

MICROCAPSULES AND PROCESS FOR PREPARING MICROCAPSULES

Technical Field

5 The present invention relates to core-shell microcapsules. A process for the preparation of said microcapsules is also an object of the invention. Perfuming compositions and consumer products comprising said microcapsules, in particular perfumed consumer products in the form of home care or personal care products, are also part of the invention.

10 Background of the Invention

One of the problems faced by the perfumery industry lies in the relatively rapid loss of olfactive benefit provided by odoriferous compounds due to their volatility, particularly that of "top-notes". In order to tailor the release rates of volatiles, delivery systems such as microcapsules containing a perfume are needed to protect and later
15 release the core payload when triggered. A key requirement from the industry regarding these systems is to survive suspension in challenging bases without physically dissociating or degrading. This is referred to as *stability* for the delivery system. For instance, fragranced personal and household cleansers containing high levels of aggressive surfactant detergents are very challenging for the stability of
20 microcapsules.

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.

25 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,
30 in particular in terms of stability in a challenging medium such as a consumer product base, as well as in delivering a good performance in terms of active ingredient delivery, e.g. olfactive performance in the case of perfuming ingredients.

The present invention is proposing a solution to the above-mentioned problem by providing new microcapsules and a process for preparing said microcapsules.

Summary of the invention

It has now been found that performing core-shell microcapsules encapsulating hydrophobic material could be obtained by reacting at least one compound having at least two P-Cl functions and a nucleophile. The microcapsules of the invention therefore provide a solution to the above-mentioned problems as it allows preparing eco-friendly microcapsules with the desired stability in challenging bases.

In a first aspect, the present invention relates to a core-shell microcapsule comprising:

- a core comprising a hydrophobic material, and
- a shell comprising the reaction product between at least a nucleophile and at least a compound having at least two P-Cl functions.

Another object of the invention is a core-shell microcapsule slurry comprising at least one microcapsule, the microcapsule comprising:

- a core comprising a hydrophobic material, and
- a shell comprising the reaction product between at least a nucleophile and at least a compound having at least two P-Cl functions.

Another object of the invention is a process for preparing a core-shell microcapsule slurry comprising the steps of:

- a) Optionally, providing at least one compound having at least two P-Cl functions
 - b) admixing at least one hydrophobic material with the optional compound obtained in step a) to form an oil phase;
 - c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;
 - d) Optionally, dispersing the two-phases dispersion into a continuous phase C2 comprising optionally at least one compound having at least two P-Cl functions to form a multiple dispersion;
 - e) curing the dispersion obtained in step c) or d) to form microcapsules in the form of a slurry,
- wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d) and/or in step e).

The present invention also relates to perfumed consumer products and flavoured edible products comprising the microcapsules or microcapsule slurry as defined above.

Detailed description of the invention

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

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

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

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

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

15 A “**microcapsule**”, or the similar, in the present invention it is meant that core-shell microcapsules have a particle size distribution in the micron range (e.g. a mean diameter ($d(v, 0.5)$) comprised preferably between about 1 and 3000 microns, more preferably between 1 and 500 microns) and comprise an external solid polymeric shell and an internal continuous oil phase enclosed by the external shell.

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

“**Dispersing phase**” and “**continuous phase**” can be used indifferently in the present invention.

25 **CORE-SHELL MICROCAPSULE**

In a first aspect, the present invention relates to a core-shell microcapsule comprising:

- a core comprising a hydrophobic material, and
 - a shell comprising the reaction product between at least a nucleophile and
- 30 at least a compound having at least two P-Cl functions.

Hydrophobic material

According to an embodiment, the core is an oil-based core.

The hydrophobic material according to the invention can be “inert” material like solvents or active ingredients.

By “hydrophobic material”, it is meant any hydrophobic material which forms a two-phase dispersion when mixed with water. The hydrophobic material is typically liquid
5 at about 20°C.

According to an embodiment, the hydrophobic material is a hydrophobic active ingredient.

When the hydrophobic materials is an active ingredient, it is preferably chosen from the group consisting of flavors, flavor ingredients, perfumes, perfume ingredients,
10 nutraceuticals, cosmetics, pest control agents, biocide actives, agrochemical ingredients 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
15 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 perfumes, nutraceuticals, cosmetics, pest control agents.

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

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

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

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

30 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 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, 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, nonanal and/or nonenal;
- Aromatic-herbal ingredients: eucalyptus oil, camphor, eucalyptol, 5-methyltricyclo[6.2.1.0^{2,7}]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;
- 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 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, 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;
- 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;
- 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;

- 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, 4,6,6,7,8,8-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;
- 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^{2,7}]undec[4]ene, (1-ethoxyethoxy)cyclododecane, 2,2,9,11-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® (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® 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 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 C₁ to C₄ alkyl or alkenyl substituent;

- Group 2: perfuming ingredients comprising a cyclopentane, cyclopentene, cyclopentanone or cyclopentenone ring substituted with at least one linear or branched C₄ to C₈ 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 C₅ to C₈ alkyl or alkenyl substituent or with at least one phenyl substituent and optionally one or more linear or branched C₁ to C₃ alkyl or alkenyl substituents;
 - Group 4: perfuming ingredients comprising at least two fused or linked C₅ and/or C₆ rings;
 - Group 5: perfuming ingredients comprising a camphor-like ring structure;
 - Group 6: perfuming ingredients comprising at least one C₇ to C₂₀ 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, (1R,3R,4R)-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), (3R,3aR,6S,7aR)-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);
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- 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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 - Group 4: Methyl cedryl ketone (origin: International Flavors and Fragrances, USA), a mixture of (1RS,2SR,6RS,7RS,8SR)-tricyclo[5.2.1.0^{2,6}]dec-3-en-8-yl 2-methylpropanoate and (1RS,2SR,6RS,7RS,8SR)-tricyclo[5.2.1.0^{2,6}]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-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-

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- 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: Firmenich SA, Geneva, Switzerland), 9/10-ethylidene-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 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, 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^{2,7}]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), pentadecenolide (origin: Firmenich SA, Geneva, Switzerland), 3-methyl-(4/5)-cyclopentadecanone, (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-ethoxyethoxycyclododecane (origin: Firmenich SA, Geneva, 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 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, 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 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/cm³.

“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”*).

A *“density balancing material”* should be understood as a material having a density greater than 1.07 g/cm³ 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.

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/cm³ 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]cyclopropylmethanol, (+-)-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-[(1RS,2SR)-2,6,6-trimethyl-1-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, methyl naphthylketone, (+)-(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-propenyl)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^{2,6}]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, methyl nonylacetaldehyde, 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, epoxydes, 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, epoxydes, 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-propanyl)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 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-[(1RS,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, 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, (+)-(1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl propanoate, and mixtures thereof.

5 According to an embodiment, the core comprises a perfume formulation comprising:

- 0 to 60 wt.% of a hydrophobic solvent (based on the total weight of the perfume formulation),
- 40 to 100 wt.% of a perfume oil (based on the total weight of the perfume
10 formulation), wherein the perfume oil has at least two, preferably all of the following characteristics:
 - o 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,
 - 15 o 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 herein defined and
 - o at least 15%, preferably at least 20%, more preferably at least 25%, even more preferably at least 30% of high impact perfume materials
20 having a Log T < -4,

optionally, further hydrophobic active ingredients.

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

25 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.

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

The term "Hansen solubility parameter" is understood refers 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 δD is the Hansen dispersion value (also referred to in the following as the atomic dispersion fore), δP is the Hansen polarizability value (also referred to in the following as the dipole moment), and δH 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 (δD) from 12 to 20, a dipole moment (δP) from 1 to 8, and a hydrogen bonding (δH) 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 (δD) from 12 to 20, preferably from 14 to 20, a dipole moment (δP) from 1 to 8, preferably from 1 to 7, and a hydrogen bonding (δH) from 2.5 to 11, preferably from 4 to 11.

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 (δD) from 12 to 20, a dipole moment (δP) from 1 to 8, and a hydrogen bonding (δH) 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 (δD) from 12 to 20, preferably from 14 to 20, a dipole moment (δP) from 1 to 8, preferably from 1 to 7, and a hydrogen bonding (δH) 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 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, preferable 4.0 and higher and more preferable 4.5
- iii. at least two Hansen solubility parameters selected from a first group consisting 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 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-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 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.

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 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.

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 rodenticide; and/or an antimicrobial such as a germicide, antibiotic, antibacterial, antiviral, antifungal, antiprotazoal 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 or animals, pests include insects notably arthropods, mites, spiders, fungi, weeds, bacteria and other microorganisms.

By "flavor oil", it is meant here a flavoring ingredient or a mixture of flavoring ingredients, solvents or adjuvants of current use for the preparation of a flavoring formulation, i.e. a particular mixture of ingredients which is intended to be added to an edible composition or chewable product to impart, improve or modify its organoleptic properties, in particular its flavor and/or taste. Flavoring ingredients are well known to a person skilled in the art and their nature does not warrant a detailed description here, which in any case would not be exhaustive, the skilled flavorist being able to select them on the basis of his general knowledge and according to the intended use or application and the organoleptic effect it is desired to achieve. Many of these flavoring ingredients are listed in reference texts such as in the book by S. Arctander, *Perfume and Flavor Chemicals*, 1969, Montclair, N.J., USA, or its more recent versions, or in other works of similar nature such as *Fenaroli's Handbook of Flavor Ingredients*, 1975, CRC Press or *Synthetic Food Adjuncts*, 1947, by M. B. Jacobs, van Nostrand Co., Inc.

Solvents and adjuvants of current use for the preparation of a flavoring formulation are also well known in the art.

In a particular embodiment, the flavor is a mint flavor. In a more particular embodiment, the mint is selected from the group consisting of peppermint and spearmint.

In a further embodiment, the flavor is a cooling agent or mixtures thereof.

In another embodiment, the flavor is a menthol flavor.

Flavors that are derived from or based on fruits where citric acid is the predominant, naturally-occurring acid include but are not limited to, for example, citrus fruits (e.g. lemon, lime), limonene, strawberry, orange, and pineapple. In one embodiment, the flavors food is lemon, lime or orange juice extracted directly from the fruit. Further embodiments of the flavor comprise the juice or liquid extracted from oranges, lemons, grapefruits, key limes, citrons, clementines, mandarins, tangerines, and any other citrus fruit, or variation or hybrid thereof. In a particular embodiment, the flavor comprises a liquid extracted or distilled from oranges, lemons, grapefruits, key limes, citrons, clementines, mandarins, tangerines, any other citrus fruit or variation or hybrid thereof, pomegranates, kiwifruits, watermelons, apples, bananas, blueberries, melons, ginger, bell peppers, cucumbers, passion fruits, mangos, pears, tomatoes, and strawberries.

In a particular embodiment, the flavor comprises a composition that comprises limonene, in a particular embodiment, the composition is a citrus that further comprises limonene.

In another particular embodiment, the flavor comprises a flavor selected from the group comprising strawberry, orange, lime, tropical, berry mix, and pineapple.

The phrase flavor includes not only flavors that impart or modify the smell of foods but include taste imparting or modifying ingredients. The latter do not necessarily have a taste or smell themselves but are capable of modifying the taste that other ingredients provides, for instance, salt enhancing ingredients, sweetness enhancing ingredients, umami enhancing ingredients, bitterness blocking ingredients and so on.

In a further embodiment, suitable sweetening components may be included in the particles described herein. In a particular embodiment, a sweetening component is selected from the group consisting of sugar (e.g., but not limited to sucrose), a stevia component (such as but not limited to stevioside or rebaudioside A), sodium

cyclamate, aspartame, sucralose, sodium saccharine, and Acesulfam K or mixtures thereof.

Compound having at least two P-Cl functions

5 According to the invention, the shell of the microcapsule comprises the reaction product between at least one compound having at least two P-Cl functions and a nucleophile.

By "compound having at least two P-Cl functions" it should be understood that the P-Cl functions can be on the same P atom or on two different P atoms in the same
10 molecule.

According to an embodiment, the term "P-Cl function" refers to a covalent bond between a P atom and a Cl atom.

According to an embodiment, the at least two Cl functions are on the same P atom.

15 According to an embodiment, the compound having at least two P-Cl functions comprises a P atom that is connected to at least two Cl atoms via covalent bonds.

The compound having at least two P-Cl functions can be selected from the group consisting of compound with formula $PXCl_3$, where X is O, S, or NH or NR^1 where R^1 is any hydrocarbon group, optionally comprising heteroatoms. Preferably,
20 the compound having at least two P-Cl functions can be selected from the group consisting of compound phosphorus oxy trichloride ($POCl_3$), thiophosphorus trichloride ($PSCl_3$), phosphorimidic trichloride ($PNHCl_3$), N-substituted-phosphorimidic trichloride (PNR^1Cl_3), phosphorus trichloride, phosphorus pentachloride, hexachlorocyclophosphazene (HCCP) and its derivatives, R_1OPXCl_2 (with X is O, NH,
25 NR^1 or S), such as alkyl phosphorodichloridate (R^1OPOCl_2) and/or alkyl dichlorothiophosphate (R^1OPSCl_2) (wherein R^1 can be any hydrocarbon group optionally with heteroatoms); poly(dichlorophosphazene), 3,9-dichloro-2,4,8,10-tetraoxa-3 λ 5,9 λ 5-diphosphaspiro[5.5]undecane 3,9-dioxide, and mixtures thereof.

