactant system essentially consisting of one or more than one non-ionic surfactant, wherein the surfactant system has a mean HLB between 10 and 14, preferably chosen in the group consisting of ethoxylated aliphatic alcohols, POE/PPG (polyoxyethylene and polyoxypropylene) ethers, mono and polyglyceryl esters, sucrose ester compounds, polyoxyethylene hydroxylesters, alkyl polyglucosides, amine oxides and combinations thereof;
- 15       - a linker chosen in the group consisting of alcohols, salts and esters of carboxylic acids, salts and esters of hydroxyl carboxylic acids, fatty acids, fatty acid salts, glycerol fatty acids, surfactant having an HLB less than 10 and mixtures thereof, and
- 20       - a microcapsule slurry as defined above, in the form of a slurry, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
- 25       - optionally free perfume oil.

#### **Hair coloration**

An object of the invention is a consumer product in the form of an oxidative hair coloring composition comprising:

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- an oxidizing phase comprising an oxidizing agent and an alkaline phase comprising an alkakine agent, a dye precursor and a coupling compound; wherein said dye precursor and said coupling compound form an oxidative hair dye in the presence of the oxidizing agent, preferably in an amount comprised between 85 and 99.95% by weight based on the total weight of the composition,
- a microcapsule slurry as defined above, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between 0.1 and 5 wt% by weight based on the total weight of the composition,
- optionally free perfume oil

### Perfuming composition

According to a particular embodiment, the consumer product is in the form of a perfuming composition comprising:

- 0.1 to 30%, preferably 0.1 to 20% of microcapsules, preferably in the form of a slurry, as defined previously,
- 0 to 40%, preferably 3-40% of perfume, and
- 20-90%, preferably 40-90% of ethanol, by weight based on the total weight of the perfuming composition.

The invention will now be further described by way of examples. It will be appreciated that the invention as claimed is not intended to be limited in any way by these examples.

### Examples

Table 1: Materials used

| Ingredients                         | Effective Amount of Ingredient (% wt) |
|-------------------------------------|---------------------------------------|
| Perfume oil <sup>1)</sup>           | 20                                    |
| Trimesoyl trichloride <sup>2)</sup> | 5                                     |
| Tween 80 <sup>3)</sup>              | 2                                     |
| Glycerol <sup>4)</sup>              | 20                                    |
| Silica HKD H15 <sup>5)</sup>        | 2                                     |

|                                       |    |
|---------------------------------------|----|
| Neobee <sup>6)</sup>                  | 46 |
| Adipoyl chloride <sup>7)</sup>        | 40 |
| Fumaryl dichloride <sup>8)</sup>      | 40 |
| Succinyl dichloride <sup>9)</sup>     | 40 |
| Tween 20 <sup>3)</sup>                | 2  |
| HPC <sup>10)</sup>                    | 1  |
| Span® 60 <sup>11)</sup>               | 1  |
| Hydroxypropylcellulose <sup>12)</sup> | 2  |

- 1) Perfume oil A; origin: Firmenich SA, Switzerland
- 2) 1,3,5-Benzene tricarbonyl chloride, origin: Sigma Aldrich, Switzerland
- 3) Polyethylene glycol sorbitan monooleate, origin: Sigma Aldrich, Switzerland
- 4) 1,2,3-Propanetriol, origin: Sigma Aldrich, Switzerland
- 5) Hydrophobic silica particles - Silica HKD H15 nanoparticles possessing 50% silanol groups, origin: Wacker Silicones, Germany
- 6) Neobee M5, origin: Firmenich SA, Switzerland
- 7) Adipoyl chloride, origin: Sigma Aldrich, Switzerland
- 8) Fumaryl chloride, origin: Sigma Aldrich, Switzerland
- 9) Succinyl chloride, origin: Sigma Aldrich, Switzerland
- 10) Hydroxypropyl cellulose, origin: Sigma Aldrich, Switzerland
- 11) Sorbitan monostearate, origin: Sigma Aldrich, Switzerland
- 12) Hydroxypropylcellulose, origin: Sigma Aldrich, Switzerland

