

POLYAMIDE-BASED MICROCAPSULES

Technical Field

5 The present invention relates to a new process for the preparation of polyamide-based microcapsules. Polyamide-based microcapsules are 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 release the core payload
15 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 microcapsules.

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

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

There is therefore still a need to provide new microcapsules using more eco-friendly materials, while not compromising on the performance of the microcapsules, in particular in terms of stability in a challenging medium such as a consumer product base, as well as in
30 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 polyamide-based microcapsules and a process for preparing said microcapsules.

Summary of the Invention

It has now been surprisingly found that performing core-shell microcapsules encapsulating hydrophobic material could be obtained by reacting an acyl chloride with at least one amino-compound and a protein in the presence of a cross-linker, preferably an enzymatic cross-linker. The process of the invention therefore provides a solution to the above-mentioned problems as it allows preparing microcapsules with the desired stability in challenging bases.

In a first aspect, the present invention relates to a process for preparing a polyamide-based core-shell microcapsule slurry comprising the following steps:

- a) Dissolving at least one acyl chloride in a hydrophobic material, preferably a perfume to form an oil phase;
- b) Dispersing the oil phase obtained in step a) into a water phase to form an oil-in-water emulsion;
- c) Performing a curing step to form polyamide-based microcapsules in the form of a slurry; wherein at least one amino-compound A is added in the water phase before the formation of the oil- in-water emulsion and/or in the oil-in water emulsion obtained after step b), wherein a protein is added in the oil phase and/or in the water phase, and wherein a cross-linker is added in step b) and/or in step c) and/or after step c).

In a second aspect, the present invention relates to a polyamide-based core-shell microcapsule slurry obtainable by the process according to anyone of the preceding claims.

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

- an oil-based core comprising a hydrophobic material, preferably a perfume, and
 - a polyamide-based shell comprising a reaction product of:
 - an acyl chloride,
 - an amino compound A,
 - a protein, and
 - optionally an amino compound B
- wherein the shell has a rupture stress less than 10 MPa.

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

- a core, preferably an oil-based core, comprising a hydrophobic material, preferably a perfume, and
- a polyamide-based shell comprising a reaction product of:
 - an acyl chloride,
 - 5 • an amino compound A,
 - optionally an amino compound B,
 - a protein,

wherein the shell has a rupture stress less than 10 MPa.

- 10 In a fifth and sixth aspects, the invention relates to perfumed consumer products and flavoured edible products comprising the microcapsules defined above.

Detailed description of the invention

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

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.

- 20 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.

- 25 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 between about 1 and 3000 microns, preferably between 1 and 500 microns) and comprise an external solid polyamide-based shell and an internal continuous oil phase enclosed by the external shell.

- 30 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.

By “**amino-compound**” it should be understood a compound having at least two reactive amine groups.

In the present invention, the terms “acyl chloride” and “acid chloride” are used indifferently.

By “polyamide-based microcapsules”, it means that the microcapsule’s shell comprises a polyamide material. The wording “polyamide-based microcapsules” can also encompass a shell made of a composite comprising a polyamide material and another material, for example a protein.

“Polyamide-based microcapsules” and “polyamide microcapsules” are used indifferently in the present invention.

It has been found that core-shell polyamide-based microcapsules with overall good performance in challenging bases could be obtained when an acyl chloride reacts with at least one amino-compound and a protein in the presence of a cross-linker (for example transglutaminase) during the process.

PROCESS FOR PREPARING A POLYAMIDE-BASED MICROCAPSULE SLURRY

In a first aspect, the present invention relates to a process for preparing a polyamide-based core-shell microcapsule slurry comprising the following steps:

- a) Dissolving at least one acyl chloride in a hydrophobic material, preferably a perfume to form an oil phase;
- b) Dispersing the oil phase obtained in step a) into a water phase to form an oil-in-water emulsion;
- c) Performing a curing step to form polyamide-based microcapsules in the form of a slurry; wherein at least one amino-compound A is added in the water phase before the formation of the oil- in-water emulsion and/or in the oil-in water emulsion obtained after step b), wherein a protein is added in the oil phase and/or in the water phase, and wherein a cross-linker is added in step b) and/or in step c) and/or after step c).

In one step of the process, an oil phase is formed by admixing at least one hydrophobic material with at least one acyl chloride.

Hydrophobic material

The hydrophobic material according to the invention can be “inert” material like solvents or active ingredients. The core is preferably an oil-based core.

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

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

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

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

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

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

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

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

According to a particular embodiment, the hydrophobic material consists of pest control
20 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
25 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
30 improve, enhance or modify the delivery of the perfuming ingredients, such as perfume precursors, modulators, emulsions or dispersions, as well as combinations which impart an additional benefit beyond that of modifying or imparting an odor, such as long-lastingness, blooming, malodor counteraction, antimicrobial effect, microbial stability, pest control.