The compound having at least two P-Cl functions can be selected from the
30 group consisting of compounds with formula $PXCl_3$, $PNHCl_3$ and PNR^1Cl_3 , where X is O, S, where R^1 is any hydrocarbon group, optionally comprising heteroatoms. Preferably, the compound having at least two P-Cl functions can be selected from the group consisting of compound phosphorus oxy trichloride ($POCl_3$), thiophosphorus trichloride ($PSCl_3$), phosphorimidic trichloride ($PNHCl_3$), N-substituted-phosphorimidic

trichloride (PNR^1Cl_3), phosphorus trichloride, phosphorus pentachloride, hexachlorocyclophosphazene (HCCP), 2,2,4,4-tetraamino-6,6-dichlorocyclotriphosphazene (TCPA), a copolymer of hexa-(4-aldehyde-phenoxy)-cyclo-triphosphazene and 2,2,4,4-tetraamino-6,6-dichlorocyclotriphosphazene (HACP-co-TCPA), R_1OPXCl_2 (with X is O, NH, NR^1 or S), such as alkyl phosphorodichloridate (R^1OPOCl_2) and/or alkyl dichlorothiophosphate (R^1OPSCl_2) (wherein R^1 can be any hydrocarbon group optionally with heteroatoms); poly(dichlorophosphazene), 3,9-dichloro-2,4,8,10-tetraoxa-3 λ 5,9 λ 5-diphosphaspiro[5.5]undecane 3,9-dioxide, and mixtures thereof.

10

According to an embodiment, the hydrocarbon group is a functional or not C_1 - C_{50} hydrocarbon group.

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.

30

It is understood that with the term "... a hydrocarbon group, optionally comprising ..." it is meant that said hydrocarbon group optionally comprises one, two, three or more heteroatoms. 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. For example, a $-\text{CH}_2-\text{CH}_2-\text{CHOH}-\text{CH}_2-$ group represents a C_4 hydrocarbon group comprising an alcohol group (substitution of a hydrogen atom), i.e. a C_4 hydrocarbon comprising an oxygen atom; a $-\text{CH}_2-\text{CH}_2-\text{COO}-\text{CH}_2-\text{CH}_2\text{CH}_2-\text{CH}_2-$ group represents a C_7 hydrocarbon group comprising one ester group (substitution of carbon atoms/insertion into the hydrocarbon chain), i.e. a C_7 hydrocarbon comprising two oxygen atoms and, similarly, a $-\text{CH}_2-\text{CH}_2-\text{O}-\text{CH}_2-\text{CH}_2-\text{O}-\text{CH}_2-\text{CH}_2-$ group represents a C_6 hydrocarbon group comprising two ether groups, i.e. a C_6 hydrocarbon comprising two oxygen atoms.

10 According to a particular embodiment, the compound having at least two P-Cl functions is hexachlorocyclophosphazene (HCCP) or its derivatives or its polymer.

According to a particular embodiment, the compound having at least two P-Cl functions is selected from the group consisting of hexachlorocyclophosphazene (HCCP), 2,2,4,4-tetraamino-6,6-dichlorocyclotriphosphazene (TCPA), a copolymer of
15 hexa-(4-aldehyde-phenoxy)-cyclo-triphosphazene (HACP) and 2,2,4,4-tetraamino-6,6-dichlorocyclotriphosphazene (HACP-co-TCPA) or mixtures thereof.

According to an embodiment, the compound having at least two P-Cl functions is not p-(chlorocarbonyl)-phenyl-dichlorophosphate, [2,2-bis-(4-hydroxyphenyl)-propane]-bisdichlorophosphate, phenylphosphonic dichloride or reaction products
20 thereof or mixtures thereof.

Nucleophile

According to the invention, a nucleophile compound is defined as a chemical
25 species that donates an electron pair to an electrophile to form a chemical bond in relation to a reaction.

The nucleophile compound may be chosen in the group consisting of nitrogen nucleophile, sulfur nucleophile, oxygen nucleophile, carbon nucleophile, phosphor nucleophile, and mixtures thereof.

30 Nitrogen nucleophile compound may have at least one functional group chosen in the group consisting of ammonia, azide, amines, nitrites, hydroxylamine, hydrazin, carbazide, phenylhydrazine, semicarbazide, and amide, and mixtures thereof.

Sulfur nucleophile compound may have at least one functional group chosen in the group consisting of hydrogen sulfide and its salts, thiols (RSH), thiolate anions

(RS⁻), anions of thiolcarboxylic acids (RC(O)-S⁻), and anions of dithiocarbonates (RO-C(S)-S⁻) and dithiocarbamates (R₂N-C(S)-S⁻) and mixtures thereof.

Oxygen nucleophile compound may have at least one functional group chosen in the group consisting of water, hydroxide anion, alcohols, alkoxide anions, carboxylate anions, carbonates, sulfonate, sulfate, sodium phosphates, sodium silicates, borax, sodium tetraborate, and mixtures thereof.

Carbone nucleophile compound may have at least one functional group chosen in the group consisting of enols carbon nucleophiles, malonate and acetoacetate.

Phosphor nucleophile compound may have at least one functional group chosen in the group consisting of phosphine, phosphite anion and mixture thereof.

According to an embodiment, the nucleophile is chosen in the group consisting of alcohol, amine, thiol and mixtures thereof.

"Alcohol" and "polyol" can be used indifferently in the present invention.

According to a particular embodiment, the amine is chosen in the group consisting of cystamine, EDA, DETA, Pentamethylenediamine, hexamethylenediamine, poly(L-lysine) (that can be modified), L-lysine, L-lysine ethyl ester, cystine dimethyl ester, and mixtures thereof.

According to a particular embodiment, the amine is chosen in the group consisting of cystamine, cystamine hydrochloride, cystine, cystine hydrochloride, cystine dialkyl ester, cystine dialkyl ester hydrochloride, a xylylene diamine, 1,2-diaminocyclohexane, 1,4-diaminocyclohexane, polyetheramines, ethylene diamine, diethylene triamine, spermine, spermidine, polyamidoamine (PAMAM), guanidine carbonate, chitosan, tris-(2-aminoethyl)amine, 1,4 Diaminobutane, 2,2Dimethyl-1,3-propanediamine, 1,3-diaminopentane (Dytek EP diamine), 1,2 Diaminopropane, triethylenetetramine, 1,3-diaminopropane; urea; ethylene urea; aminoguanidine bicarbonate; 1-(2-aminoethyl)imidazolidin-2-one; N-(3-aminopropyl)-N-dodecylpropane-1,3-diamine; N1-(2-Aminoethyl)-N1-dodecyl-1,2-ethanediamine; aminoethylethanolamine; N1-(3-aminopropyl)propane-1,3-diamine, polyethyleneimine, C36-alkylenediamines, (as an example PriamineTM - (1E,19E)-10,11-dioctylcosa-1,19-diene-1,20-diamine (low-viscous, bio-based, dimer diamine)), amino-acid such as lysine, arginine, leucine, histidine, tryptophane, serin, glutamine, threonine, alanine, asparagine, aspartic acid, cysteine, glutamic acid, glycine, isoleucine, methionine, phenylalanine, proline, tyrosine, valine, 2,2,4,4-tetraamino-6,6-dichlorocyclotriphosphazene (TCPA) and mixtures thereof.

According to a particular embodiment, the alcohol (or polyol) is chosen in the group consisting of phloroglucinol, Hydroquinone, Hydroquinone bis(2-hydroxyethyl)ether, 1,4-cyclohexanedimethanol, Resorcinol, Isosorbide, Pentaerythritol, Erythritol, Trimethylolpropane, Di-trimethylolpropane, sorbitol, mannitol, xylitol, triethanolamine, diglycerol, triglycerol and corresponding partial esters, polyglycerol and corresponding partial esters, and mixtures thereof.

According to a particular embodiment, the thiol (or polythiol) is chosen Ethylene glycol bis-mercaptoacetate, dithiothreitol, trimethylolpropane tris(3-mercaptopropionate), 2,2'-Thiodiethanethiol, Pentaerythritol tetrakis(3-mercaptopropionate), 2-mercaptoethanol, 3-Aminopropane-1-thiol, 3-Aminopropane-1-thiol hydrochloride, 3-mercapto-1,2-propanediol, 3-Mercapto-1-propanol, 1-Mercapto-2-propanol, 6-Mercapto-1-hexanol, Mercapto-acetic acid 4-mercaptoacetoxyl ester, 1-Thioglycéról, 2-{2-[2-(2-Mercaptoethoxy)ethoxy]ethoxy}ethanol, cysteamine, cysteamine hydrochloride, 4-Mercaptophenol, 4,6-Diamino-2-pyrimidinethiol, 4,6-Dihydroxy-2-mercaptopyrimidine, Propane-1,3-dithiol, and mixtures thereof.

According to an embodiment, the nucleophile is not an amine or an alcohol.

According to an embodiment, the nucleophile is not hexamethylenediamine, polyvinyl alcohol, ethylenediamine, diethylenetriamine, a melamine-based crosslinker or any reaction product thereof or mixtures thereof.

According to an embodiment, the nucleophile and the compound having at least two P-Cl functions are the same molecule.

According to an embodiment, the nucleophile and the compound having at least two P-Cl functions are the same molecule, wherein the molecule is 2,2,4,4-tetraamino-6,6-dichlorocyclotriphosphazene.

According to an embodiment, the nucleophile and the compound having at least two P-Cl functions are not the same molecule.

According to an embodiment, the core-shell microcapsule does not comprise a silicon containing monomer or a reaction product thereof.

According to an embodiment, the core-shell microcapsule does not comprise silicon.

5 Biodegradability

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

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

- 10 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.

- Thereby it is understood that the core-shell microcapsule including all components, such as the core, shell and optionally coating may have a
15 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.

In a particular embodiment, the oil core, preferably perfume oil, 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.

- 20 OECD301F is a standard test method on the biodegradability from the Organization of 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*.

25 Outer coating

- According to a particular embodiment of the invention, the microcapsule comprises an outer coating, wherein the outer coating comprises a coating material selected from the group consisting of a non-ionic polymer (such as non-ionic polysaccharide), anionic polymer (such as polysaccharide), a cationic polymer, a
30 polysuccinimide derivative (as described for instance in WO2021185724) and mixtures thereof to form an outer coating to the microcapsule.

According to a particular embodiment, the microcapsule does not comprise an outer coating.

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.

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 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 (M_w) 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 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. 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 microcapsule of the present invention comprises a mineral layer. The mineral layer preferably comprises a material chosen in the group consisting of iron oxides, iron oxyhydroxide, titanium oxides, zinc oxides, calcium carbonates, calcium phosphates, barium salt, strontium salt, magnesium salt, and mixtures thereof and mixtures thereof.

15 Solid particles

Another object of the invention is a solid particle comprising:

- a carrier material, preferably a polymeric carrier material chosen in the group consisting of polyvinyl acetate, polyvinyl alcohol, dextrans, natural or modified starch, vegetable gums, pectins, xanthans, alginates, carragenans, cellulose derivatives and mixtures thereof, and
- microcapsules as defined above entrapped in said carrier material, and
- optionally free perfume entrapped in said carrier material.

Solid particle as defined above and microcapsule powder can be used indifferently in the present invention.

25 **PROCESS FOR PREPARING MICROCAPSULES**

Another object of the invention is a process for preparing a core-shell microcapsule slurry comprising the steps of:

- a) Optionally, providing at least one compound having at least two P-Cl functions
- 30 b) admixing at least one hydrophobic material with the optional compound obtained in step a) to form an oil phase;
- c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;

- d) Optionally, dispersing the two-phases dispersion into a continuous phase C2 comprising optionally at least one compound having at least two P-Cl functions to form a multiple dispersion;
- e) curing the dispersion obtained in step c) or d) to form microcapsules in the form of a slurry,
- 5 wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d) and/or in step e).

It should be understood that, according to the invention, at least one compound having at least two P-Cl functions is added in step a) and/or in step d).

It should also be understood that, the at least one compound having at least two P-Cl functions used in step a) and/or in step d) can be used as a pure compound (meaning without any reaction with a reactant) or can be used as a modified compound if a reactant is added in step a) and/or step b) and/or step c) and/or step d) and/or in step e).

It should be understood that the reactant is a nucleophile which can be the same or a different nucleophile defined in the present invention.

- 20 According to a particular embodiment, the process comprises the steps of:
- a) Providing at least one compound having at least two P-Cl functions
 - b) admixing at least one hydrophobic material with the compound obtained in step a) to form an oil phase;
 - c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;
 - 25 d) curing the dispersion obtained in step c) to form microcapsules in the form of a slurry,
- wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d).

30

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

- a) Providing at least one compound having at least two P-Cl functions
- b) admixing at least one hydrophobic material with the compound obtained in step a) to form an oil phase;

- 5 c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;
d) dispersing the two-phases dispersion into a continuous phase C2 comprising optionally at least one compound having at least two P-Cl functions to form a multiple dispersion;
e) curing the dispersion obtained in step c) or d) to form microcapsules in the form of a slurry,
wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d) and/or in step e).

10

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

- a) Providing at least one compound having at least two P-Cl functions
b) admixing at least one hydrophobic material with the compound obtained in step a) to form an oil phase;
15 c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;
d) dispersing the two-phases dispersion into a continuous phase C2 comprising at least one compound having at least two P-Cl functions to form a multiple dispersion;
20 e) curing the dispersion obtained in step c) or d) to form microcapsules in the form of a slurry,
wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d) and/or in step e).