15 Table 2: Perfume oil A composition

| Raw Materials                             | % in oil |
|-------------------------------------------|----------|
| 2,4-Dimethyl-3-cyclohexene-1-carbaldehyde | 3.30%    |
| Allyl Heptanoate                          | 5.50%    |
| Allyl amyl glycolate                      | 10.99%   |
| Delta Damascone                           | 1.65%    |
| Verdyl acetate                            | 20.30%   |

|                                                                          |        |
|--------------------------------------------------------------------------|--------|
| Methyl dihydrojasmonate <sup>1)</sup>                                    | 4.95%  |
| 1-(octahydro-2,3,8,8-tetramethyl-2-naphtalenyl)-1-ethanone <sup>2)</sup> | 16.49% |
| Ald. Hexylcinnamique                                                     | 9.89%  |
| Ethyl-2-methylvalerate                                                   | 3.3%   |
| (+)-2-methyl-3-[4-(2-methyl-2-propanyl)phenyl]propanal                   | 21.98% |
| (Z)-hex-3-en-1-yl butyrate                                               | 1.1%   |
| (-)-(8R)-8,12-epoxy-13,14,15,16-tetranorlabdane <sup>3)</sup>            | 0.55%  |
| Total                                                                    | 100%   |

1) Firmenich SA, Geneva, Switzerland

2) International Flavors & Fragrances, USA

3) Firmenich SA, Geneva, Switzerland

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### Example 1

#### Preparation of microcapsules comprising trimesoyl trichloride in the oil phase

**General protocol:** Perfume oil A and trimesoyl trichloride was dispersed in a solution of surfactant S1 in glycerol with the help of an ultra turrax at 15,000 rpm for 5 min. The reaction mixture was stirred (up to 24h).

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Table 3: Microcapsule compositions

| Capsule | Perfume A (g) | Trimesoyl chloride (g) | Glycerol (g) | S1       | (g)  |
|---------|---------------|------------------------|--------------|----------|------|
| 1A      | 9.80          | 0.20                   | 19.90        | HKD H15  | 0.10 |
| 1B      | 9.80          | 0.20                   | 19.40        | HKD H15  | 0.60 |
| 1C      | 10.00         | 3.01                   | 19.40        | HKD H15  | 0.60 |
| 1D      | 7.00          | 2.00                   | 18.80        | Tween 80 | 1.20 |

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**Example 2**Preparation of microcapsules comprising adipoyl chloride added in the emulsion E1

- 5 Capsules H: Perfume oil A (12.02 g) was dispersed in a solution of Tween 20 (0.96 g) in glycerol (47.12 g) with the help of an ultra turrax at 15,000 rpm for 5 min. Adipoyl chloride (7.21 g) was added dropwise to the multiple emulsion and the reaction mixture was stirred at 30°C for 24 h.

**Example 3**

- 10 Preparation of microcapsules comprising succinyl chloride added in the emulsion E1

**General protocol:** Tween 20 was dissolved in glycerol. This solution was mixed with 1,2-propylene glycol to afford the continuous phase. Perfume oil A was dispersed in this phase with the help of an ultra turrax for 5 min (up to 24,000). Succinyl chloride was added dropwise to the multiple emulsion and the reaction mixture, which was stirred (up to 60°C and up to 24h) of time and temperature.

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Table 4: Microcapsule compositions

| Capsule | Perfume A (g) | 1,2-propylene glycol (g) | Glycerol (g) | Tween 20 (g) | Succinyl chloride (g) |
|---------|---------------|--------------------------|--------------|--------------|-----------------------|
| 3A      | 5             | 3.00                     | 6.80         | 0.20         | 6.44                  |
| 3B      | 0.83          | 0.50                     | 1.13         | 0.03         | 0.69                  |
| 3C      | 5.00          | 6.80                     | 3.00         | 0.20         | 12.80                 |
| 3D      | 5.00          | 3.00                     | 6.80         | 0.20         | 1.50                  |
| 3E      | 2.50          | 2.45                     | 2.45         | 0.10         | 6.10                  |
| 3F      | 5.00          | 3.00                     | 6.80         | 0.20         | 1.50                  |
| 3G      | 5.00          | 3.00                     | 6.80         | 0.20         | 3.00                  |
| 3H      | 5.00          | 3.00                     | 6.80         | 0.20         | 4.60                  |
| 3I      | 5.00          | 3.00                     | 6.80         | 0.20         | 6.10                  |
| 3J      | 5.00          | 3.00                     | 6.80         | 0.20         | 3.00                  |