The nature and type of the perfuming ingredients present in the oil phase do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled person being able to select them on the basis of its general knowledge and according to intended use or application and the desired organoleptic effect. In general terms, these perfuming ingredients
 5 belong to chemical classes as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitriles, terpenoids, nitrogenous or sulfurous heterocyclic compounds and essential oils (for example Thyme oil), and said perfuming co-ingredients can be of natural or synthetic origin. Many of these co-ingredients are in any case listed in reference texts such as the book by S. Arctander, *Perfume and Flavor Chemicals*, 1969, Montclair, New Jersey, USA, or its more
 10 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,
 15 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;
- 20 - Balsamic ingredients: coumarin, ethylvanillin and/or vanillin;
- Citrus ingredients: dihydromyrcenol, citral, orange oil, linalyl acetate, citronellyl nitrile, orange terpenes, limonene, 1-p-menthen-8-yl acetate and/or 1,4(8)-p-menthadiene;
- Floral ingredients: methyl dihydrojasmonate, linalool, citronellol, phenylethanol, 3-(4-tert-butylphenyl)-2-methylpropanal, hexylcinnamic aldehyde, benzyl acetate, benzyl
 25 salicylate, tetrahydro-2-isobutyl-4-methyl-4(2H)-pyranol, beta ionone, methyl 2-(methylamino)benzoate, (E)-3-methyl-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-3-buten-2-one, (1E)-1-(2,6,6-trimethyl-2-cyclohexen-1-yl)-1-penten-3-one, 1-(2,6,6-trimethyl-1,3-cyclohexadien-1-yl)-2-buten-1-one, (2E)-1-(2,6,6-trimethyl-2-cyclohexen-1-yl)-2-buten-1-one, (2E)-1-[2,6,6-trimethyl-3-cyclohexen-1-yl]-2-buten-1-one, (2E)-1-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-buten-1-one, 3-(3,3/1,1-dimethyl-5-indanyl)propanal,
 30 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, verdyol 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;
- 5 - 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;
- 10 - 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;
- 15 - 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,
- 20 oxacyclohexadecan-2-one and/or (1S,1'R)-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxycarbonyl]methyl propanoate;
- 25 - Woody ingredients: 1-[(1R,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-
- 30

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 (bulky materials) and in particular those from one of the following groups:

- Group 1: perfuming ingredients comprising a cyclohexane, cyclohexene, cyclohexanone or cyclohexenone ring substituted with at least one linear or branched 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, (1RS,3RS,4SR)-3-p-mentanyl acetate, (1R,2S,4R)-4,6,6-trimethyl-bicyclo[3,1,1]heptan-2-ol, tetrahydro-4-methyl-2-phenyl-2H-pyran (origin: Firmenich SA, Geneva, Switzerland), cyclohexyl acetate, cyclanol acetate, 1,4-cyclohexane diethyldicarboxylate (origin: Firmenich SA, Geneva, Switzerland), (3RS,3aRS,6SR,7ASR)-perhydro-3,6-dimethyl-benzo[B]furan-2-one (origin: Firmenich SA, Geneva, Switzerland), ((6R)-perhydro-3,6-dimethyl-benzo[B]furan-2-one (origin:

- Firmenich SA, Geneva, Switzerland), 2,4,6-trimethyl-4-phenyl-1,3-dioxane, 2,4,6-trimethyl-3-cyclohexene-1-carbaldehyde;
- Group 2: (E)-3-methyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-4-penten-2-ol (origin: Givaudan SA, Vernier, Switzerland), (1'R,E)-2-ethyl-4-(2',2',3'-trimethyl-3'-cyclopenten-1'-yl)-2-buten-1-ol (origin: Firmenich SA, Geneva, Switzerland), (1'R,E)-3,3-dimethyl-5-(2',2',3'-trimethyl-3'-cyclopenten-1'-yl)-4-penten-2-ol (origin: Firmenich SA, Geneva, Switzerland), 2-heptylcyclopentanone, methyl-cis-3-oxo-2-pentyl-1-cyclopentane acetate (origin: Firmenich SA, Geneva, Switzerland), 2,2,5-trimethyl-5-pentyl-1-cyclopentanone (origin: Firmenich SA, Geneva, Switzerland), 3,3-dimethyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-4-penten-2-ol (origin: Firmenich SA, Geneva, Switzerland), 3-methyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-2-pentanol (origin: Givaudan SA, Vernier, Switzerland);
 - Group 3: damascones, 1-(5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one (origin: Firmenich SA, Geneva, Switzerland), (1'R)-2-[2-(4'-methyl-3'-cyclohexen-1'-yl)propyl]cyclopentanone, alpha-ionone, beta-ionone, damascenone, mixture of 1-(5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one and 1-(3,3-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one (origin: Firmenich SA, Geneva, Switzerland), 1-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-buten-1-one (origin: Firmenich SA, Geneva, Switzerland), (1S,1'R)-[1-(3',3'-Dimethyl-1'-cyclohexyl)ethoxycarbonyl]methyl propanoate (origin: Firmenich SA, Geneva, Switzerland), 2-tert-butyl-1-cyclohexyl acetate (origin: International Flavors and Fragrances, USA), 1-(2,2,3,6-tetramethyl-cyclohexyl)-3-hexanol (origin: Firmenich SA, Geneva, Switzerland), trans-1-(2,2,6-trimethyl-1-cyclohexyl)-3-hexanol (origin: Firmenich SA, Geneva, Switzerland), (E)-3-methyl-4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-3-buten-2-one, terpenyl isobutyrate, 4-(1,1-dimethylethyl)-1-cyclohexyl acetate (origin: Firmenich SA, Geneva, Switzerland), 8-methoxy-1-p-menthene, (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl) ethoxy]-2-methylpropyl propanoate (origin: Firmenich SA, Geneva, Switzerland), para tert-butylcyclohexanone, menthenethiol, 1-methyl-4-(4-methyl-3-pentenyl)-3-cyclohexene-1-carbaldehyde, allyl cyclohexylpropionate, cyclohexyl salicylate, 2-methoxy-4-methylphenyl methyl carbonate, ethyl 2-methoxy-4-methylphenyl carbonate, 4-ethyl-2-methoxyphenyl methyl carbonate;
 - 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-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-ethyldiene-3-oxatricyclo[6.2.1.0(2,7)]undecane, (perhydro-5,5,8A-trimethyl-2-naphthalenyl acetate (origin: Firmenich SA, Geneva, Switzerland), octalynol, (dodecahydro-3a,6,6,9a-tetramethyl-naphtho[2,1-b]furan, origin: Firmenich SA, Geneva, Switzerland), tricyclo[5.2.1.0(2,6)]dec-3-en-8-yl acetate and tricyclo[5.2.1.0(2,6)]dec-4-en-8-yl acetate as well as tricyclo[5.2.1.0(2,6)]dec-3-en-8-yl 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;
- 15 - 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);
- 20 - 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)-cyclopentadecenone, (origin: Firmenich SA, Geneva, Switzerland), 3-methylcyclopentadecanone (origin: Firmenich SA, Geneva, Switzerland), pentadecanolide (origin: Firmenich SA, Geneva, Switzerland), cyclopentadecanone (origin: Firmenich SA, Geneva, Switzerland), 1-ethoxyethoxycyclododecane (origin: Firmenich SA, Geneva, Switzerland), 1,4-dioxacycloheptadecane-5,17-dione, 4,8-cyclododecadien-1-one;
- 25 - Group 7: (+)-2-methyl-3-[4-(2-methyl-2-propanyl)phenyl]propanal (origin: Givaudan SA, Vernier, Switzerland), 2,2,2-trichloro-1-phenylethyl acetate.
- 30

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,
5 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
10 secondary and tertiary alcohols.