25

The compound having at least two P-Cl functions of step d) and be the same or can be different from the compound having at least two P-Cl functions of step a).

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

- a) providing at least one hydrophobic material to form an oil phase;
30 b) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;
c) dispersing the two-phases dispersion into a continuous phase C2 comprising at least one compound having at least two P-Cl functions to form a multiple dispersion;

d) curing the dispersion obtained in step c) or d) to form microcapsules in the form of a slurry,
wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d).

5

According to a particular embodiment, the process comprises the steps of

- a) Optionally, providing at least one compound having at least two P-Cl functions
- b) admixing at least one hydrophobic material with the optional compound obtained in step a) to form an oil phase;
- 10 c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;
- d) Optionally, dispersing the two-phases dispersion into a continuous phase C2 comprising optionally at least one compound having at least two P-Cl functions to form a multiple dispersion;
- 15 e) curing the dispersion obtained in step c) or d) to form microcapsules in the form of a slurry,
wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d) and/or in step e), and
wherein a compound having at least two P-Cl functions is added in step a)
- 20 and/or step b) and/or step c) and/or step d).

The embodiments disclosed for the core-shell microcapsules also apply for the process for preparing core-shell microcapsules. It applies notably to the compound having at least two P-Cl functions, the hydrophobic material, or the nucleophile.

25

In step a) of the process, a solution comprising at least one compound having at least two P-Cl functions is prepared.

The compound having at least two P-Cl functions is defined as previously.

- The compound having at least two P-Cl functions is added in an amount comprised
- 30 between 0.5 and 10%, preferably between 2 and 6% by weight based on the total weight of the two-phases dispersion.

Step b) of the process consists of admixing at least one hydrophobic material with solution obtained in step a) to form an oil phase.

In step c) of the process, the oil phase is dispersed into a continuous phase C1 or a continuous phase C1 is dispersed into the oil phase to form a two-phases dispersion.

According to an embodiment, the continuous phase C1 comprises, preferably
5 consists of water.

According to an embodiment, the continuous phase C1 is a water phase.

According to an embodiment, the two-phases dispersion is an oil-in-water emulsion.

According to an embodiment, the two-phases dispersion is a water-in-oil emulsion.

10 According to an embodiment, the continuous phase C1 comprises water and an alcohol such as glycerol, 1,4-butanediol, ethylene glycol and mixtures thereof.

According to an embodiment, the continuous phase C1 consists of alcohol.

Then, optionally, in step d) the two-phases dispersion is dispersed into a
15 continuous phase C2 to form a multiple dispersion.

The continuous phase C2 can be any hydrophobic solvent or free oil.

The multiple dispersion can be an O/W/O (oil in water in oil) or W/O/W (water in oil in water) emulsions.

A stabilizer can be added in the oil phase and/or in the continuous phase C1.

20 By "stabilizer", it is meant a compound capable to stabilize oil/dispersing phase interface (typically oil/water interface) as an emulsion.

The stabilizer is preferably used in an amount comprised between 0.05% to 20 %, preferably between 0.1 to 10%, even more preferably between 0.1 and 5% by weight based on the two-phases dispersion, preferably oil-in-water emulsion.

25 "Stabilizer" or "emulsifier" can be used indifferently in the present invention.

According to an embodiment, the stabilizer can act as a nucleophile. Therefore, according to an embodiment, the stabilizer is the nucleophile of the present invention.

According to an embodiment, the stabilizer is a colloidal stabilizer.

The colloidal stabilizer can be a polymeric emulsifier (standard emulsion), a
30 surfactant, or solid particles (Pickering emulsion).

By "polymeric emulsifier", it meant an emulsifier having both a polar group with an affinity for water (hydrophilic) and a nonpolar group with an affinity for oil (hydrophobic). The hydrophilic part will dissolve in the water phase and the hydrophobic part will dissolve in the oil phase providing a film around droplets.

By "surfactant", it meant a substance with a polar and a non-polar group that is added to the liquid to reduce the liquid surface tension.

According to an embodiment, the stabilizer is chosen in the group consisting of inorganic particles, polymeric emulsifier such as polysaccharides, proteins, glycoproteins, and mixtures thereof.

When the stabilizer is solid particles, it can be chosen in the group consisting of 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.

According to a particular embodiment, the stabilizer is a biopolymer.

By "biopolymers" it is meant biomacromolecules produced by living organisms. Biopolymers are characterized by molecular weight distributions ranging from 1,000 (1 thousand) to 1,000,000,000 (1 billion) Daltons. These macromolecules may be carbohydrates (sugar based) or proteins (amino-acid based) or a combination of both (gums) and can be linear or branched.

According to an embodiment, the polymeric emulsifier is chosen in the group consisting of gum Arabic, modified starch, polyvinyl alcohol, polyvinylpyrrolidone (PVP), carboxymethylcellulose (CMC), anionic polysaccharides, acrylamide copolymer, protein such as soy protein, rice protein, whey protein, white egg albumin, sodium caseinate, gelatin, bovine serum albumin, hydrolyzed soy protein, hydrolyzed sericin, pseudocollagen, silk protein, sericin powder, potato protein, chickpea protein, pea protein, algae protein, faba bean protein, barley protein, oat protein, wheat gluten protein, lupin protein, and mixtures thereof.

Potato proteins are typically extracted from potato tuber (*Solanum tuberosum*). According to an embodiment, the potato protein is a native potato protein and preferably patatin.

The protein used in this invention may be native, partially or completely denaturated by any suitable method. Denaturation is a process which modify the conformational structure of a protein by unfolding, i.e., it involves the disruption and possible destruction of both the secondary and tertiary structures of the protein. Indeed, denaturation implicates the breaking of many of the weak linkages, or bonds (e.g., hydrogen bonds), within a protein molecule that are responsible for the highly

ordered structure of the protein in its native state. Denaturation is reversible (the proteins can regain their native state when the denaturing influence is removed) or irreversible.

Denaturation can be brought about in various ways. Proteins can be denatured
5 by exposure to temperature, radiation or mechanical stress including shear, changes in pH (treatment with a base or an acid), treatment with oxidizing or reducing agents, inorganic salt, certain organic solvents, chaotropic agents (i.e, compounds having a positive chaotropic value - kJ Kg^{-1} mole on the Hallsworth Scale - such as guanidine salts - e.g., guanidine carbonate, guanidine hydrochloride -, urea, calcium chloride, n-
10 butanol, ethanol, lithium perchlorate, lithium acetate, magnesium chloride, phenol, 2-propanol, sodium dodecyl sulfate, thiourea).

The protein used in this invention can also be derivatized or modified (e.g., derivatized or chemically modified). For example, the protein can be modified by covalently attaching sugars, lipids, peptides or chemical groups such as phosphates
15 or methyl.

According to an embodiment, a base is added in the continuous phase C1. Preferably the base is chosen in the group consisting of guanidine carbonate, sodium carbonate, potassium carbonate, sodium hydroxide, potassium hydroxide, and mixtures thereof.

20 When used, the base is typically used in an amount comprised between 0.1 and 10%, preferably between 3 and 7% by weight based on the total weight of the continuous phase C1.

The nucleophile is defined as previously.

According to an embodiment, the nucleophile is added in the oil phase and/or
25 in the continuous phase C1.

The nucleophile can be added in an amount comprised between 0.5 and 10%, preferably between 1 and 4% by weight based on the total weight of the two-phases dispersion.

In step d), the dispersion obtained in step c) or d) is cured to form microcapsules
30 in the form of a slurry.

Optionally, a non-ionic polymer (such as non-ionic polysaccharide), anionic polymer (such as polysaccharide), a cationic polymer, a polysuccinimide derivative (as described for instance in WO2021185724) and mixtures thereof, as defined previously, can be added in step e) or after step e) to form an outer coating.

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 24 hours. More preferably it is performed at between 10 and 80°C for between
5 30 minutes and 5 hours.

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

- a) Preparing a solution comprising admixing at least one hydrophobic material with at least one compound having at least two P-Cl functions to form an oil
10 phase;
- b) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous phase C1 into the oil phase to form a two-phases dispersion;
- c) Optionally, dispersing the two-phases dispersion into a continuous phase C2 to form a multiple dispersion;
- 15 d) curing the dispersion obtained in step c) or d) to form microcapsules in the form of a slurry,
wherein a nucleophile is added in step a) and/or step b).

According to an embodiment, the process does not comprise the addition of a silicon
20 containing monomer.

MULTIPLE MICROCAPSULES 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.

- 25 Another object of the invention is a microcapsule delivery system comprising:
- the microcapsules of the present invention as a first type of microcapsule, and
 - a second type of microcapsules, wherein the first type of microcapsule and the second type of microcapsules differ in their hydrophobic material and/or their wall material and/or in their coating material.

30

According to a particular embodiment, the microcapsule delivery system is in the form of a slurry.

The wall of the second type of microcapsules can vary. As non-limiting examples, the polymer shell of the second type of microcapsules comprises a material selected from the group consisting of polyurea, polyurethane, polyamide, polyhydroxyalkanoates, polyacrylate, polyesters, polyaminoesters, polyepoxides, organosilicon, polycarbonate, polysulfonamide, urea formaldehyde, melamine formaldehyde resin, melamine formaldehyde resin cross-linked with polyisocyanate or aromatic polyols, melamine urea resin, melamine glyoxal resin, gelatin/ gum arabic shell wall, and mixtures thereof.

The second type of microcapsule can comprise an oil-based core comprising a hydrophobic active, preferably perfume, and a composite shell comprising a first material and a second material, wherein the first material and the second material are different, the first material is a coacervate, the second material is a polymeric material. In a particular embodiment, the weight ratio between the first material and the second material is comprised between 50:50 and 99.9:0.1. In a particular embodiment, the coacervate comprises a first polyelectrolyte, preferably selected among proteins (such as gelatin), polypeptides or polysaccharides (such as chitosan), most preferably Gelatin and a second polyelectrolyte, preferably alginate salts, cellulose derivatives guar gum, pectinate salts, carrageenan, polyacrylic and methacrylic acid or xanthan gum, or yet plant gums such as acacia gum (Gum Arabic), most preferably Gum Arabic. The coacervate first material can be hardened chemically using a suitable cross-linker such as glutaraldehyde, glyoxal, formaldehyde, tannic acid or genipin or can be hardened enzymatically using an enzyme such as transglutaminase. The second polymeric material can be selected from the group consisting of polyurea, polyurethane, polyamide, polyester, polyacrylate, organosilicon, polycarbonate, polysulfonamide, polymers of urea and formaldehyde, melamine and formaldehyde, melamine and urea, or melamine and glyoxal and mixtures thereof, preferably polyurea and/or polyurethane. The second material is preferably present in an amount less than 3 wt. %, preferably less than 1 wt. % based on the total weight of the second type of microcapsule slurry.

As non-limiting examples, the shell of the second type of microcapsules can be aminoplast-based, polyurea-based or polyurethane-based. The shell of the second type of microcapsules can also be hybrid, namely organic-inorganic such as a hybrid shell composed of at least two types of inorganic particles that are cross-linked, or yet

a shell resulting from the hydrolysis and condensation reaction of a polyalkoxysilane macro-monomeric composition.

According to an aspect, the shell of the second type of microcapsules comprises an aminoplast copolymer, such as melamine-formaldehyde or urea-
5 formaldehyde or cross-linked melamine formaldehyde or melamine glyoxal.

According to another aspect the shell of the second type of microcapsules is polyurea-based made from, for example but not limited to isocyanate-based monomers and amine-containing crosslinkers such as guanidine carbonate and/or guanazole. Certain polyurea microcapsules comprise a polyurea wall which is the
10 reaction product of the polymerisation between at least one polyisocyanate comprising at least two isocyanate functional groups and at least one reactant selected from the group consisting of an amine (for example a water-soluble guanidine salt and guanidine); a colloidal stabilizer or emulsifier; and an encapsulated perfume. However, the use of an amine can be omitted. According to a particular aspect, the
15 colloidal stabilizer includes an aqueous solution of between 0.1% and 0.4% of polyvinyl alcohol, between 0.6% and 1% of a cationic copolymer of vinylpyrrolidone and of a quaternized vinylimidazol (all percentages being defined by weight relative to the total weight of the colloidal stabilizer). According to another aspect, the emulsifier is an anionic or amphiphilic biopolymer, which may be, in one aspect, chosen from the
20 group consisting of gum Arabic, soy protein, gelatin, sodium caseinate and mixtures thereof.

According to another embodiment, the microcapsule wall material of the second type of microcapsules may comprise any suitable resin and especially including melamine, glyoxal, polyurea, polyurethane, polyamide, polyester, etc. Suitable resins
25 include the reaction product of an aldehyde and an amine, suitable aldehydes include, formaldehyde and glyoxal. Suitable amines include melamine, urea, benzoguanamine, glycoluril, and mixtures thereof. Suitable melamines include, methylol melamine, methylated methylol melamine, imino melamine and mixtures thereof. Suitable ureas include, dimethylol urea, methylated dimethylol urea, urea-
30 resorcinol, and mixtures thereof. Suitable materials for making may be obtained from one or more of the following companies Solutia Inc. (St Louis, Missouri U.S.A.), Cytec Industries (West Paterson, New Jersey U.S.A.), Sigma-Aldrich (St. Louis, Missouri U.S.A.).