|    |       |       |       |      |       |
|----|-------|-------|-------|------|-------|
| 3K | 5.00  | 3.00  | 6.80  | 0.20 | 4.60  |
| 3L | 5.00  | 5.00  | 4.90  | 0.10 | 7.60  |
| 3M | 20.00 | 12.00 | 27.20 | 0.80 | 18.40 |
| 3N | 20.00 | 20.00 | 19.60 | 0.40 | 40.67 |
| 3O | 15.00 | 15.00 | 7.50  | 0.30 | 30.50 |
| 3P | 15.00 | 15.00 | 3.00  | 0.30 | 30.50 |
| 3Q | 20.00 | 20.00 | 19.60 | 0.40 | 30.40 |
| 3R | 5.00  | 5.00  | 3.13  | 0.10 | 7.60  |

#### Example 4

Preparation of microcapsules comprising succinyl chloride and adipoyl chloride added in the emulsion E1

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**Capsules 4A:** Tween 20 (0.40 g) was dissolved in a mixture of glycerol (12.60 g) and 1,2-propylene glycol (20.00 g) with the help of an ultra turrax at 7,000 rpm for 10 s. Perfume oil A (20.00 g) was dispersed in this solution with the help of an ultra turrax at 7,000 rpm for 2 min at 0°C. Succinyl chloride (30.40 g) was added to the emulsion over the course of 1.25 h at room temperature. Then, adipoyl chloride (3.96 g) was added to this emulsion over the course of 1.25 h at room temperature. The reaction mixture was stirred at room temperature for 4 h.

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#### Example 5

Preparation of microcapsules comprising succinyl chloride added in the emulsion E1

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**Capsules 5A:** Hydroxypropyl cellulose (0.03 g) was dissolved in 1,2-propylene glycol (2.97 g). This solution was mixed with glycerol (3.00 g) with the help of an ultra turrax at 7,000 rpm for 10 s. Perfume oil A (3.00 g) was dispersed in this solution with the help of an ultra turrax at 7,000 rpm for 2 min. Succinyl chloride (4.55 g) was added to the emulsion over the course of 1.25 h at room temperature. The reaction mixture was stirred at room temperature for 24 h.

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**Capsules 5B:** Hydroxypropyl cellulose (0.40 g) was dissolved in 1,2-propylene glycol (19.60 g). This solution was mixed with glycerol (12.60 g) with the help of an ultra turrax at 10,000 rpm for

10 s. Perfume oil A (20.00 g) was dispersed in this solution with the help of an ultra turrax at 10,000 rpm for 2 min. Succinyl chloride (30.30 g) was added to the emulsion over the course of 1.25 h at room temperature. The reaction mixture was stirred at room temperature for 4 h.

- 5    **Capsules 5C:** Capsules 5B were prepared according to the protocol of capsules 5B with the help of a mechanical stirrer equipped with an anchor stirred at 650 rpm for 10 min.

10    **Capsules 5D:** Tween 20 (0.40 g) was dissolved in a mixture of glycerol (12.60 g) and 1,2-propylene glycol (20.00 g) with the help of an ultra turrax at 7,000 rpm for 10 s. Perfume oil A (20.00 g) was dispersed in this solution with the help of an ultra turrax at 7,000 rpm for 2 min at 0°C. Succinyl chloride (10.00 g) was added to the emulsion over the course of 1.25 h at room temperature. Then, adipoyl chloride (10.00 g) was added to this emulsion over the course of 1.25 h at room temperature. The reaction mixture was stirred at room temperature for 4 h.

- 15    **Capsules 5E:** Capsules 5E were prepared according to the protocol of capsules 5D with hydroxypropylcellulose (0.4 g) instead of Tween 20.

20    **Capsules 5F:** Tween 20 (0.056 g) was dissolved in 1,4-butanediol (2.80 g). Perfume oil A (1.90 g) was dispersed in this solution with the help of an ultra turrax at 25,000 rpm for 2 min. Succinyl chloride (1.20 g) was added to the emulsion over the course of 1 h at room temperature. The reaction mixture was stirred at 40°C for 24 h.

25    **Capsules 5G:** Tween 20 (0.67 g) was dissolved in a mixture of 1,4-butanediol (33.60 g) and glycerol (21.12 g). Perfume oil A (22.80 g) was dispersed in this solution with the help of an ultra turrax at 25,000 rpm for 2 min. Succinyl chloride (14.40 g) was added to the emulsion over the course of 1 h at room temperature. The reaction mixture was stirred at 40°C for 4 h.