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

- 25-100wt%, preferably 25-98% of a perfume oil comprising at least 15wt% of high impact perfume raw materials having a Log T<-4, and
- 0-75wt%, preferably 2-75% of a density balancing material having a density
15 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
20 [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. According to an embodiment, the density balancing material is chosen in the group consisting of benzyl salicylate, benzyl benzoate, cyclohexyl salicylate, benzyl phenylacetate, phenylethyl phenoxyacetate, triacetin,
25 methyl and ethyl salicylate, benzyl cinnamate, and mixtures thereof.

The density of a component is defined as the ratio between its mass and its volume (g/cm³).

Several methods are available to determine the density of a component.

One may refer for example to the ISO 298:1998 method to measure d₂₀ densities of
30 essential oils.

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-dimethylbenzo[b]furan-2-one and (3SR,3aRS,6SR,7ASR)-perhydro-3,6-dimethylbenzo[b]furan-2-one, (+)-1-(5-ethyl-5-methyl-1-cyclohexen-1-yl)-4-penten-1-one, (1'S,3'R)-1-methyl-2-[(1',2',2'-trimethylbicyclo[3.1.0]hex-3'-yl)methyl]cyclopropyl} methanol, (+)-3-mercaptohexyl acetate, (2E)-1-(2,6,6-trimethyl-1,3-cyclohexadien-1-yl)-2-buten-1-one, H-methyl-2h-1,5-benzodioxepin-3(4H)-one, (2E,6Z)-2,6-nonadien-1-ol, (4Z)-4-dodecenal, (+)-4-hydroxy-2,5-dimethyl-3(2H)-furanone, methyl 2,4-dihydroxy-3,6-dimethylbenzoate, 3-methylindole, (+)-perhydro-4alpha,8abeta-dimethyl-4a-naphthalenol, patchoulol, 2-methoxy-4-(1-propenyl)phenol, mixture comprising (+)-5,6-dihydro-4-methyl-2-phenyl-2H-pyran and tetrahydro-4-methylene-2-phenyl-2H-pyran, mixture comprising 4-methylene-2-phenyltetrahydro-2H-pyran and (+)-4-methyl-2-phenyl-3,6-dihydro-2H-pyran, 4-hydroxy-3-methoxybenzaldehyde, nonylenic aldehyde, 2-methoxy-4-propylphenol, 3-methyl-5-phenyl-2-pentenitrile, 1-(spiro[4.5]dec-6/7-en-7-yl)-4-penten-1-one, 2-methoxynaphthalene, (-)-(3aR,5AS,9AS,9BR)-3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-b]furan, 5-nonanolide, (3aR,5AS,9AS,9BR)-3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-b]furan, 7-isopropyl-

2H,4H-1,5-benzodioxepin-3-one, coumarin, 4-methylphenyl isobutyrate, (2E)-1-(2,6,6-trimethyl-1,3-cyclohexadien-1-yl)-2-buten-1-one, beta,2,2,3-tetramethyl-delta-methylene-3-cyclopentene-1-butanol, delta damascone ((2E)-1-[(1R,2SR)-2,6,6-trimethyl-3-cyclohexen-1-yl]-2-buten-1-one), (+)-3,6-dihydro-4,6-dimethyl-2-phenyl-2h-pyran, anisaldehyde, paracresol, 3-ethoxy-4-hydroxybenzaldehyde, methyl 2-aminobenzoate, ethyl methylphenylglycidate, octalactone gamma, ethyl 3-phenyl-2-propenoate, (-)-(2E)-2-ethyl-4-[(1R)-2,2,3-trimethyl-3-cyclopenten-1-yl]-2-buten-1-ol, paracresyl acetate, dodecalactone, tricyclone, (+)-(3R,5Z)-3-methyl-5-cyclopentadecen-1-one, undecalactone, (1R,4R)-8-mercapto-3-p-menthanone, (3S,3AS,6R,7AR)-3,6-dimethylhexahydro-1-benzofuran-2(3H)-one, beta ionone, (+)-6-pentyltetrahydro-2H-pyran-2-one, (3E,5Z)-1,3,5-undecatriene, 10-undecenal, (9E)-9-undecenal (9Z)-9-undecenal, (Z)-4-decenal, (+)-ethyl 2-methylpentanoate, 1,2-diallyldisulfane, 2-tridecenenitrile, 3-tridecenenitrile, , (+)-2-ethyl-4,4-dimethyl-1,3-oxathiane, (+)-(3R,5Z)-3-methyl-5-cyclopentadecen-1-one, 3-(4-tert-butylphenyl)propanal, allyl (cyclohexyloxy)acetate, 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-propanyl)cyclohexanone oxime, 6-hexyltetrahydro-2H-pyran-2-one, (+)-3-(3-isopropyl-1-phenyl)butanal, methyl 2-(3-oxo-2-pentylcyclopentyl)acetate, 1-(2,6,6-trimethyl-1-cyclohex-2-enyl)pent-1-en-3-one, indol, 7-propyl-2H,4H-1,5-benzodioxepin-3-one, ethyl praline, (4-methylphenoxy)acetaldehyde, ethyl tricyclo[5.2.1.0.^{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-naphthalenyl)-1-ethanone, methyl 2-(((1R,2R)-3-oxo-2-pentylcyclopentyl)acetate, (+-)-(E)-4-methyl-3-decen-5-ol, 2,4-dimethyl-3-cyclohexene-1-carbaldehyde, 1,3,3-trimethyl-2-oxabicyclo[2.2.2]octane, tetrahydro-4-methyl-2-(2-methyl-1-propenyl)-2H-pyran, dodecanal, 1-oxa-12/13-cyclohexadecen-2-one, (+-)-3-(4-isopropylphenyl)-2-methylpropanal, aldehyde C11, (+-)-2,6-dimethyl-7-octen-2-ol, allyl 3-cyclohexylpropanoate, (Z)-3-hexenyl acetate, 5-methyl-2-(2-propenyl)cyclohexanone, allyl heptanoate, 2-(2-methyl-2-propenyl)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-propenyl 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-[(1R,6SR)-2,2,6-trimethylcyclohexyl]-3-hexanol, 1,3,3-trimethyl-2-oxabicyclo[2.2.2]octane, 1,3,3-trimethyl-2-oxabicyclo[2.2.2]octane, ethyl hexanoate, undecanal, 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.