According to another embodiment, the second type of microcapsules is a one-shell aminoplast core-shell microcapsule obtainable by a process comprising the steps of:

- 1) admixing a perfume oil with at least a polyisocyanate having at least two isocyanate functional groups to form an oil phase;
- 5 2) dispersing or dissolving into water an aminoplast resin and optionally a stabilizer to form a dispersing phase;
- 3) preparing an oil-in-water dispersion, wherein the mean droplet size is comprised between 1 and 100 microns, by admixing the oil phase and the dispersing phase;
- 10 4) performing a curing step to form the wall of said microcapsule; and
- 5) optionally drying the final dispersion to obtain the dried core-shell microcapsule.

According to an embodiment, the second type of microcapsules is a formaldehyde-free capsule. A typical process for the preparation of aminoplast formaldehyde-free microcapsules slurry comprises the steps of

- 15 1) preparing an oligomeric composition comprising the reaction product of, or obtainable by reacting together:
 - a. a polyamine component in the form of melamine or of a mixture of melamine and at least one C₁-C₄ compound comprising two NH₂ functional groups;
 - 20 b. an aldehyde component in the form of a mixture of glyoxal, a C₄₋₆ 2,2-dialkoxy-ethanal and optionally a glyoxalate, said mixture having a molar ratio glyoxal/C₄₋₆ 2,2-dialkoxy-ethanal comprised between 1/1 and 10/1; and
 - c. a protic acid catalyst;
- 25 2) preparing an oil-in-water dispersion, wherein the droplet size is comprised between 1 and 600 microns, and comprising:
 - a. an oil;
 - b. a water medium;
 - c. at least an oligomeric composition as obtained in step 1;
 - 30 d. at least a cross-linker selected amongst:
 - i. C₄-C₁₂ aromatic or aliphatic di- or tri-isocyanates and their biurets, triurets, trimers, trimethylol propane-adduct and mixtures thereof; and/or
 - ii. a di- or tri-oxiran compounds of formula:

A-(oxiran-2-ylmethyl)_n

wherein _n stands for 2 or 3 and 1 represents a C₂-C₆ group optionally comprising from 2 to 6 nitrogen and/or oxygen atoms;

5 e. optionally a C₁-C₄ compounds comprising two NH₂ functional groups;

3) Heating the dispersion; and

4) Cooling the dispersion.

In another particular embodiment, the second type of microcapsule comprises

- an oil-based core comprising a hydrophobic active, preferably perfume,
- 10 – optionally an inner shell made of a polymerized polyfunctional monomer;
- a biopolymer shell comprising a protein, wherein at least one protein is cross-linked.

According to a particular embodiment, the protein is chosen in the group consisting of milk proteins, caseinate salts such as sodium caseinate or calcium
15 caseinate, casein, whey protein, hydrolyzed proteins, gelatins, gluten, pea protein, soy protein, silk protein and mixtures thereof, preferably sodium caseinate, most preferably sodium caseinate.

According to a particular embodiment, the protein comprises sodium caseinate and a globular protein, preferably chosen in the group consisting of whey protein, beta-lactoglobulin, ovalbumine, bovine serum albumin, vegetable proteins, and mixtures
20 thereof.

The protein is preferably a mixture of sodium caseinate and whey protein.

According to a particular embodiment, the biopolymer shell comprises a crosslinked protein chosen in the group consisting of sodium caseinate and/or whey
25 protein.

According to a particular embodiment, the second type of microcapsules slurry comprises at least one microcapsule made of:

- an oil-based core comprising the hydrophobic active, preferably perfume;
- an inner shell made of a polymerized polyfunctional monomer; preferably a
30 polyisocyanate having at least two isocyanate functional groups
- a biopolymer shell comprising a protein, wherein at least one protein is cross-linked; wherein the protein contains preferably a mixture comprising sodium caseinate and a globular protein, preferably whey protein,

- optionally at least an outer mineral layer.

According to an embodiment, sodium caseinate and/or whey protein is (are) cross-linked protein(s).

The weight ratio between sodium caseinate and whey protein is preferably comprised between 0.01 and 100, preferably between 0.1 and 10, more preferably between 0.2 and 5.

In another particular embodiment, the second type of microcapsules is a polyamide core-shell polyamide microcapsule comprising:

- an oil-based core comprising comprising a hydrophobic active, preferably perfume, and
- a polyamide shell comprising or being obtainable from:
 - an acyl chloride,
 - a first amino compound,
 - a second amino compound,
 - optionally, a carbohydrate.

According to a particular embodiment, the second type of microcapsules comprises:

- an oil-based core comprising a hydrophobic active, preferably perfume, and
- a polyamide shell comprising or being obtainable from:
 - an acyl chloride, preferably in an amount comprised between 5 and 98%, preferably between 20 and 98%, more preferably between 30 and 85% w/w
 - a first amino compound, preferably in an amount comprised between 1% and 50% w/w, preferably between 7 and 40% w/w;
 - a second amino compound, preferably in an amount comprised between 1% and 50% w/w, preferably between 2 and 25% w/w
 - a stabilizer, preferably a biopolymer, preferably in an amount comprised between 0 and 90%, preferably between 0.1 and 75%, more preferably between 1 and 70%,
 - optionally, a carbohydrate.

According to a particular embodiment, the second type of microcapsules comprises:

- an oil-based core comprising a hydrophobic active, preferably perfume, and
- 5 – a polyamide shell comprising or being obtainable from:
 - an acyl chloride,
 - a first amino-compound being an amino-acid, preferably chosen in the group consisting of L-Lysine, L-Arginine, L-Histidine, L-Tryptophane and/or mixtures thereof.
 - 10 • a second amino-compound, preferably chosen in the group consisting of ethylene diamine, diethylene triamine, cystamine and/or mixtures thereof, and
 - 15 • a biopolymer, preferably chosen in the group consisting of potato protein, chickpea protein, pea protein, algae protein, faba bean protein, barley protein, oat protein, wheat gluten protein, lupin protein, soy protein, rice protein, whey protein, white egg albumin, casein, sodium caseinate, gelatin (preferably fish gelatin), bovine serum albumin, hydrolyzed soy protein, hydrolyzed sericin, pseudocollagen, silk protein, sericin powder, gelatin and mixtures thereof,
 - 20 • optionally a carbohydrate, preferably selected from the group consisting of anionic salt of alginic acid, preferably alginic acid sodium salt, pectin, lignin, anionic modified starch, carboxymethylcellulose, carrageenan and mixtures thereof.

25 According to another aspect, the shell of the second type of microcapsules is polyurea- or polyurethane-based. Examples of processes for the preparation of polyurea and polyurethane-based microcapsule slurry are for instance described in International Patent Application Publication No. WO2007/004166, European Patent Application Publication No. EP 2300146, and European Patent Application Publication No. EP25799. Typically a process for the preparation of polyurea or polyurethane-based microcapsule slurry include the following steps:

- a) Dissolving at least one polyisocyanate having at least two isocyanate groups in an oil to form an oil phase;

- b) Preparing an aqueous solution of an emulsifier or colloidal stabilizer to form a dispersing phase;
- c) Adding the oil phase to the dispersing phase to form an oil-in-water dispersion, wherein the mean droplet size is comprised between 1 and 500 μm , preferably between 5 and 50 μm ; and
- d) Applying conditions sufficient to induce interfacial polymerisation and form microcapsules in the form of a slurry.

PERFUMING COMPOSITION AND CONSUMER PRODUCTS

- The present invention also relates to a perfuming composition comprising
- a core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above,
 - at least one ingredient selected from the group consisting of a perfumery carrier and a perfumery base, and
 - optionally, at least one perfumery adjuvant.

In a particular embodiment, the composition comprises:

- a core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above;
- an active ingredient, preferably chosen in the group consisting of a cosmetic ingredient, skin caring ingredient, perfume ingredient, flavor ingredient, malodour counteracting ingredient, bactericide ingredient, fungicide ingredient, pharmaceutical or agrochemical ingredient, a sanitizing ingredient, an insect repellent or attractant, and mixtures thereof.

- In a particular embodiment, a core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above can also be added in different perfumed consumer products

In a particular embodiment, present invention relates to a perfuming composition comprising

- a core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above and
- optionally, a free perfume oil.

Preferably, the perfuming composition according to the invention comprises between 0.1 and 40 %, preferably between 0.1 and 30% by weight of a core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above.

5 By "free perfume" it is herein understood a perfume or perfume oil which is comprised in the perfuming composition and not entrapped in the core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above.

10 In a particular embodiment, the total amount of the core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above is 0.05 to 5 wt.% (based on the total weight of the perfuming composition) and the total amount of the free perfume oil is 0.05 to 5 wt.% (based on the total weight of the perfuming composition).

15 In a particular embodiment, the total perfume oil of the perfume formulation entrapped in the core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above and total free perfume oil are present in the perfuming composition in a weight ratio of 1:20 to 20:1, preferably 10:1 to 1:10.

20 The perfuming composition can further comprise at least one perfuming co-ingredient and, optionally a perfumery adjuvant,

By "perfuming co-ingredient" it is herein understood a compound, which is used in a perfuming preparation or a composition to impart a hedonic effect and which is not a microcapsule as 20 defined above. In other words such a co-ingredient, to be considered as being a perfuming one, must be recognized by a person skilled in the art as being able to 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 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 sulfur heterocyclic compounds and essential oils, and said perfuming co-ingredients can be of natural or synthetic origin. Many of these co-30 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

5 in a controlled manner various types of perfuming compounds. Co-ingredients may be chosen in the group consisting of 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-

10 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-

15 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-

20 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 or a mixture thereof.

25 By "perfumery adjuvant" it is herein understood an ingredient capable of imparting additional 5 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.

30 The core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above can advantageously be used in many application fields and used in consumer products. The present invention also relates to a perfumed consumer product comprising

- a core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above, and
- a personal care, home care, or fabric care active base.

5 The consumer 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.

- 10 In particular a liquid consumer product comprising:
- from 2 to 65% by weight, relative to the total weight of the consumer product, of at least one surfactant;
 - water or a water-miscible hydrophilic organic solvent; and
 - a perfuming composition or the core-shell microcapsule or core-shell microcapsule
- 15 slurry obtained by the process according to the present invention or as described herein-above.

Also a powder consumer product comprising

- from 2 to 65% by weight, relative to the total weight of the consumer product, of at least one surfactant; and
- 20 - a perfuming composition or the core-shell microcapsule or core-shell microcapsule slurry obtained by the process according to the present invention or as described herein-above.

For the sake of clarity, it has to be mentioned that, by “perfumed consumer product” it is meant a consumer product which is expected to deliver among different

25 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 to the

30 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 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
5 perfectly able to select the suitable components on the basis of his general knowledge and of the available literature.

Non-limiting examples of suitable perfumed consumer products can be a fine perfume, a splash or eau de perfume, a cologne, a shave or after-shave lotion, a liquid or solid detergent, a mono or multi chamber unidose detergent, a fabric softener, a
10 fabric refresher, liquid or solid scent-boosters (PEG / urea or salts), a dryer sheet, an ironing water, a paper, a bleach, a carpet cleaners, curtain-care products, a shampoo, a coloring preparation, a color care product, a hair shaping product, a dental care product, a disinfectant, an intimate care product, a hair spray, a hair conditioning product, a vanishing cream, a deodorant or antiperspirant, hair remover, tanning or
15 sun product, nail products, skin cleansing, a makeup, a perfumed soap, shower or bath mousse, oil or gel, or a foot/hand care products, a hygiene product, an air freshener, a "ready to use" powdered air freshener, a mold remover, furnisher care, wipe, a dish detergent or hard-surface detergent, a leather care product, a car care product.

20 In a particular embodiment, the perfumed consumer product is preferably selected from the group consisting of personal care composition, home care composition or fabric care composition, most preferably in form of antiperspirants, hair care products, such as shampoo or hair-conditioner, body care products such as a shower gel, oral care products, laundry care products, preferably a detergent or a
25 fabric softener.

Another object of the invention is a consumer product comprising:

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

Personal care active bases 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 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 coloring preparation or a hair spray),
5 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);

Another object of the invention is a consumer product comprising:

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

wherein the consumer product is in the form of a home care or a fabric care composition.

- 15 Home care or fabric care active bases 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 components on the basis of
20 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
25 effect desired in each product.

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

- For solid consumer product mention below, by “active base”, it should be
30 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).

The home or fabric care composition is preferably chosen in the group consisting of fabric softener, liquid detergent, powder detergent, liquid scent booster and solid scent booster.

5 **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,
- microcapsules or 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.

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 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,

- microcapsules or 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.

5

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,

- microcapsules or 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.

5

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,
- microcapsules or 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,

- microcapsules or 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.

5

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,
- 10 – 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,
- 15 – amine oxides and combinations thereof;
- 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
- 20 – than 10 and mixtures thereof, and
- microcapsules or 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,
- optionally free perfume oil.

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Hair coloration

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

- an oxidizing phase comprising an oxidizing agent and an alkaline phase
- 30 – 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,

- microcapsules or 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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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 or a microcapsule 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.

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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.