30    **Capsules 5H:** Capsules 5H were prepared according to the protocol of capsules 5G with a curing for 6 h.

**Example 6**Storage stability in a Liquid Detergent composition

The storage stability of the capsules in Liquid Detergent was evaluated. Microcapsule dispersion 3R (i.e slurry) of the present invention was diluted in the liquid detergent composition (P&G US Tide Free & Gentle HE, ingredients listed in Table 5) to obtain a concentration of encapsulated perfume of 0.116%. The detergent was stored at 37°C. The amount of perfume having leaked out of the capsules was then measured by solvent extraction and GC-FID analysis.

Table 5: Liquid Detergent composition

| Ingredients                        |
|------------------------------------|
| Water                              |
| C10-16 Pareth                      |
| Sodium C10-16 Alkylbenzensulfonate |
| Sodium salts of C12-18 fatty acids |
| Sodium citrate                     |
| Propylene glycol                   |
| Polyethyleneimines alkoxylated     |
| C10-16 Alkyldimethylamine oxide    |
| Sodium borate                      |
| Sodium Cumenesulfonate             |
| Subtilisin                         |
| Amylase enzyme                     |
| Mannase enzyme                     |
| Sodium formate                     |

Leakage after 3 days is 38%.

**Example 7**Storage stability in a Fabric Conditioner composition

The storage stability of the capsules in fabric conditioner was evaluated. Microcapsule dispersion 3Q (i.e slurry) of the present invention was diluted in the fabric conditioner composition described in Table 6 to obtain a concentration of encapsulated perfume of 0.116%. The fabric conditioner was stored at 37°C. The amount of perfume having leaked out of the capsules was then measured by solvent extraction and GC-FID analysis.

Table 6: Fabric Conditioner composition

| Product                   | Wt %  |
|---------------------------|-------|
| Stepantex VL 90A          | 8.88  |
| Calcium Chloride Sol. 10% | 0.36  |
| Proxel GXL                | 0.04  |
| Perfume                   | 1     |
| Water                     | 89.72 |
| TOTAL                     | 100   |

Leakage after 3 days is 38%.

**Example 8**Rinse-off conditioner

Microcapsule slurry (see examples 1-5) is dispersed in a rinse-off conditioner base described in table 10 to obtain a concentration of encapsulated perfume oil at 0.5%.

Table 10 – Rinse-off conditioner composition

|  | Ingredients | Concentration<br>[wt %] |
|--|-------------|-------------------------|
|--|-------------|-------------------------|

|          |                                                                                     |             |
|----------|-------------------------------------------------------------------------------------|-------------|
| <b>A</b> | Water deionized                                                                     | <b>81.8</b> |
|          | Behentrimonium Chloride <sup>1)</sup>                                               | <b>2.5</b>  |
|          | Hydroxyethylcellulose <sup>2)</sup>                                                 | <b>1.5</b>  |
|          |                                                                                     |             |
| <b>B</b> | Cetearyl Alcohol <sup>3)</sup>                                                      | <b>4</b>    |
|          | Glyceryl Stearate (and) PEG-100 Stearate <sup>4)</sup>                              | <b>2</b>    |
|          | Behentrimonium Methosulfate (and) Cetyl alcohol (and) Butylene Glycol <sup>5)</sup> | <b>4</b>    |
|          | Ethoxy (20) Stearyl Alcohol <sup>6)</sup>                                           | <b>1</b>    |
|          |                                                                                     |             |
| <b>C</b> | Amodimethicone (and) Trideceth-12 (and) Cetrimonium Chloride <sup>7)</sup>          | <b>3</b>    |
|          | Chlorhexidine Digluconate <sup>8)</sup> 20% aqueous solution                        | <b>0.2</b>  |
|          |                                                                                     |             |
| <b>D</b> | Citric acid 10% aqueous sol. till pH 3.5-4                                          | <b>q.s.</b> |
|          | <b>T O T A L:</b>                                                                   | <b>100</b>  |

1) Genamin KDM P, Clariant

2) Tylose H10 Y G4, Shin Etsu

3) Lanette O, BASF

4) Arlacel 165-FP-MBAL-PA-(RB), Croda

5) Incroquat Behenyl TMS-50-MBAL-PA-(MH) HA4112, Croda

6) SP Brij S20 MBAL-PA(RB), Croda

7) Xiameter DC MEM-0949 Emulsion, Dow Corning

8) Alfa Aesar

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### Example 9

#### Shampoo composition

Microcapsule slurry (see examples 1-5) is weighed and mixed in a shampoo composition to add the equivalent of 0.2% perfume.