According to an embodiment, the perfume formulation comprises:

- 0 to 60 wt.% of a hydrophobic solvent (based on the total weight of the perfume formulation),
- 40 to 100 wt.% of a perfume oil (based on the total weight of the perfume formulation), wherein the perfume oil has at least two, preferably all of the following characteristics:
 - 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,
 - 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 defined previously 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 having a Log T < -4 as defined previously,

optionally, further hydrophobic active ingredients.

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

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

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

The term "Hansen solubility parameter" is understood 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

- 5 i. a vapor pressure of less than 0.0008 Torr at 22°C;
- ii. a clogP of 3.5 and higher, preferably 4.0 and higher and more preferably 4.5
- iii. at least two Hansen solubility parameters selected from a first group consisting 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,
- 10 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
- 15 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.
 - 25

- 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,
- 30 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.

According to a particular embodiment, the hydrophobic material comprises an active ingredient (preferably a perfume) and a hydrophobic solvent such as isopropyl myristate, tryglycerides (e.g. Neobee® MCT oil, vegetable oils such as sunflower oil), D-limonene, silicone oil, mineral oil, benzyl salicylate, benzyl benzoate, cyclohexyl salicylate, benzyl phenylacetate, phenylethyl phenylacetate, triacetin, ethyl citrate, methyl and ethyl salicylate, benzyl cinnamate 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, antiprotozoal and/or antiparasite.

As used herein, a "pest control agent" indicates a substance that serves to repel or attract pests, to decrease, inhibit or promote their growth, development or their activity. Pests refer to any living organism, whether animal, plant or fungus, which is invasive or troublesome to plants 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.

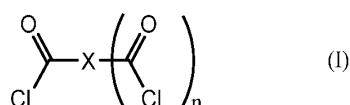
According an embodiment, the hydrophobic material represents between about 10% and 95% by weight, relative to the total weight of the oil phase. According another embodiment, the hydrophobic material represents between about 10% and 80% by weight, relative to the total weight of the oil phase. According another embodiment, the hydrophobic material represents between about 10% and 60% by weight, relative to the total weight of the

oil phase. According another embodiment, the hydrophobic material represents between about 15% and 45% by weight, relative to the total weight of the oil phase.

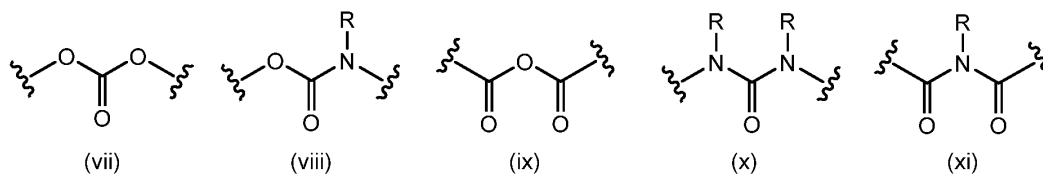
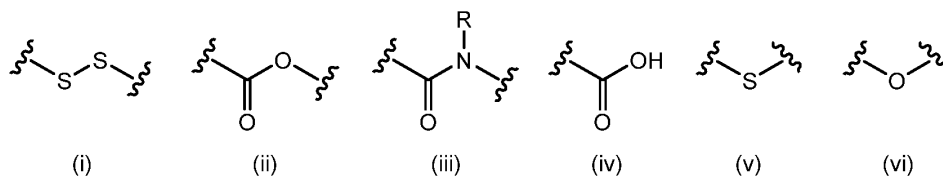
Acyl chloride

5

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



- 10 wherein n is an integer varying between 1 and 8, preferably between 1 and 6, more preferably between 1 and 4, and
wherein X is an (n+1)-valent C₂ to C₄₅ hydrocarbon group optionally comprising at least one group selected from (i) to (xi), particularly from (i) to (vi)



15

wherein R is a hydrogen atom or an alkyl group such as a methyl or an ethyl group, preferably a hydrogen atom.

- 20 It is understood that by "... **hydrocarbon group** ..." it is meant that said group consists of hydrogen and carbon atoms and can be in the form of an aliphatic hydrocarbon, i.e. linear or branched saturated hydrocarbon (e.g. alkyl group), a linear or branched unsaturated hydrocarbon (e.g. alkenyl or alkynyl group), a saturated cyclic hydrocarbon (e.g. cycloalkyl) or an unsaturated cyclic hydrocarbon (e.g. cycloalkenyl or cycloalkynyl), or can be in the form of

an aromatic hydrocarbon, i.e. aryl group, or can also be in the form of a mixture of said type of groups, e.g. a specific group may comprise a linear alkyl, a branched alkenyl (e.g. having one or more carbon-carbon double bonds), a (poly)cycloalkyl and an aryl moiety, unless a specific limitation to only one type is mentioned. Similarly, in all the embodiments of the invention, when a group is mentioned as being in the form of more than one type of topology (e.g. linear, cyclic or branched) and/or being saturated or unsaturated (e.g. alkyl, aromatic or alkenyl), it is also meant a group which may comprise moieties having any one of said topologies or being saturated or unsaturated, as explained above. Similarly, in all the embodiments of the invention, when a group is mentioned as being in the form of one type of saturation or unsaturation, (e.g. alkyl), it is meant that said group can be in any type of topology (e.g. linear, cyclic or branched) or having several moieties with various topologies.