Example 1

Preparation of microcapsules with polyol in water and HCCP in oil phase

General procedure: A solution of modified starch HI-CAP (Origin: Ingredion, USA) at 2wt% in water (35 g) was added in a beaker. Guanidine carbonate and a polyol were added to the solution to afford an aqueous phase. A solution of perfume oil (see Table 1 - 15 g) and HCCP (hexachlorocyclophosphazene, Origin: Sigma-Aldrich, Switzerland) was prepared in a second beaker to afford an oil phase. The latter was added to the aqueous phase and the reaction mixture was sheared with an Ultra Turrax at 20,000 rpm for two minutes to obtain an emulsion under basic conditions (pH = 9). The emulsion was finally cured to obtain microcapsules.

Table 1: Perfume oil A formulation

Ingredient	% in oil
Ethyl 2-methyl-pentanoate	3.20%
Eucalyptol	7.80%
2,4-Dimethyl-3-cyclohexene-1-carbaldehyde	0.75%
Aldehyde C ₁₀	0.75%
Citronellyl Nitrile	4.30%
Isobornyl acetate	3.00%
2-tert-butyl-1-cyclohexyl acetate	9.80%
Citronellyl Acetate	1.30%
2-Methylundecanal	3.00%
Diphenyloxide	0.80%
Aldehyde C ₁₂	1.30%
Dicyclopentadiene acetate	9.85%
Ionone beta	3.30%
Undecalactone gamma	18.75%
Hexyl Salicylate	15.9%
Benzyl Salicylate	16.2%

Table 2: Capsule A compositions

Capsules	Guanidine carbonate (g)	Polyol	Alcohol (g)	HCC P (g)	Curing conditions
A1	3.15	Phloroglucinol	1.43	2	40°C, 1h + 60°C, 3h
A2	2.18		1	1.4	40°C, 1h + 60°C, 3h
A3	1.09		0.5	0.7	40°C, 1h + 50°C, 4h
A4	1.09		0.5	0.7	40°C, 1h + 60°C, 3h
A5	1.09		1.5	2.1	40°C, 1h + 60°C, 3h
A6	2.76	Erythritol	1.1	2.1	40°C, 1h + 60°C, 6h
A7	3.48		1.38	2.1	40°C, 1h + 60°C, 6h
A8	3.49		1.65	2.1	40°C, 1h + 60°C, 6h
A9	3.89*		1.1	2.1	40°C, 0.5h + 60°C, 7h
A10	10*		2.2	4.2	40°C, 0.5h + 60°C, 4h
A11	2.76	Pentaerythritol	1.3	2.1	40°C, 1h + 60°C, 6h

*from an aq. sln at 20 wt%

Example 2

Preparation of microcapsules with polyol and HCCP in oil phase

General procedure: A solution of HI-CAP at 2wt% in water (35 g) was added in a beaker. Guanidine carbonate was added to the solution to afford an aqueous phase. A solution of perfume oil (15 g), HCCP and a polyol was prepared in a second beaker to afford an oil phase. The latter was added to the aqueous phase and the reaction mixture was sheared with an Ultra Turrax at 20,000 rpm for two minutes to obtain an emulsion under basic conditions (pH = 9). The emulsion was finally cured to obtain microcapsules.

Table 3: Capsule B compositions

Capsules	Guanidine carbonate (g)	Polyol	Alcohol (g)	HCC P (g)	Curing conditions
B1	1.09	Phloroglucinol	0.5	0.7	40°C, 1h + 60°C, 3h
B2	3.08	Erythritol	1.1	2.1	40°C, 0.5h + 60°C, 7h
B3	3.08	Pentaerythritol	1.3	2.1	40°C, 0.5h + 60°C, 7h

Example 3

Preparation of microcapsules with a part of polyol in water and another in oil phase and HCCP in oil phase

General procedure: A solution of HI-CAP at 2wt% in water (35 g) was added in a beaker. Guanidine carbonate was added to the solution to afford an aqueous phase. A solution of perfume oil (15 g), HCCP and polyol was prepared in a second beaker to afford an oil phase. The latter was added to the aqueous phase and the reaction mixture was sheared with an Ultra Turrax at 20,000 rpm for two minutes to obtain an

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emulsion under basic conditions (pH = 9). The emulsion was finally cured to obtain microcapsules.

Table 4: Capsule C compositions

Capsules	Guanidine carbonate (g)	Alcohol	Alcohol in water (g)	Alcohol in oil (g)	HCC P (g)	Curing conditions
C1	3.27	Erythritol	0.825	0.275	2.1	40°C, 0.5h + 60°C, 7h
C2			0.55	0.55	2.1	
C3			0.275	0.825	2.1	
C4		Pentaerythritol	0.975	0.325	2.1	
C5			0.65	0.65	2.1	
C6			0.325	0.975	2.1	

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Example 4

Preparation of microcapsules with HCCP, modified e-poly(L-lysine) and potato protein

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Example 4-1 (Capsules D1 - NICP2712): e-Poly(L-lysine) (1 g), 2,2-dimethoxyethanal (60%, 0.22 g), glyoxylic acid (40%, 0.23 g), and n-octanal (0.04 g) were dissolved in water (10 g). The pH value was adjusted above 10 with a solution of sodium hydroxide in water at 30%. The reaction mixture was added to a solution of potato protein (Solanic 200, origin: Avebe, NL, 0.8 g) in water (79.2 g) and the pH value was measured at 10.1. HCCP (0.21 g) was dissolved in perfume oil (20 g). The organic phase was added and dispersed with the help of Ultra-turrax mixer (S25N) at a speed of 18,000 rpm for 1 minute (pH = 9.59). The pH value was reduced to 6.1 with acetic acid. The emulsion was transferred to a 250 mL reactor and stirred with an anchor.

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The temperature increased to 80°C over the period of 1 h and was maintained at 80°C for 3 h. The dispersion was cooled down to room temperature to afford a slurry.

- Example 4-2 (Capsules D2 - NICP2776): The capsules were prepared according to the protocol of example 4-1 with an aqueous phase comprising e-Poly(L-lysine) (2 g), 2,2-dimethoxyethanal (60%, 0.45 g), glyoxylic acid (40%, 0.48 g), n-octanal (0.09 g) in water (10 g) and a solution of potato protein (1.6 g) in water (78.4 g) and an oil phase comprising HCCP (0.42 g) and perfume oil (20 g).

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Example 5

Preparation of microcapsules with modified HCCP, modified e-poly(L-lysine) and potato protein

- 15 *Preparation of tri(4-formylphenoxy)-trichlorocyclophosphazenes*: In a 100 mL round bottom flask under inert atmosphere, HCCP (8 g) and anhydrous potassium carbonate (10 g) were added in anhydrous THF (30 mL). A solution of 4-hydroxybenzaldehyde (8.5 g) in anhydrous THF (30 mL) was added and the reaction mixture was stirred at 60°C for 15 h. The reaction mixture was filtered and the solvent was distilled off under vacuum to afford the product as an oil. The reaction product was characterized by NMR as a mixture of isomers. The product was used as a raw material.

- Capsules of example 5-2 (Capsules E1): e-Poly(L-lysine) (2 g), 2,2-dimethoxyethanal (60%, 0.45 g), glyoxylic acid (40%, 0.47 g), and n-octanal (0.09 g) were dissolved in water (10 g). The pH value was adjusted to 12.2 with a solution of sodium hydroxide in water at 30%. The reaction mixture was added to a solution of Solanic 200 (1.6 g) in water (78.4 g) and the pH value was measured at 9.9. Tri(4-formylphenoxy)-trichlorocyclophosphazenes (1.07 g) were dissolved in perfume oil (20 g). The organic phase was added and dispersed with the help of Ultra-turrax mixer (S25N) at a speed of 18,000 rpm for 1 minute (pH = 9.7). The emulsion was stirred at room temperature for 30 minutes. The pH value was reduced to 5.5 with acetic acid. The emulsion was transferred to a 250 mL Schmizo reactor and stirred with an anchor. The temperature increased to 80°C over the period of 1 h and was maintained at 80°C for 3 h (pH = 5.3). The dispersion was cooled down to room temperature to afford a slurry.

Example 6

Preparation of microcapsules with amines in water and potato protein, HCCP in oil phase

5 Gum arabic (0.7 g) and L-lysine (1 g) were dissolved in water (70 g). Solanic 200 (0.5 g) was dispersed in a solution of HCCP (1 g) in perfume oil (10 g). This oil phase was added to the aqueous phase and the reaction mixture was sheared with an Ultra Turrax at 20,000 rpm for two minutes to obtain an emulsion. The emulsion was
10 transferred to a 250 mL Schmizo reactor and stirred with an anchor. A solution of amine in water was then added dropwise to the emulsion and the mixture was cured at 80°C for 6 h to obtain microcapsules. The dispersion was cooled down to room temperature to afford a slurry.

15 **Table 5:** Capsule F compositions

Capsules	Amine	Amine (g)
F1	Cystamine dihydrochloride	0.31
F2	Ethylene diamine	0.13
F3	Diethylene triamine	0.22
F4	Xylylene diamine	0.29

Example 7

Preparation of 3,9-dichloro-2,4,8,10-tetraoxa-3λ5,9λ5-diphosphaspiro[5.5]undecane
3,9-dioxide

20 Pentaerythritol (5.1 g, 36.7 mmol) was dissolved in acetonitrile (50 mL) in a four-necked round bottom flask equipped with a thermometer, a mechanical stirrer, a condenser and an addition funnel under nitrogen. Phosphorus oxychloride (10.5 mL,

112.5 mmol) was added. The temperature was increased to 110°C until the reactants were fully dissolved. The reaction was stirred for 24 h. The reaction product was recovered by filtration and washed with dichloromethane to afford a white solid (yield = 70%).

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Example 7A

Preparation of copolymer with 4,4',4'',4''',4''',4''''-((1,3,5,2I⁵,4I⁵,6I⁵-
 triazatriphosphinine-2,2,4,4,6,6 hexayl)hexakis(oxy))hexabenzaldehyde (named as
 hexa-(4-aldehyde-phenoxy)-cyclo-triphosphazene = HACP) and 6,6-dichloro-
 1,3,5,2I⁵,4I⁵,6I⁵-triazatriphosphinine-2,2,4,4-tetraamine (named as 2,2,4,4-
 tetraamino-6,6-dichlorocyclotriphosphazene = TCPA)

Preparation of HACP : 4-hydroxybenzaldehyde (3.75 g, 30.7 mmol), anhydrous K₂CO₃ (4.20 g, deacidification reagent) were dissolved in anhydrous THF (15 mL) in a 25 mL
 three-necked round-bottom flask equipped with a condenser and a stirring bar under
 N₂. Then a solution of HCCP (1.5 g, 4.27 mmol) in anhydrous THF (3 mL) was added
 dropwise at 0°C and the mixture was heated at 60°C for 7 h. Then the solution was
 poured in water, a white solid precipitated. The product was washed with excess of
 water and then isopropanol. Finally, the white product was dried under vacuum at
 room temperature overnight. 3.32 g of white powder was obtained. Yield: 89.3%. ³¹P
 NMR, ppm (202.5 MHz, DMSO-d₆): -8.08. ¹³C NMR, ppm (125.8 MHz, DMSO-d₆):
 121.01 (aryl. CH), 131.42 (aryl. CH), 133.54 (aryl. sat. C), 153.57 (C-OH), 191.59
 (C=O).

Preparation of TCPA: Ammonia solution 0.5 M in dioxane (77 mL, 38.5 mmol) was
 placed in a 250 mL three-necked round-bottom flask equipped with a condenser and
 a stirring bar, the setup was purged with N₂. HCCP (1.04 g, 3 mmol) was dissolved in
 acetone (30 mL) and introduced dropwise with a dropping funnel for 30 minutes. The
 reaction mixture was stirred for 2 h to form white precipitate. Then the precipitate was
 filtered and washed with excess of hot acetonitrile. The filtrate was concentrated, the
 solid obtained from the filtrate was precipitated with diethyl ether. The product was
 dried on air overnight to afford a white powder (m = 0.59 g). Yield: 73%. ¹H NMR, ppm
 (500 MHz, DMSO-d₆): 3.87 (s, NH₂); ³¹P NMR (202.5 MHz, DMSO-d₆), δ (ppm): 19.50
 (t, J = 52 Hz, 1P, P-Cl), 12.45 (d, J = 52 Hz, 2P, P-NH₂). HRMS, m/z: for [M+H]⁺ calc.

269.94, found 269.95. IR: P=N (1347, 1216, 1159 cm⁻¹), P-N (1405, 944 cm⁻¹), H-N-H (1563 cm⁻¹), N-H (797 cm⁻¹) and P-Cl (608 cm⁻¹).

Preparation of HACP-TCPA copolymer: HACP (0.086 g, 0.1 mmol) and TCPA (0.162 g, 0.6 mmol) were mixed and dissolved in DMSO (5 mL) in a 10 mL screw-cap vial equipped with a stirring bar. Formic acid (0.3 mL, 80 mmol) was added, and the vial was placed in water bath at 80°C for 5 h. Then the vial was left stirring at rt overnight. Precipitate was filtered and washed with acetone, ethanol, and water, and dried on air overnight to afford a red solid (0.1 g). FTIR, cm⁻¹: 1700 (C=O), 1601 (N=C), 1047 (C-OH). ³¹P solid-state NMR, ppm (162 MHz): 8.8. ¹³C solid-state NMR, ppm (100.6 MHz): 194, 170, 153, 149, 130, 62.