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Table 11 – Shampoo composition

|          | <b>Ingredients</b>                         | <b>Concentration<br/>[wt%]</b> |
|----------|--------------------------------------------|--------------------------------|
| <b>A</b> | Water deionized                            | <b>44.4</b>                    |
|          | Polyquaternium-10 <sup>1)</sup>            | <b>0.3</b>                     |
|          | Glycerin 85% <sup>2)</sup>                 | <b>1</b>                       |
|          | DMDM Hydantoin <sup>3)</sup>               | <b>0.2</b>                     |
|          |                                            |                                |
| <b>B</b> | Sodium Laureth Sulfate <sup>4)</sup>       | <b>28</b>                      |
|          | Cocamidopropyl Betaine <sup>5)</sup>       | <b>3.2</b>                     |
|          | Disodium Cocoamphodiacetate <sup>6)</sup>  | <b>4</b>                       |
|          | Ethoxy (20) Stearyl Alcohol <sup>6)</sup>  | <b>1</b>                       |
|          |                                            |                                |
| <b>C</b> | Sodium Laureth Sulfate <sup>4)</sup>       | <b>3</b>                       |
|          | Glyceryl Laureate <sup>7)</sup>            | <b>0.2</b>                     |
|          |                                            |                                |
| <b>D</b> | Water deionized                            | <b>1</b>                       |
|          | Sodium Methylparaben <sup>8)</sup>         | <b>0.1</b>                     |
|          |                                            |                                |
| <b>E</b> | Sodium Chloride 10% aqueous sol.           | <b>15</b>                      |
|          | Citric acid 10% aqueous sol. till pH 5.5-6 | <b>q.s.</b>                    |
|          | Perfume                                    | <b>0.5</b>                     |
|          | <b>T O T A L:</b>                          | <b>100</b>                     |

1) Ucare Polymer JR-400, Noveon

2) Schweizerhall

3) Glydant, Lonza

4) Texapon NSO IS, Cognis

5) Tego Betain F 50, Evonik

6) Amphotensid GB 2009, Zschimmer &amp; Schwarz

7) Monomuls 90 L-12, Gruenau

8) Nipagin Monosodium, NIPA

### Example 10

#### Antiperspirant roll-on emulsion composition

- 5 Microcapsule slurry (see examples 1-5) is weighed and mixed in antiperspirant roll-on emulsion composition to add the equivalent of 0.2% perfume.

Table 12 – Antiperspirant composition

| Ingredient                                                            | Amount (wt %) |
|-----------------------------------------------------------------------|---------------|
| Steareth-2 <sup>1)</sup> (Part A)                                     | 3.25          |
| Steareth-21 <sup>2)</sup> (Part A)                                    | 0.75          |
| PPG-15 Stearyl Ether <sup>3)</sup> (Part A)                           | 4             |
| WATER deionised (Part B)                                              | 51            |
| Aluminum Chlorohydrate 50%<br>aqueous solution <sup>4)</sup> (Part C) | 40            |
| Fragrance (Part D)                                                    | 1             |

- 1) BRIJ 72; origin : ICI  
 10 2) BRIJ 721; origin : ICI  
 3) ARLAMOL E; origin : UNIQEMA-CRODA  
 4) LOCRON L; origin : CLARIAN

- Part A and B are heated separately to 75°C; Part A is added to Part B under stirring and the mixture  
 15 is homogenized for 10 min. Then, the mixture is cooled under stirring; and Part C is slowly added  
 when the mixture reached 45°C and Part D when the mixture reached at 35 °C while stirring. Then  
 the mixture is cooled to room temperature.

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**Example 11**Shower-gel composition

Microcapsule slurry (see examples 1-5) is weighed and mixed in the following composition to  
 5 add the equivalent of 0.2% perfume.