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

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

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

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

The weight ratio between acyl chloride and the hydrophobic material is preferably comprised between 0.01 and 0.09, more preferably between 0.02 and 0.07.

According to a particular embodiment, the acyl chloride is used in an amount comprised between 1.7 and 7%, preferably between 2.5 and 5 % by weight based on the total weight of the hydrophobic material.

The acyl chloride can be dissolved (or dispersed) directly in the perfume oil or can be pre-dispersed (or pre-dissolved) in an inert solvent or any inert perfumery solvent/ingredient such as benzyl benzoate, triethyl citrate, ethyl acetate, vegetable oil (such as sunflower oil), hexyl salicylate, Neobee (caprylic/capric triglyceride), isopropyl myristate, triglycerides, D-limonene, silicone oil, mineral oil, benzyl salicylate, benzyl benzoate, cyclohexyl salicylate, benzyl phenylacetate, phenylethyl phenylacetate, triacetin, ethyl citrate, methyl and ethyl salicylate, benzyl cinnamate and mixtures thereof, before mixing with the perfume oil.

According to an embodiment, a polyfunctional monomer is added in the oil phase.

By “**polyfunctional monomer**”, it is meant a molecule that, as unit, reacts or binds chemically to form a polymer or supramolecular polymer. The polyfunctional polymer of the invention has at least two functions capable of forming a microcapsule shell.

It should be understood that, when added, the polyfunctional monomer is added in
5 addition to the acyl chloride.

The polyfunctional monomer is preferably chosen in the group consisting of at least one isocyanate, maleic anhydride, acyl chloride, epoxide, acrylate monomers, alkoxy silane and mixtures thereof.

According to an embodiment, the polyfunctional monomer used in the process of the
10 invention is present in amounts representing from 0.1 to 15%, preferably from 0.5 to 10% and more preferably from 0.8 to 6%, and even more preferably between 1 and 3% by weight based on the total amount of the oil phase.

According to a particular embodiment, a polyisocyanate having at least two isocyanate functional groups is further added in the oil phase in addition to the acyl chloride.

15 Suitable polyisocyanates used according to the invention can include aromatic polyisocyanate, aliphatic polyisocyanate and mixtures thereof. Said polyisocyanate comprises at least 2, preferably at least 3 but may comprise up to 6, or even only 4, isocyanate functional groups. According to a particular embodiment, a triisocyanate (3 isocyanate functional group) is used.

20 According to one embodiment, said polyisocyanate is an aromatic polyisocyanate.

The term “aromatic polyisocyanate” is meant here as encompassing any polyisocyanate comprising an aromatic moiety. Preferably, it comprises a phenyl, a toluyl, a xylyl, a naphthyl or a diphenyl moiety, more preferably a toluyl or a xylyl moiety. Preferred aromatic polyisocyanates are biurets, polyisocyanurates and trimethylol propane adducts of
25 diisocyanates, more preferably comprising one of the above-cited specific aromatic moieties. More preferably, the aromatic polyisocyanate is a polyisocyanurate of toluene diisocyanate (commercially available from Bayer under the tradename Desmodur[®] RC), a trimethylol propane-adduct of toluene diisocyanate (commercially available from Bayer under the tradename Desmodur[®] L75), a trimethylol propane-adduct of xylylene diisocyanate
30 (commercially available from Mitsui Chemicals under the tradename Takenate[®] D-110N). In a most preferred embodiment, the aromatic polyisocyanate is a trimethylol propane-adduct of xylylene diisocyanate.

According to another embodiment, said polyisocyanate is an aliphatic polyisocyanate. The term “aliphatic polyisocyanate” is defined as a polyisocyanate which does not comprise

any aromatic moiety. Preferred aliphatic polyisocyanates are a trimer of hexamethylene diisocyanate, a trimer of isophorone diisocyanate, a trimethylol propane-adduct of hexamethylene diisocyanate (available from Mitsui Chemicals) or a biuret of hexamethylene diisocyanate (commercially available from Bayer under the tradename Desmodur® N 100),
5 among which a biuret of hexamethylene diisocyanate is even more preferred.

According to another embodiment, the at least one polyisocyanate is in the form of a mixture of at least one aliphatic polyisocyanate and of at least one aromatic polyisocyanate, both comprising at least two or three isocyanate functional groups, such as a mixture of a biuret of hexamethylene diisocyanate with a trimethylol propane-adduct of xylylene diisocyanate, a
10 mixture of a biuret of hexamethylene diisocyanate with a polyisocyanurate of toluene diisocyanate and a mixture of a biuret of hexamethylene diisocyanate with a trimethylol propane-adduct of toluene diisocyanate. Most preferably, it is a mixture of a biuret of hexamethylene diisocyanate with a trimethylol propane-adduct of xylylene diisocyanate. Preferably, when used as a mixture the molar ratio between the aliphatic polyisocyanate and
15 the aromatic polyisocyanate is ranging from 80:20 to 10:90.

According to an embodiment, the at least one polyisocyanate used in the process of the invention is present in amounts representing from 0.1 to 15%, preferably from 0.5 to 10% and more preferably from 0.8 to 6%, and even more preferably between 1 and 3% by weight based on the total amount of the oil phase.

20 In another step of the process according to the invention, the oil phase of step a) is dispersed into an aqueous solution to form an oil-in-water emulsion.