The analysis showed the presence of copolymer indicating the potential of both monomers to prepare microcapsules.

Example 8

Capsule shell biodegradability

Polymer shell of microcapsules from example 5 was purified according to the protocol published by Gasparini *et al.* in *Molecules* (2020), 25(3), 718. The polymer was characterized by solid-state NMR and then tested under the OECD301F conditions to afford a biodegradation greater than 60% after 28 days.

Example 9

Fabric softener composition

A sufficient amount of exemplified microcapsules is weighed and mixed in a fabric softener composition to add the equivalent of 0.116% perfume.

Table 6: Fabric Softener composition

Product	Wt %
Stepantex VL 90A	8.88
Calcium Chloride Sol. 10%	0.36
Proxel GXL	0.04
Perfume	1.00

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Water	89.72
TOTAL	100.00

Example 10

Powder detergent composition

A sufficient amount of exemplified microcapsules is weighed and mixed in a powder
5 detergent composition to add the equivalent of 0.2% perfume.

Table 7: Powder detergent composition

Ingredients	Part
Anionic (Linear Alkyl Benzene Sulphonates)	20%
Nonionics (Alcohol Ethoxylates (5-9 ethylene oxide)	6%
Builders (zeolites, sodium carbonate)	25%
Silicates	6%
Sodium Sulphate	35%
Others (Enzymes, Polymers, Bleach)	7.5%
Spray-dried granule powder A-E	0.5%

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Example 11

Spray-dried microcapsules preparation

15 Emulsions A-E having the following ingredients are prepared.

Table 8: Composition of Emulsions A-E and composition of granulated powder A-E after spray-drying

Ingredients	Emulsion A	Emulsion B	Emulsion C	Emulsion D	Emulsion E
Modified starch ¹⁾	2.6%	2.6%	2.6%	12.5%	2%
Maltodextrin ²⁾	26.8%	22.8%	19.3%	0%	19.1%
Maltose ³⁾	0%	0%	0%	7.9%	0%
Citric Acid	0%	0%	0%	1%	0%
Tripotassium Citrate	0%	0%	0%	1.9%	0%
Exemplified Microcapsules	12.0%	24%	37.0%	8.9%	56.2%
Silica ⁴⁾	1.1%	1.1%	1.1%	0%	0%
Free Perfume B ⁵⁾	0%	0%	0%	11%	0%
Water	57.6%	49.6%	40.1%	56.9%	22.7%
	Granule A	Granule B	Granule C	Granule D	Granule E
Modified starch ¹⁾	7.5%	7.4%	7.2%	31.6%	4.9%
Maltodextrin ²⁾	77.4%	65.5%	53.8%	0%	44.7%
Maltose	0%	0%	0%	20.9%	
Citric Acid	0%	0%	0%	2.6%	0%
Tripotassium citrate	0%	0%	0%	4.9%	0%
Encapsulated perfume B	0%	0%	0%	28.1%	0%
Exemplified microcapsules	12.%	24.1%	36.1%	9.8%	48.4%
Silica	3.0	3.0%	2.9%	2.0%	2%
Fragrance loading in	10.1%	20.1%	30%	35.8%	40.2%

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powder after spray-drying					
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- 1) CapsulTM, Ingredion
- 2) Maltodextrin 10DE origin: Roquette
- 3) Maltose, Lehmann & Voss
- 5 4) Silica, Evonik
- 5) see table 8a

Table 8a: Composition of Perfume B

Component	%
ACÉTATE DE 4-(1,1-DIMÉTHYLÉTHYL)-1-CYCLOHEXYLE ¹⁾	14.50
LINALOL BJ	10.50
LILIAL® ²⁾	10.00
ISO E SUPER ³⁾	10.00
CITRONELLYL NITRILE	9.00
DIPHENYLOXYDE	6.50
ISOBORNYL ACETATE	6.00
BETA IONONE	6.00
TRICYCLO[5.2.1.0~2,6~]DEC-3-EN-8-YL ACETATE (A) + TRICYCLO[5.2.1.0~2,6~]DEC-4-EN-8-YL ACETATE (B) ⁴⁾	5.50
ETHER MT	4.00
HEDIONE® ⁵⁾	4.00
GERANIOL 60	3.00
CITRAL	2.50
ALDEHYDE C 10	2.50
ALLYL HEPTANOATE	2.50
ETHYL METHYL-2-BUTYRATE	1.50
GERANYL ACETATE	1.00
2,4-DIMETHYL-3-CYCLOHEXENE-1-CARBALDEHYDE ⁶⁾	1.00

- 1) Firmenich SA, Switzerland
- 2) 3-(4-tert-butylphenyl)-2-methylpropanal, Givaudan SA, Vernier, Switzerland
- 3)1-(octahydro-2,3,8,8-tetramethyl-2-naphtalenyl)-1-ethanone, International Flavors & Fragrances, USA
- 5 4) Firmenich SA, Switzerland
- 5) Methyl dihydrojasmonate, Firmenich SA, Switzerland
- 6) Firmenich SA, Switzerland

Components for the polymeric matrix (Maltodextrin and capsulTM, or capsulTM , citric acid and tripotassium citrate) are added in water at 45-50°C until complete dissolution.

For emulsion D, free perfume B is added to the aqueous phase.

Microcapsules slurry is added to the obtained mixture. Then, the resulting mixture is then mixed gently at 25°C (room temperature).

Granulated powder A-E are prepared by spray-drying Emulsion A-E using a Sodeva

15 Spray Dryer (Origin France), with an air inlet temperature set to 215°C and a throughput set to 500 ml per hour. The air outlet temperature is of 105°C. The emulsion before atomization is at ambient temperature.

Example 12

20 Liquid scent booster composition

A sufficient amount of exemplified microcapsules is weighed and mixed in a liquid scent booster to add the equivalent of 0.2% perfume.

25 **Table 9:** Liquid scent booster composition

Ingredients	Amount (% wt)					
	1	2	3	4	5	6
Water	71.20%	89.5%	78.8%	79.4%	70%	70%
Propylene glycol	20.30%	-	-	-	20%	20%
Polyethylene glycol ethers of decyl alcohol ¹⁾	4.00%	6%				

Polyethylene glycol ether of Lauryl Alcohol ²⁾					4.00%	4.00%
alkyl polyglucoside C8-C10 ³⁾			8.30%	7.7%		
Deceth-3 ¹⁾	1.50%					
Lauryl lactate				1%		
Lauric acid		1.5%	1.60%			
Glyceryl Caprylate					3.00%	3.00%
Fragrance	3.00%	3.0%	3.00%	3.00%	3.00%	0%

1) Deceth-8; trademark and origin : KLK Oleo

2) Laureth-9; ; trademark and origin

3) Plantacare 2000UP; trademark and origin : BASF

5 Different ringing gel compositions are prepared (compositions 1-6) according to the following protocol.

In a first step, the aqueous phase (water), the solvent (propylene glycol) if present and surfactants are mixed together at room temperature under agitation with magnetic stirrer at 300 rpm for 5 min.

10 In a second step, the linker is dissolved in the hydrophobic active ingredient (fragrance) at room temperature under agitation with magnetic stirrer at 300 rpm. The resulting mixture is mixed for 5 min.

15 Then, the aqueous phase and the oil phase are mixed together at room temperature for 5 min leading to the formation of a transparent or opalescent ringing gel.

Example 13

Liquid detergent composition

20 A sufficient amount of exemplified microcapsules is weighed and mixed in a liquid detergent to add the equivalent of 0.2% perfume.

Table 10: Liquid detergent composition

Ingredients	Concentration [wt%]
Sodium C ₁₄₋₁₇ Alkyl Sec Sulfonate ¹⁾	7
Fatty acids, C ₁₂₋₁₈ and C ₁₈ -unsaturated ²⁾	7.5
C _{12/14} fatty alcohol polyglycol ether with 7 mol EO ³⁾	17
Triethanolamine	7.5
Propylene Glycol	11
Citric acid	6.5
Potassium Hydroxyde	9.5
Properase L ⁴⁾	0.2
Puradax EG L ⁴⁾	0.2
Purastar ST L ⁴⁾	0.2
Acrylates/Steareth-20 Methacrylate structuring Crosspolymer ⁵⁾	6
Deionized Water	27.4

- 1) Hostapur SAS 60; Origin: Clariant
- 2) Edenor K 12-18; Origin: Cognis
- 5 3) Genapol LA 070; Origin: Clariant
- 4) Origin: Genencor International
- 5) Aculyn 88; Origin: Dow Chemical

A sufficient amount of exemplified microcapsules is weighed and mixed in a unit dose formulation to add the equivalent of 0.2% perfume.
The unit dose formulation can be contained in a PVOH (polyvinyl alcohol) film.

5 **Table 11:** Unit dose composition

Ingredients	Concentration [wt%]
C12 - C14 alkyl poly ethoxylate	15
C12 - C14 alkyl poly ethoxylate sulfate Mono Ethanol Amine salt	9.5
Linear Alkylbenzene sulfonic acid	17
Citric Acid	0.5
C12 - C18 Fatty Acid	17
Enzymes	1.2
Fluorescent brightener	0.3
1,2 propanediol	12
Glycerol	9
Sodium Hydroxide	1
Mono Ethanol Amine	6
PDMS	2.5
Potassium sulphite	0.2
Water	8.8
Total	100

Example 15

Concentrated All Purpose Cleaner composition

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A sufficient amount of exemplified microcapsules is weighed and mixed in a concentrated all-purpose cleaner composition to add the equivalent of 0.2% perfume.

Table 12: concentrated all-purpose cleaner composition

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Ingredients	Amount (%) wt)	Function
Ethoxylated Alcohol (C9-C11, 8EO) ⁽¹⁾	20	Non-ionic surfactant
Sodium Dodecyl Benzene Sulfonate ⁽²⁾	16	Anionic surfactant
Sodium Cumene Sulfonate ⁽³⁾	8	Hydrotrope
Methyl chloro isothiazolinone Methyl isothiazolinone 3.3:1 ⁽⁴⁾	0.8%	preservative
Water	55.9	solvent

- 1) Neodol 91-8 ®; trademark and origin : Shell Chemical
- 2) Biosoft D-40®; trademark and origin : Stepan Company
- 3) Stepanate SCS®; trademark and origin : Stepan Company
- 4) Kathon CG®; trademark and origin : Dow Chemical Company
- 5 All ingredients are mixed together and then the mixture was diluted with water to 100%.

Example 16

Solid scent booster composition

- 10 The following compositions are prepared.

Table 13: Salt-based solid scent booster compositions

Ingredients	Part
Sodium chloride	95
Spray-dried granule powder A-E	5

- 15 Table 14: Urea-based solid scent booster compositions

Ingredients	Part
Urea (beads)	94
Spray-dried granule powder A-E	8
Bentonite	3
Perfume	3

Example 17

Shampoo composition

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A sufficient amount of exemplified microcapsules is weighed and mixed in a shampoo composition to add the equivalent of 0.2% perfume.

Table 15: Shampoo composition

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

- 1) Ucare Polymer JR-400, Noveon
- 2) Schweizerhall
- 3) Glydant, Lonza
- 4) Texapon NSO IS, Cognis
- 5) Tego Betain F 50, Evonik

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- 6) Amphotensid GB 2009, Zschimmer & Schwarz
- 7) Monomuls 90 L-12, Gruenau
- 8) Nipagin Monosodium, NIPA

Polyquaternium-10 is dispersed in water. The remaining ingredients of phase A are mixed separately by addition of one after the other while mixing well after each adjunction. Then this pre-mix is added to the Polyquaternium-10 dispersion and was
5 mixed for 5 min. Then Phase B and the premixed Phase C (heat to melt Monomuls 90L-12 in Texapon NSO IS) are added. The mixture is mixed well. Then, Phase D and Phase E are added while agitating. The pH was adjusted with citric acid solution till pH: 5.5 – 6.0.

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Example 18

Shampoo composition

A sufficient amount of exemplified microcapsules is weighed and mixed in a shampoo composition to add the equivalent of 0.2% perfume.

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Table 16: Shampoo composition

	Ingredients	Concentration [wt%]
A	Water deionized	45.97
	Tetrasodium EDTA ¹⁾	0.05
	Guar Hydroxypropyltrimonium Chloride ²⁾	0.05
	Polyquaternium-10 ³⁾	0.075
B	NaOH 10% aqueous sol.	0.3
C	Ammonium Lauryl Sulfate ⁴⁾	34
	Ammonium Laureth Sulfate ⁵⁾	9.25
	Cocamidopropyl Betaine ⁶⁾	2
	Dimethicone (&) C12-13 Pareth-4 (&) C12-13 Pareth-23 (&) Salicylic Acid ⁷⁾	2.5
D	Cetyl Alcohol ⁸⁾	1.2

71

	Cocamide MEA ⁹⁾	1.5
	Glycol Distearate ¹⁰⁾	2
E	Methylchloroisothiazolinone & Methylisothiazolinone ¹¹⁾	0.1
	D-Panthenol 75% ¹²⁾	0.1
	Water deionized	0.3
F	Sodium Chloride 25% aqueous sol.	0.6
	TOTAL:	100

1) EDETA B Powder, BASF

2) Jaguar C14 S, Rhodia

3) Ucare Polymer JR-400, Noveon

4) Sulfetal LA B-E, Zschimmer & Schwarz

5) Zetesol LA, Zschimmer & Schwarz

6) Tego Betain F 50, Evonik

7) Xiameter MEM-1691, Dow Corning

8) Lanette 16, BASF

9) Comperlan 100, Cognis

10) Cutina AGS, Cognis

11) Kathon CG, Rohm & Haas

12) D-Panthenol, Roche

- A premix comprising Guar Hydroxypropyltrimonium Chloride and Polyquaternium-10 are added to water and Tetrasodium EDTA while mixing. When the mixture is homogeneous, NaOH is added. Then, Phase C ingredients are added and the mixture was heat to 75 °C. Phase D ingredients are added and mixed till homogeneous. The heating is stopped and temperature of the mixture is decreased to RT. At 45 °C, ingredients of Phase E while mixing final viscosity is adjusted with 25% NaCl solution and pH of 5.5-6 is adjusted with 10% NaOH solution.