Table 13 – Shower gel composition

| <b>Ingredients</b>                                                  | <b>Amount (% wt)</b> | <b>Function</b> |
|---------------------------------------------------------------------|----------------------|-----------------|
| WATER deionised                                                     | 49.350               | Solvent         |
| Tetrasodium EDTA <sup>1)</sup>                                      | 0.050                | Chelating agent |
| Acrylates Copolymer <sup>2)</sup>                                   | 6.000                | Thickener       |
| Sodium C12-C15 Pareth Sulfate <sup>3)</sup>                         | 35.000               | Surfactant      |
| Sodium Hydroxide 20% aqueous solution                               | 1.000                | pH adjuster     |
| Cocamidopropyl Betaine <sup>4)</sup>                                | 8.000                | Surfactant      |
| Methylchloroisothiazolinone and Methylisothiazolinone <sup>5)</sup> | 0.100                | Preservative    |
| Citric Acid (40%)                                                   | 0.500                | pH adjuster     |

13) EDETA B POWDER; trademark and origin: BASF

14) CARBOPOL AQUA SF-1 POLYMER; trademark and origin: NOVEON

10 15) ZETESOL AO 328 U; trademark and origin: ZSCHIMMER & SCHWARZ

16) TEGO-BETAIN F 50; trademark and origin: GOLDSCHMIDT

17) KATHON CG; trademark and origin: ROHM & HASS

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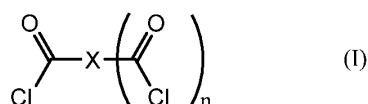
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### Claims

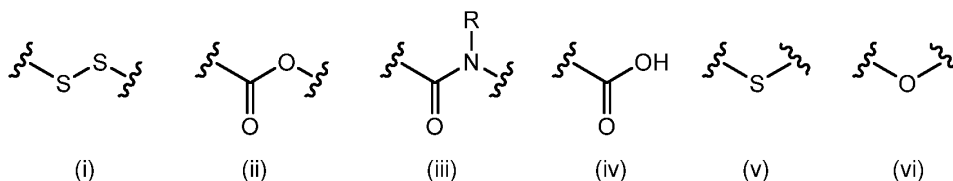
1. Process for preparing a core-shell polyester microcapsule slurry, said process comprising the steps of:
  - 5 (i) Dispersing an oil phase O1 comprising a hydrophobic material H1, into a continuous phase comprising a polyol to obtain a two-phases dispersion E1, wherein the oil phase O1 and/or the continuous phase comprise a stabilizer S1,
  - (ii) applying conditions sufficient to induce interfacial polymerization and form polyester microcapsules in the form of a slurry,
  - 10 wherein at least one carboxylic acid derivative A1 is added into E1 after step i) and/or at least one carboxylic acid derivative A2 is added into the oil phase O1.
2. The process according to claim 1, wherein it comprises the following steps:
  - 15 (i) Dispersing an oil phase O1 comprising a hydrophobic material H1, into a continuous phase comprising a polyol to obtain a two-phases dispersion E1, wherein the oil phase and/or the continuous phase comprise a stabilizer S1 and,
  - (ii) applying conditions sufficient to induce interfacial polymerization and form polyester microcapsules in the form of a slurry,
  - 20 wherein at least one carboxylic acid derivative A1 is added into E1 after step i).
3. The process according to claim 1 or 2, wherein at least a carboxylic acid derivative A1 is added into E1 after step i) and at least a carboxylic acid derivative A2 is added into the oil phase O1.
- 25 4. The process according to anyone of the preceding claims, wherein the hydrophobic material H1 comprises a perfume oil.
5. The process according to anyone of the preceding claims, wherein the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is chosen in the group consisting of  
30 acid, amide, ester, anhydride and acid chloride and mixtures thereof.

6. The process according to claim 5, wherein the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is an acyl chloride.

7. The process according to claim 6, wherein the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is an acyl chloride having the following formula (I)



wherein n is an integer varying between 1 and 8, preferably between 1 and 6, more preferably between 1 and 4, and  
wherein X is an (n+1)-valent C<sub>2</sub> to C<sub>45</sub> hydrocarbon group optionally comprising at least one group selected from (i) to (vi),



wherein R is a hydrogen atom or a methyl or ethyl group, preferably a hydrogen atom.