The mean droplet size of the emulsion is preferably comprised between 1 and 1000 microns, more preferably between 1 and 500 microns, and even more preferably between 5 and 50 microns.

25 The oil-in-water emulsion can be prepared by using high speed mechanical disperser or ultrasonic dispersers, well-known from the person skilled in the art.

Amino-compound A

According to the invention, at least one amino-compound A is added in the water phase
30 before the formation of the oil- in-water emulsion and/or in the oil-in water emulsion obtained after step b).

According to a particular embodiment, at least one amino-compound A is added in the water phase before the formation of the oil- in-water emulsion.

According to a particular embodiment, at least one amino-compound A is added in the oil-in water emulsion obtained after step b).

According to a particular embodiment, at least one amino-compound A is added in the water phase before the formation of the oil- in-water emulsion and in the oil-in water
5 emulsion obtained after step b).

The amino-compound A is preferably chosen in the group consisting of a xylylene diamine, 1,2-diaminocyclohexane, 1,4-diaminocyclohexane, L-lysine, L-Lysine ethyl ester, polyetheramines (Jeffamine®), ethylene diamine, diethylene triamine, spermine, spermidine, polyamidoamine (PAMAM), guanidine carbonate, chitosan, tris-(2-aminoethyl)amine, 3-
10 aminopropyltriethoxysilane, L-arginine, 1,4 diaminobutane, 2,2 Dimethyl-1,3-propanediamine, 1,3-diaminopentane (Dytek EP diamine), 1,2 diaminopropane, an amine having a disulfide bond such as cystamine, cystamine hydrochloride, cystine, cystine hydrochloride, cystine dialkyl ester, cystine dialkyl ester hydrochloride; 1,3-diaminopropane; urea; ethylene urea; aminoguanidine bicarbonate; 1-(2-aminoethyl)imidazolidin-2-one; N-(3-
15 aminopropyl)-N-dodecylpropane-1,3-diamine; N1-(2-Aminoethyl)-N1-dodecyl-1,2-ethanediamine; aminoethylethanolamine; N1-(3-aminopropyl)propane-1,3-diamine and mixtures thereof.

According to a particular embodiment, the amino-compound A is ethylene diamine and is added in the water phase and/or in the oil-in water emulsion obtained after step b).

20 According to an embodiment, the molar ratio between the functional groups NH_2 of the amino compound A and the functional groups COCl of the acyl chloride is comprised between 0.2 and 3, preferably from 0.5 to 2, more preferably between 0.2 and 1.

Base

25 According to an embodiment, the water phase comprises a base, preferably chosen in the group consisting of sodium carbonate, sodium bicarbonate, sodium hydroxide, guanidine carbonate, triethanolamine and mixtures thereof.

According to a particular embodiment, the base is not an amino compound.

According to an embodiment, the water phase comprises a base preferably chosen in
30 the group consisting of sodium carbonate, sodium bicarbonate, sodium hydroxide, and mixtures thereof.

The base can be added in an amount comprise between 0.01 and 1.5%, preferably between 0.01 and 0.7% by weight based on the total weight of the water phase.

Protein

According to a particular embodiment, a protein is added in the oil phase and/or in the water phase. According to a particular embodiment, the protein is added in the oil phase.

The protein is preferably used in an amount comprised between 0.1 and 10%,
5 preferably between 0.5 and 7% by weight based on the total weight of the oil phase or based of the water phase.

According to an embodiment, the protein is a biopolymer.

According to an embodiment, the protein is selected from the group consisting of whey protein, sodium caseinate, bovine serum albumin, casein, gelatin (preferably fish
10 gelatin), plant-based protein, and mixtures thereof.

According to an embodiment, the protein is chosen in the group consisting of soy protein, rice protein, whey protein, white egg albumin, casein, sodium caseinate, gelatin, bovine serum albumin, hydrolyzed soy protein, hydrolyzed sericin, pseudocollagen, silk protein, sericin powder, gelatin and mixtures thereof. According to a particular embodiment,
15 the protein is sodium caseinate.

According to another embodiment, the protein is 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, and mixtures thereof.

According to another embodiment, the protein is chosen in the group consisting of
20 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, bovine serum albumin, hydrolyzed soy protein, hydrolyzed sericin, pseudocollagen, silk protein, sericin powder, gelatin and mixtures thereof.

25 Potato proteins are typically extracted from potato tuber (*Solanum tuberosum*). According to an embodiment, the potato protein is a native potato protein and preferably comprises or consisting of 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
30 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 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-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 or methyl.

According to an embodiment, the protein acts as a stabilizer.

According to an embodiment, a stabilizer can be further added in the water phase and/or the oil phase to form the emulsion. According to an embodiment, the stabilizer is a colloidal stabilizer.

By “stabilizer”, it is meant a compound capable to stabilize oil/water interface as an emulsion typically by lowering the interfacial tension between the oil phase and the water phase.

“**Stabilizer**” or “**emulsifier**” can be used indifferently in the present invention.

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

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

“Molecular emulsifier” and “polymeric emulsifier” are used indifferently in the present invention.

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 non-polymeric substance with a polar and a non-polar group.

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.

According to a particular embodiment, the stabilizer is the polymer as defined above. 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 colloid stabilizer is chosen in the group consisting of gum Arabic, modified starch, polyvinyl alcohol, polyvinylpyrrolidone (PVP), carboxymethylcellulose (CMC), anionic polysaccharides, acrylamide copolymer, inorganic particles, 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, and mixtures thereof.

According to a particular embodiment, the stabilizer is a biopolymer chosen in the group consisting of protein such as whey protein, casein, sodium caseinate, bovine serum albumin, and mixtures thereof.

According to another embodiment, the stabilizer is 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, and mixtures thereof.

According to another embodiment, the stabilizer is 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, bovine serum albumin, hydrolyzed soy protein, hydrolyzed sericin, pseudocollagen, silk protein, sericin powder, gelatin and mixtures thereof.