10

Example 19Rinse-off hair composition

A sufficient amount of exemplified microcapsules is weighed and mixed in a rinse-off composition to add the equivalent of 0.2% perfume.

Table 17: rinse-off composition

5

	Ingredients	Concentration [wt%]
A	Water deionized	81.8
	Behentrimonium Chloride ¹⁾	2.5
	Hydroxyethylcellulose ²⁾	1.5
B	Cetearyl Alcohol ³⁾	4
	Glyceryl Stearate (and) PEG-100 Stearate ⁴⁾	2
	Behentrimonium Methosulfate (and) Cetyl alcohol (and) Butylene Glycol ⁵⁾	4
	Ethoxy (20) Stearyl Alcohol ⁶⁾	1
C	Amodimethicone (and) Trideceth-12 (and) Cetrimonium Chloride ⁷⁾	3
	Chlorhexidine Digluconate ⁸⁾ 20% aqueous solution	0.2
D	Citric acid 10% aqueous sol. till pH 3.5-4	q.s.
	TOTAL:	100

- 1) Genamin KDMP, Clariant
- 2) Tylose H10 Y G4, Shin Etsu
- 3) Lanette O, BASF
- 4) Arlacel 165, Croda
- 5) Incroquat Behenyl TMS-50-PA- (MH), Croda
- 6) Brij S20, Croda
- 7) Xiameter MEM-949, Dow Corning
- 8) Alfa Aesar

10

Ingredients of Phase A are mixed until an uniform mixture was obtained. Tylose is allowed to completely dissolve. Then the mixture is heated up to 70-75°C. Ingredients of Phase B are combined and melted at 70-75°C. Then ingredients of Phase B are

15

added to Phase A with good agitation and the mixing is continued until cooled down to 60°C. Then, ingredients of Phase C are added while agitating and keeping mixing until the mixture cooled down to 40°C. The pH is adjusted with citric acid solution till pH: 3.5 – 4.0.

5

Example 20

Antiperspirant spray anhydrous composition

A sufficient amount of exemplified microcapsules is weighed and mixed in an antiperspirant spray anhydrous composition to add the equivalent of 0.2% perfume.

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Table 18: antiperspirant spray anhydrous composition

Ingredient	Amount (wt %)
Cyclomethicone ¹⁾	53.51
Isopropyl miristate	9.04
Silica ²⁾	1.03
Quaternium-18-Hectorite ³⁾	3.36
Aluminium Chlorohydrate ⁴⁾	33.06

1) Dow Corning® 345 Fluid; trademark and origin: Dow Corning

15

2) Aerosil® 200 ; trademark and origin : Evonik

3) Bentone® 38; trademark and origin : Elementis Specialities

4) Micro Dry Ultrafine; origin : Reheis

Using a high speed stirrer, Silica and Quaternium-18-Hectorite are added to the Isopropyl miristate and Cyclomethicone mixture. Once completely swollen, Aluminium Chlorohydrate is added portion wise under stirring until the mixture was homogeneous and without lumps. The aerosol cans are filled with 25 % Suspension of the suspension and 75 % of Propane/Butane (2,5 bar).

20

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Example 21

Antiperspirant spray emulsion composition

A sufficient amount of exemplified microcapsules is weighed and mixed in antiperspirant spray emulsion composition to add the equivalent of 0.2% perfume.

Table 19: antiperspirant spray emulsion composition

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Ingredient	Amount (wt %)
Polysorbate 65 ¹⁾ (Part A)	0.95
Polyglyceryl-2 dipolyhydroxystearate ²⁾ (Part A)	1.05
Cetyl PEG/PPG-10/1 Dimethicone ³⁾ (Part A)	2.75
Cyclomethicone ⁴⁾ (Part A)	16.4
Isopropylisostearate ⁵⁾ (Part A)	4.5
Phenoxyethanol ⁶⁾ (Part A)	0.5
Ethylhexylglycerin ⁷⁾ (Part A)	0.2
C12-15 Alkyl Benzoate ⁸⁾ (Part A)	5.65
Silica Silylate ⁹⁾ (Part A)	0.1
Sodium Methylparaben ¹⁰⁾ (Part B)	0.1
Aluminium Chlorohydrate ¹¹⁾ (Part B)	20
Water (Part B)	44.47
Fragrance (Part C)	3.33

- 1) Tween 65; trademark and origin : CRODA
- 2) Dehymuls PGPH; trademark and origin : BASF
- 3) Abil EM-90; trademark and origin : BASF
- 10 4) Dow Corning 345 fluid; trademark and origin : Dow Corning
- 5) Crodamol ipis; trademark and origin : CRODA
- 6) Phenoxyethanol; trademark and origin : LANXESS
- 7) Sensiva sc 50; trademark and origin : KRAFT
- 8) Tegosoft TN; trademark and origin : Evonik
- 15 9) Aerosil R 812; trademark and origin : Evonik
- 10)Nipagin mna; trademark and origin : CLARIANT

11)Locron L; trademark and origin : CLARIANT

The ingredients of Part A and Part B are weighted separately. Ingredients of Part A are heated up to 60°C and ingredients of Part B are heated to 55 °C. Ingredients of Part B are poured small parts while continuous stirring into A. Mixture were stirred well until the room temperature was reached. Then, ingredients of part C are added. The emulsion is mixed and is introduced into the aerosol cans. The propellant is crimped and added.

10 Aerosol filling: 30% Emulsion: 70% Propane / Butane 2,5 bar

Example 22

Deodorant spray composition

15 A sufficient amount of exemplified microcapsules is weighed and mixed in antiperspirant deodorant spray composition to add the equivalent of 0.2% perfume.

Table 20: deodorant spray composition

Ingredient	Amount (wt %)
Ethanol 95 %	90.65
Triclosan ¹⁾	0.26
Isopropyl miristate	9.09

20 1) Irgasan® DP 300; trademark and origin : BASF

All the ingredients according to the sequence of the Table 24 are mixed and dissolved. Then the aerosol cans are filled, crimp and the propellant is added (Aerosol filling: 25 40% active solution 60% Propane / Butane 2.5 bar).

Example 23

Antiperspirant roll-on emulsion composition

A sufficient amount of exemplified microcapsules is weighed and mixed in antiperspirant roll-on emulsion composition to add the equivalent of 0.2% perfume.

5 **Table 21:** antiperspirant roll-on emulsion composition

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

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

10

Part A and B are heated separately to 75°C; Part A is added to part B under stirring and the mixture is homogenized for 10 minutes. Then, the mixture is cooled down 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 down to RT.

15

Example 24

Antiperspirant roll-on composition

20

A sufficient amount of exemplified microcapsules is weighed and mixed in antiperspirant roll-on composition to add the equivalent of 0.2% perfume.

Table 22: antiperspirant roll-on composition

Ingredient	QUANTITY
Water (Part A)	45
Aluminum Chlorohydrate 50% aqueous solution ¹⁾ (Part B)	20
Alcohol Denat. (Ethanol 96%) (Part B)	30
Ceteareth-12 ²⁾ (Part C)	2
Ceteareth-30 ³⁾ (Part C)	2
Fragrance (Part D)	1

- 1) LOCRON L; origin: CLARIANT
- 2) EUMULGIN B-1; origin : BASF
- 3) EUMULGIN B-3; origin : BASF

5

The ingredients of part B are mixed in the vessel then ingredient of part A is added. Then dissolved part C in part A and B. With perfume, 1 part of Cremophor RH40 for 1 part of perfume is added while mixing well

10

Example 25

Antiperspirant roll-on composition

A sufficient amount of exemplified microcapsules is weighed and mixed in antiperspirant roll-on emulsion composition to add the equivalent of 0.2% perfume.

15

Table 23: antiperspirant roll-on emulsion composition

Ingredient	Amount (wt %)
Water (Part A)	50.51
Hydroxyethylcellulose ¹⁾ (Part A)	0.71
Ethanol 95 % (Part B)	40.40
1,2-Propylene Glycol (Part B)	5.05
Triclosan ²⁾ (Part B)	0.30
PEG-40 Hydrogenated castor oil ³⁾ (Part C)	3.03

- 1) Natrosol® 250 H; trademark and origin: Ashland
- 2) Irgasan® DP 300; trademark and origin : BASF
- 3) Cremophor® RH 40; trademark and origin : BASF
- 5 Part A is prepared by sprinkling little by little the Hydroxyethylcellulose in the water whilst rapidly stirring with the turbine. Stirring is continued until the Hydroxyethylcellulose is entirely swollen and giving a limpid gel. Then, Part B is poured little by little in Part A whilst continuing stirring until the whole is homogeneous. Part C is added.
- 10

Example 26

Deodorant pump without alcohol formulation

- 15 A sufficient amount of exemplified microcapsules is weighed and mixed in the following composition to add the equivalent of 0.2% perfume.

Table 24: deodorant composition

Ingredients	Amount (wt %)
C12-15 Alkyl Lactate ¹⁾	5
Dimethicone ²⁾	91.6
Cetyl Lactate ³⁾	1
Octyldodecanol ⁴⁾	0.8
Triclosan ⁵⁾	0.1
PERFUME	1.5

- 20 1) Ceraphyl 41; trademark and origin ASHLAND
- 2) DOW CORNING 200 FLUID 0.65cs; trademark and origin DOW CORNING CORPORATION
- 3) Ceraphyl 28; trademark and origin ASHLAND
- 4) Eutanol G; trademark and origin BASF
- 25 5) Irgasan® DP 300; trademark and origin : BASF

All the ingredients are mixed according to the sequence of the table and the mixture is heated slightly to dissolve the Cetyl Lactate.

5

Example 27

Deodorant pump with alcohol formulation

A sufficient amount of exemplified microcapsules is weighed and mixed in the following composition to add the equivalent of 0.2% perfume.

10

Table 25: deodorant composition

Ingredients	Amount (wt %)
Ethyl Alcohol (Part A)	60
PEG-6 Caprylic/Capric Glycerides ¹⁾ (Part A)	2
Water (Part A)	35.6
PEG-40 Hydrogenated Castor Oil ²⁾ (Part B)	0.4
PERFUME (Part B)	2

- 1) Softigen 767; trademark and origin CRODA
- 2) Cremophor[®] RH 40; trademark and origin : BASF

15 Ingredients from Part B are mixed together. Ingredients of Part A are dissolved according to the sequence of the Table and are poured into part B.

Example 28

Talc formulation

20

A sufficient amount of granules A-E is weighed and mixed in introduced in a standard talc base: 100% talc, very slight characteristic odor, white powder, origin: LUZENAC to add the equivalent of 0.2% perfume.

25

Example 29

Shower-gel Reference

A sufficient amount of exemplified microcapsules is weighed and mixed in the following composition to add the equivalent of 0.2% perfume.

5

Table 26: shower gel composition

Ingredients	Amount (% wt)	Function
WATER deionized	49.35	Solvent
Tetrasodium EDTA ¹⁾	0.05	Chelating agent
Acrylates Copolymer ²⁾	6	Thickener
Sodium C12-C15 Pareth Sulfate ³⁾	35	Surfactant
Sodium Hydroxide 20% aqueous solution	1	pH adjuster
Cocamidopropyl Betaine ⁴⁾	8	Surfactant
Methylchloroisothiazolinone and Methylisothiazolinone ⁵⁾	0.1	Preservative
Citric Acid (40%)	0.5	pH adjuster

- 1) EDETA B POWDER; trademark and origin: BASF
- 2) CARBOPOL AQUA SF-1 POLYMER; trademark and origin: NOVEON
- 3) ZETESOL AO 328 U; trademark and origin: ZSCHIMMER & SCHWARZ
- 4) TEGO-BETAIN F 50; trademark and origin: GOLDSCHMIDT
- 5) KATHON CG; trademark and origin: ROHM & HASS

10

Ingredients are mixed, pH is adjusted to 6-6.3 (Viscosity: 4500cPo +/-1500cPo (Brookfield RV / Spindle#4 / 20RPM)).

15

Example 30

Shower-gel composition

A sufficient amount of exemplified microcapsules is weighed and mixed in the following composition to add the equivalent of 0.2% perfume.