8. The process according to claim 7, wherein the carboxylic acid derivative A1 and/or the carboxylic acid derivative A2 is chosen from the group consisting of benzene-1,3,5-tricarbonyl trichloride (trimesoyl trichloride), benzene-1,2,4-tricarbonyl trichloride, benzene-1,2,4,5-tetracarboxyl tetrachloride, cyclohexane-1,3,5-tricarbonyl trichloride, isophthaloyl dichloride, diglycolyl dichloride, terephthaloyl chloride, fumaryl dichloride, adipoyl dichloride, succinic acid dichloride, propane-1,2,3-tricarbonyl trichloride, cyclohexane-1,2,4,5-tetracarboxyl tetrachloride, 2,2'-disulfanediylsuccinyl dichloride, 2-(2-chloro-2-oxo-ethyl)sulfanylbutanedioyl dichloride, (4-chloro-4-oxobutanoyl)-L-glutamoyl dichloride, (S)-4-((1,5-dichloro-1,5-dioxopentan-2-

yl)amino)-4-oxobutanoic acid, 2,2-bis[(4-chloro-4-oxo-butanoyl)oxymethyl]butyl 4-chloro-4-oxo-butanoate, [2-[2,2-bis[(4-chloro-4-oxo-butanoyl)oxymethyl]butoxymethyl]-2-[(4-chloro-4-oxo-butanoyl)oxymethyl]butyl] 4-chloro-4-oxo-butanoate, 2,2-bis[(2-chlorocarbonylbenzoyl)oxymethyl]butyl 2-chlorocarbonyl-benzoate, [2-[2,2-bis[(2-chlorocarbonylbenzoyl)oxymethyl]butoxymethyl]-2-[(2-chlorocarbonylbenzoyl)oxymethyl]butyl] 2-chlorocarbonylbenzoate, 4-(2,4,5-trichlorocarbonylbenzoyl)oxybutyl 2,4,5-trichlorocarbonyl-benzoate, propane-1,2,3-triyl tris(4-chloro-4-oxobutanoate), propane-1,2-diyl bis(4-chloro-4-oxobutanoate) and mixtures thereof.

9. The process according to anyone of the preceding claims, wherein the continuous phase does not comprise water.

10. The process according to anyone of the preceding claims, wherein the polyol is chosen in the group consisting of glycerol, 1,4-butanediol, ethylene glycol, 1,2-propylene glycol, 1,3-propylene glycol, triethanolamine, di(trimethylolpropane), 1,2-hexanediol, 1,6-hexanediol, 2-ethyl-2-(hydroxymethyl)propane-1,3-diol (trimethylolpropane, TMP), 2,2-bis(hydroxymethyl)propane-1,3-diol (pentaerythritol), 2-amino-2-ethylpropane-1,3-diol, 2-amino-2-(hydroxymethyl)propane-1,3-diol, 2,2'-azanediyldis(ethan-1-ol), 2-aminopropane-1,3-diol, 2-amino-2-methylpropane-1,3-diol, polyphenols and mixtures thereof.

11. The process according to any one of the preceding claims, wherein the stabilizer S1 is chosen in the group consisting secondary alcohol ethoxylate, polyethylene glycol sorbitan monooleate, hydrophobized silica particles; silk protein; sorbitan stearate, hydroxyapatite particles, polyglycerol polyricinoleate, sorbitane trioleate, polyglyceryl-3 diisostearate, cellulose derivatives such as hydroxypropylcellulose, and mixtures thereof.

12. A core-shell polyester microcapsule slurry obtainable by the process as described in any one of the preceding claims, wherein it comprises at least

- a core comprising an oil phase, wherein the oil phase comprises a hydrophobic material H1, and
- a polyester shell comprising the reaction product between at least one carboxylic acid derivative A1 and/or at least one carboxylic acid derivative A2, with a polyol.

13. A perfuming composition comprising
- (i) a microcapsule slurry as defined in claim 12, wherein the hydrophobic active ingredient comprises a perfume;
- 5 (ii) at least one ingredient selected from the group consisting of a perfumery carrier and a perfuming co-ingredient; and
- (iii) optionally a perfumery adjuvant.
- 10 14. A consumer product comprising:
- a personal care active base, and
  - microcapsules as defined in claim 12 or the perfuming composition as defined in claim 13, wherein the consumer product is in the form of a personal care composition.
- 15 15. A consumer product comprising:
- a home care or a fabric care active base, and
  - microcapsules as defined in claim 12 or the perfuming composition as defined in claim 13, wherein the consumer product is in the form of a home care or a fabric care composition.

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