When added in the oil phase, the stabilizer can be pre-dispersed (or pre-dissolved) in an inert solvent or any inert perfumery solvent/ingredient such as benzyl benzoate, triethyl citrate, ethyl acetate, vegetable oil (such as sunflower oil), hexyl salicylate, Neobee (caprylic/capric triglyceride), isopropyl myristate, triglycerides, D-limonene, silicone oil, mineral oil, benzyl salicylate, benzyl benzoate, cyclohexyl salicylate, benzyl phenylacetate,

phenylethyl phenylacetate, triacetin, ethyl citrate, methyl and ethyl salicylate, benzyl cinnamate and mixtures thereof, or can be mixed to the active ingredient, preferably comprising a perfume oil.

5 The stabilizer and acyl chloride can be premixed and can be heated at a temperature between for example 10 and 80°C before mixing with the hydrophobic material, preferably comprising a perfume oil.

When the colloidal stabilizer is added in the water phase, it is preferably chosen in the group consisting of gum Arabic, modified starch, polyvinyl alcohol, polyvinylpyrrolidone (PVP), carboxymethylcellulose (CMC), anionic polysaccharides, acrylamide copolymer, 10 inorganic particles, 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, and mixtures thereof.

According to any one of the above embodiments of the present invention, the dispersion comprises between about 0.01% and 3.0% of at least a stabilizer, preferably a colloid stabilizer, 15 percentage being expressed on a w/w basis relative to the total weight of the oil-in-water emulsion as obtained after step b). In still another aspect of the invention, the dispersion comprises between about 0.05% and 2.0%, preferably between 0.05 and 1% of at least a stabilizer, preferably a colloid stabilizer. In still another aspect of the invention, the dispersion comprises between about 0.1% and 1.6%, preferably between 0.1% and 0.8% by weight of at 20 least a stabilizer, preferably a colloid stabilizer.

Cross-linker

According to the invention, a cross-linker is added during the process to cross-link the protein.

25 Even if the presence of the cross-linker is an essential feature of the present invention, said cross-linker can be added at different stages of the process.

According to the invention, the cross-linker can be added in step b) and/or in step c) and/or after step c).

When added in step b), the cross-linker can be added in the water phase before the 30 formation of the oil-in-water emulsion and/or once the oil-in-water emulsion is formed.

The cross-linker can be added during the curing step c) and/or after the curing step c).

The cross-linker used in the present invention can be an enzymatic cross-linker such as an enzyme or a non-enzymatic cross-linker such as glutaraldehyde or genipin.

According to a particular embodiment, the cross-linker is an enzyme.

According to a particular embodiment, the enzyme is transglutaminase.

The enzyme may be used in an amount comprised between 0.001 and 5%, preferably between 0.001 to 1%, preferably between 0.001 and 0.1%, preferably between 0.005 and 0.02% based on the total weight of the slurry of step c).

5 In some commercial products, the enzyme is dispersed in a carrier. One may cite for example Activa® TI (Origin: Ajinomoto). In other words, the commercial product is added in the process so as to have the enzyme actives in an amount preferably between 0.001 to 5%, preferably from 0.001 to 1%, even more preferably 0.001 and 0.1%, and even more preferably preferably between 0.005 and 0.02% based total weight of the slurry of step c).

10 According to an embodiment, when the cross-linker is added before the curing step c), (typically when the cross-linker is added in step b) in the water phase before the formation of the oil-in-water emulsion and/or once the oil-in-water emulsion is formed), the process typically includes a curing step c) which allows ending up with microcapsules in the form of a slurry and at the same time cross-link the protein within the shell. According to a preferred
15 embodiment, to enhance the kinetics, said curing step is performed at a temperature comprised between 5 and 90°C, possibly under pressure, for 1 to 8 hours. More preferably it is performed at between 10 and 80°C for between 30 minutes and 5 hours.

When the cross-linker is an enzyme, once the shell is formed and once the protein is cross-linked, a heating treatment can be performed on the slurry to deactivate the enzyme.

20 Typically, the heating treatment can be performed at a temperature comprised between 70 °C and 90 °C.

 According to an embodiment, when the cross-linker is added during the curing step c), the process typically includes a curing step c) which allows ending up with microcapsules in the form of a slurry and at the same time cross-link the protein within the shell. According to
25 a preferred embodiment, to enhance the kinetics, said curing step is performed at a temperature comprised between 5 and 90°C, possibly under pressure, for 1 to 8 hours. More preferably it is performed at between 10 and 80°C for between 30 minutes and 5 hours.

When the cross-linker is an enzyme, once the shell is formed and once the protein is cross-linked, a heating treatment can be performed on the slurry to deactivate the enzyme.

30 Typically, the heating treatment can be performed at a temperature comprised between 70 °C and 90 °C.

 According to an embodiment, when the cross-linker is added after the curing step c), in addition to the curing step c) which allows ending up with microcapsules in the form of a slurry,

the process further comprises an additional curing step d) after the addition of the cross-linker to cross-link the protein within the shell.

The curing step c) and/or the curing step d) can be performed at a temperature comprised between 5 and 90°C, possibly under pressure, for 1 to 8 hours. More preferably it is performed at between 10 and 80°C for between 30 minutes and 5 hours.

When the cross-linker is an enzyme, once the shell is formed and once the protein is cross-linked, an additional heating treatment can be performed on the slurry to deactivate the enzyme. Typically, the heating treatment can be performed at a temperature comprised between 70 °C and 90 °C.

According to the invention, the curing step c) and/or curing step d) can be performed at room temperature under stirring or can comprise a heating step to enhance the kinetics.

Amino-compound B

According to an embodiment, at least one amino-compound B is added in the water phase before the formation of the oil-in-water emulsion and/or in the oil-in water emulsion obtained after step b).

According to a particular embodiment, at least one amino-compound B is added in the water phase before the formation of the oil- in-water emulsion.