20

Table 27: shower gel composition

Ingredients	Amount (% wt)	Function
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81

WATER deionized	52.40	Solvent
Tetrasodium EDTA ¹⁾	0.10	Chelating agent
Sodium Benzoate	0.50	Preservative
Propylene Glycol	2.00	Solvent
Sodium C12-C15 Pareth Sulfate ²⁾	35.00	Surfactant
Cocamidopropyl Betaine ³⁾	8.00	Surfactant
Polyquaternium-7 ⁴⁾	0.20	Conditioning agent
Citric Acid (40%)	1.00	pH adjuster
Sodium Chloride	0.80	Viscosity adjuster

- 1) EDETA B POWDER; trademark and origin: BASF
- 2) ZETESOL AO 328 U; trademark and origin: ZSCHIMMER & SCHWARZ
- 3) TEGO-BETAIN F 50; trademark and origin: GOLDSCHMIDT
- 4) MERQUAT 550; trademark and origin: LUBRIZOL

5

Ingredients are mixed, pH is adjusted to 4.5 (Viscosity: 3000cPo +/-1500cPo (Brookfield RV / Spindle#4 / 20RPM)).

10

Example 31
Shower-gel composition

A sufficient amount of exemplified microcapsules is weighed and mixed in the following composition to add the equivalent of 0.2% perfume.

15

Table 28: shower gel composition

Ingredients	Amount (% wt)	Function
WATER deionized	50.95	Solvent
Tetrasodium EDTA ¹⁾	0.05	Chelating agent
Sodium Benzoate	0.5	Preservative
Glycerin 86%	3.5	Solvent
Sodium Laureth Sulfate ²⁾	27	Surfactant

Polyquaternium-7 ³⁾	1	Conditioning Agent
Coco-Betaine ⁴⁾	6	Surfactant
PEG-120 Methyl Glucose trioleate ⁵⁾	1	Thickener
Citric Acid (40%)	1	pH adjuster
Glycol Distearate & Laureth-4 & Cocamidopropyl Betaine ⁶⁾	3	Pearlizing agent
Sodium Chloride 20%	5	Viscosity adjuster
PEG-40 Hydrogenated Castor Oil ⁷⁾	1	Viscosity adjuster

- 1) EDETA B POWDER; trademark and origin: BASF
- 2) Texapon NSO IS; trademark and origin: COGNIS
- 3) MERQUAT 550; trademark and origin: LUBRIZOL
- 4) DEHYTON AB-30; trademark and origin: COGNIS
- 5) GLUCAMATE LT; trademark and origin: LUBRIZOL
- 6) EUPERLAN PK 3000 AM; trademark and origin: COGNIS
- 7) CREMOPHOR RH 40; trademark and origin: BASF

Ingredients are mixed, pH is adjusted to 4.5 (Viscosity: 4000cPo +/-1500cPo
(Brookfield RV / Spindle#4 / 20RPM))

Example 32

Soap bar

A sufficient amount of exemplified microcapsules is weighed and mixed in a soap bar
formulation a concentration of 7.5% w/w.

Table 29: composition of soap formulation

Ingredients	Soap Formulation (%)
Surfactants	

83

Sodium Linear Alkyl Benzene Sulfonate	
Soap	10 to 25
Alpha Olefin Sulfonate (AOS)	
sodium lauryl sulfate	
Builders	
Sodium Carbonate	5 to 15
Sodium Tri Polyphospate	
Zeolite	
Fillers	
Sodium Sulphate	5 to 30
Talc	5 to 30
Dolomite	5 to 50
China Clay	5 to 50
Calcite - Calcium Carbonate	
Sodium Chloride	5 to 20
Structuring Ingredients	
Aluminium Sulphate	0.5 to 5
Alkaline Silicate	1 to 5
Others	
Colour	0.1 to 1
Perfume A	0.1 to 1
Moisture	5 to 15

Example 33

Cosmetic day cream

5

A sufficient amount of exemplified microcapsules is weighed and mixed into a cosmetic skin cream (see composition below) at a concentration of 5%w/w.

Table 30: Cream composition

Phase	Ingredients	Amounts (%)
-------	-------------	-------------

84

A	Ethoxylated Fatty Alcohol Ester ¹⁾	5
	Cetyl alcohol	0.5
	Ceteth-20 (and) Glyceryl Stearate (and) PEG-6 Stearate (and) Steareth-20 ²⁾	4
	Squalan ³⁾	1
	Paraffin oil	2
	Petrolatum	6
B	Water	55.85%
	Hydrated coacervate phase	20
C	Propylene glycol	5
	DMDM Hydantoin (and) Iodopropynyl Butylcarbamate ⁴⁾	0.15
D	Sodium Carbomer	0.2
E	Perfume	0.3

1) ARLATONE 985

2) TEFOSE 2561

3) COSBIOL

5 4) GLYDANT PLUS

Example 34

Hand Dishwash

- 10 A sufficient amount of exemplified microcapsules is weighed and mixed in the following composition to add the equivalent of 0.2% perfume.

Table 31: Hand dishwash composition

Ingredients	Amount (% wt)	Function
Linear alkylbenzene sulfonic acid ⁽¹⁾	20	Anionic surfactant
Diethanolamide ⁽²⁾	3.5	Foam booster
Sodium Hydroxide (50%) ⁽³⁾	3.4	pH Adjuster/neutralizer
Secondary alcohol ethoxolate ⁽⁴⁾	2.5	Non-ionic surfactant
Sodium xylene sulfonate	6.3	Hydrotrope
Water	64.3	Solvent

- 1) Biosoft S-118®; trademark and origin : Stepan Company
- 2) Ninol 40-CO®; trademark and origin : Stepan Company
- 3) Stepanate SXS®; trademark and origin : Stepan Company
- 4) Tergitol 15-S-9®; trademark and origin : Dow Chemical Company

5

Water with sodium hydroxide and diethanolamide are mixed. LAS is added. After the LAS is neutralized, the remaining ingredients are added. The pH was Checked (=7-8) and adjusted if necessary.

10

Example 35
Toothpaste formulation

A sufficient amount of a microcapsule slurry M (prepared according to the protocol disclosed in example 1 except that a menthol flavor is encapsulated) is weighed and mixed in the following composition to add the equivalent of 0.2% flavor.

15

Table 32: Toothpaste formulation

Ingredients	Amount (% wt)
Polyethylene glycol 400	2.0%
Xanthan Gum	0.6%
Sorbitol 70% Solution	50%
Sodium Fluoride	0.220%
Sodium Benzoate	0.2%

Water	15.230%
Hydrated Silica ¹⁾	22.0%
Hydrated Silica ²⁾	7.0%
Titanium Dioxide CI77891	0.5%
Sodium Lauryl Sulfate	1.250%
Flavor	1.2%
TOTAL	100%

- 1) Tixosil 73; trademark and origin :
- 2) Tixosil 43; trademark and origin :

5

Example 36

Dicalcium Phosphate based toothpaste formulation

A sufficient amount of a microcapsule slurry M (prepared according to the protocol disclosed in example 1 except that a menthol flavor is encapsulated) is weighed and
10 mixed in the following composition to add the equivalent of 0.2% flavor.

Table 33: Toothpaste formulation

Ingredients	Amount (% wt)
Sodium carboxymethyl cellulose	1.2%
Flavor	1.2%
DI/Purified Water	Q.S to Final Wt.
Sodium Lauryl Sulfate	1.3%
Glycerine	20.0%
Sodium Saccharin	0.2%
Dicalcium phosphate dihydrate	36.0%
Methylparaben	0.2%
Silica ¹⁾	3.0%
TOTAL	100%

1) Aerosil®200; trademark and origin:

Example 37

Mouthwash alcohol free formulation

5

A sufficient amount of a microcapsule slurry M (prepared according to the protocol disclosed in example 1 except that a menthol flavor is encapsulated) is weighed and mixed in the following composition to add the equivalent of 0.2% flavor.

10 **Table 34:** Mouthwash formulation

Ingredients	Amount (% wt)
Propylene Glycol	10%
Flavor	0.240%
DI/Purified Water	Q.S to Final Wt.
Poloxamer 407 NF	0.240%
Sodium Lauryl Sulfate	0.040%
Sorbitol 70% Solution	10.0%
Sodium Saccharin	0.030%
Glycerine	3.0%
Sodium Benzoate	0.100%
Sucralose	0.020%
Benzoic Acid	0.050%
TOTAL	100%

Example 38

Mouthwash formulation

15

A sufficient amount of a microcapsule slurry M (prepared according to the protocol disclosed in example 1 except that a menthol flavor is encapsulated) is weighed and mixed in the following composition to add the equivalent of 0.2% flavor.

Table 35: Mouthwash formulation

Ingredients	Amount (% wt)
Ethyl Alcohol 190 Proof	15.0%
Flavor	0.240%
DI/Purified Water	Q.S to Final Wt.
Poloxamer 407 NF	0.240%
Sodium Lauryl Sulfate	0.040%
Sorbitol 70% Solution	10.0%
Sodium Saccharin	0.030%
Glycerine	3.0%
Sodium Benzoate	0.100%
Sucralose	0.020%
Benzoic Acid	0.050%
TOTAL	100%

CLAIMS

- 1- A core-shell microcapsule comprising :
- 5 - a core comprising a hydrophobic material, and
- a shell comprising the reaction product between at least one compound having at least two P-Cl functions and a nucleophile.
- 2- The core-shell microcapsule according to claim 1, wherein the compound
- 10 having at least two P-Cl functions is selected from the group consisting of compound with formula $PXCl_3$, where X is O, S, or NH or NR^1 where R^1 is any hydrocarbon group, optionally comprising heteroatoms, such as phosphorus oxy trichloride ($POCl_3$), thiophosphorus trichloride ($PSCl_3$), phosphorimidic trichloride ($PNHCl_3$), N-substituted-phosphorimidic trichloride (PNR^1Cl_3),
- 15 phosphorus trichloride, phosphorus pentachloride, hexachlorocyclophosphazene (HCCP) and its derivatives, R^1OPXCl_2 (with X is O, NH, NR^1 or S), such as alkyl phosphorodichloridate (R^1OPOCl_2) and/or alkyl dichlorothiophosphate (R^1OPSCl_2) (wherein R^1 can be any hydrocarbon moiety optionally with heteroatoms); poly(dichlorophosphazene), 3,9-dichloro-
- 20 2,4,8,10-tetraoxa-3 λ 5,9 λ 5-diphosphaspiro[5.5]undecane 3,9-dioxide, and mixtures thereof.
- 3- The core-shell microcapsule according to claim 2, wherein the compound having at least two P-Cl functions is hexachlorocyclophosphazene (HCCP) or
- 25 its derivatives or its polymers.
- 4- The core-shell microcapsule according to anyone of the preceding claims, wherein the nucleophile is chosen in the group consisting of nitrogen nucleophile, sulfur nucleophile, enol carbon nucleophile, oxygen nucleophile,
- 30 phosphore nucleophile and mixtures thereof.
- 5- The core-shell microcapsule according to claim 4, wherein the nucleophile is chosen in the group consisting of alcohol, amine, thiol and mixtures thereof.

- 6- The core-shell microcapsule according to anyone of the preceding claims, wherein the hydrophobic material comprises a perfume.
- 7- A process for preparing a core-shell microcapsule slurry comprising the steps of:
- 5 a) Optionally, providing at least one compound having at least two P-Cl functions
- b) admixing at least one hydrophobic material with the optional compound obtained in step a) to form an oil phase;
- c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous
- 10 phase C1 into the oil phase to form a two-phases dispersion;
- d) Optionally, dispersing the two-phases dispersion into a continuous phase C2 comprising optionally at least one compound having at least two P-Cl functions to form a multiple dispersion;
- f) curing the dispersion obtained in step c) or d) to form microcapsules in the form
- 15 of a slurry,
- wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d) and/or in step e).
- 8- The process according to claim 7, wherein it comprises the steps of:
- 20 a) Providing at least one compound having at least two P-Cl functions
- b) admixing at least one hydrophobic material with the compound obtained in step a) to form an oil phase;
- c) Dispersing the oil phase into a continuous phase C1 or dispersing a continuous
- phase C1 into the oil phase to form a two-phases dispersion;
- 25 d) curing the dispersion obtained in step c) to form microcapsules in the form of a slurry,
- wherein a nucleophile is added in step a) and/or step b) and/or step c) and/or step d).
- 9- The process according to claim 7, wherein a stabilizer is added in the oil phase
- 30 and/or in the continuous phase C1.

- 10- The process according to claim 9, wherein the stabilizer is chosen in the group consisting of inorganic particles, surfactant, polymeric emulsifier such as polysaccharides, proteins, glycoproteins, and mixtures thereof.
- 5 11- The process according to any one of claims 7 to 10, wherein the compound having at least two P-Cl functions is added in an amount comprised between 0.5 and 10%, preferably between 2 and 6% by weight based on the total weight of the two-phases dispersion or the multiple dispersion.
- 10 12- The process according to any one of claims 7 to 11, wherein the nucleophile is added in an amount comprised between 0.5 and 10%, preferably between 1 and 4% by weight based on the total weight of the two-phases dispersion or the multiple dispersion.
- 15 13- The process according to any one of claims 7 to 12, wherein the multiple dispersion is a two-phases dispersion, preferably an oil-in-water emulsion.
- 14- The process according to any one of claims 7 to 13, wherein a base, preferably chosen in the group consisting of guanidine carbonate, sodium carbonate, 20 potassium carbonate, sodium hydroxide, is added in step c) and/or d).
- 15- A consumer product, preferably in the form of a home-care product or personal care product or fabric care product, comprising the microcapsules as defined in anyone of claims 1 to 6.