According to a particular embodiment, at least one amino-compound B is added in the oil-in water emulsion obtained after step b).

According to a particular embodiment, at least one amino-compound B is added in the water phase before the formation of the oil- in-water emulsion and in the oil-in water emulsion obtained after step b).

According to an embodiment, the amino-compound A and amino-compound B are the same.

According to another embodiment, the amino-compound A and amino-compound B are different.

According to a particular embodiment, the amino-compound B is is an amino-acid, preferably chosen in the group consisting of L-Lysine, L-Leucine, L-Arginine, L-Histidine, L-Tryptophane, L-Serin, L-Glutamine, L-Threonine and/or its derived oligomers and polymers, and mixtures thereof, preferably L-Lysine, L-Arginine, L-Histidine, L-Tryptophane and mixtures thereof, more preferably L-Lysine, L-Arginine, L-Histidine and mixtures thereof.

The amino-acid has preferably two nucleophilic groups.

According to a particular embodiment, the amino-compound B may be chosen in the group consisting of L-Lysine, L-Lysine ethyl ester, guanidine carbonate, chitosan, 3-aminopropyltriethoxysilane, and mixtures thereof. According to a particular embodiment, the amino compound B is L-Lysine.

- 5 According to an embodiment, the amino-compound B is L-Lysine and is added in the water phase before the formation of the oil-in-water emulsion and/or in the oil-in water emulsion obtained after step b).

- According to an embodiment, the weight percent of amino-compound B in the water phase is comprised between 0 and 5%, preferably between 0.1 and 5%, more preferably
10 between 0.1 and 2%.

 According to a particular embodiment, a multivalent salt (such as calcium chloride, magnesium chloride, zinc chloride, iron trichloride) is added after step b), before or during step c).

15 **Carbohydrate**

According to an embodiment, a carbohydrate is added in the water phase and/or in the oil phase.

 According to an embodiment, by “carbohydrate” it should be understood a polymer or an oligomer having a number of units greater than 2.

- According to another embodiment, the carbohydrate, the amino-compound A and the
20 amino compound B are different components.

 According to an embodiment, at least one carbohydrate is added in the oil phase and/or in the water phase.

 According to an embodiment, the carbohydrate is not a polyphenol.

 According to an embodiment, the carbohydrate is not a functionalized carbohydrate.

- 25 According to an embodiment, the carbohydrate is a polysaccharide.

 According to an embodiment, the polysaccharide is an anionic polysaccharide.

 According to a particular embodiment, the polysaccharide is added in the water phase.

- The polysaccharide is preferably chosen in the group consisting of anionic salt of alginic acid, preferably alginic acid sodium salt, pectin, lignin, anionic modified starch,
30 carboxymethylcellulose and mixtures thereof.

 According to a particular embodiment, the carbohydrate is an anionic salt of alginic acid, preferably alginic acid sodium salt.

 “Alginic acid sodium salt” and “sodium alginate” can be used indifferently.

According to a particular embodiment, the carbohydrate is used in an amount comprised between 0.1 and 5%, preferably between 0.5 and 1.1% by weight based on the total weight of the water phase.

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Optional outer coating

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

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 (Mw) molecular weight of the cationic polymer is preferably between 10,000 and 3.5M Dalton, more preferably between 50,000 and 1.5M Dalton. According to a particular embodiment, one will use cationic polymers based on acrylamide, methacrylamide, N-vinylpyrrolidone, quaternized N,N-dimethylaminomethacrylate, diallyldimethylammonium chloride, quaternized vinylimidazole (3-methyl-1-vinyl-1H-imidazol-3-ium chloride), vinylpyrrolidone, acrylamidopropyltrimonium chloride, cassia hydroxypropyltrimonium chloride, guar hydroxypropyltrimonium chloride or polygalactomannan 2-hydroxypropyltrimethylammonium chloride ether, starch hydroxypropyltrimonium chloride and cellulose hydroxypropyltrimonium chloride. Preferably copolymers shall be selected from the group consisting of polyquaternium-5, polyquaternium-6, polyquaternium-7,

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

According to any one of the above embodiments of the invention, there is added an amount of polymer described above comprised between about 0% and 5% w/w, or even between about 0.1% and 2% w/w, percentage being expressed on a w/w basis relative to the total weight of the slurry as obtained after step c) or d). 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.

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

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

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

CORE-SHELL MICROCAPSULE

Another object of the invention is a microcapsule or a microcapsule slurry obtainable by the process as described above.

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

- 5 - a core, preferably an oil-based core, comprising a hydrophobic material, preferably a perfume, and
- a polyamide-based shell comprising a reaction product of:
 - an acyl chloride,
 - an amino compound A,
 - 10 • optionally an amino compound B,
 - a protein,

wherein the shell has a rupture stress less than 10 MPa.

According to an embodiment, the shell comprises a carbohydrate as defined previously.

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According to an embodiment, the shell has a rupture stress less than 9MPa. According to an embodiment, the shell has a rupture stress less than 8MPa. According to an embodiment, the shell has a rupture stress less than 7MPa. According to an embodiment, the shell has a rupture stress less than 6MPa. According to an embodiment, the shell has a rupture stress less than 5MPa. According to an embodiment, the shell has a rupture stress less than 4MPa. According to an embodiment, the shell has a rupture stress less than 3MPa.

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According to an embodiment, the shell has a rupture stress comprised between 0.1 and 10MPa. According to an embodiment, the shell has a rupture stress comprised between 0.1 and 9MPa.

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According to an embodiment, the shell has a rupture stress comprised between 0.1 and 8MPa.

According to an embodiment, the shell has a rupture stress comprised between 0.1 and 7MPa.

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According to an embodiment, the shell has a rupture stress comprised between 0.1 and 6MPa.

According to an embodiment, the shell has a rupture stress comprised between 0.1 and 5MPa.

According to an embodiment, the shell has a rupture stress comprised between 0.1 and 4MPa.

According to an embodiment, the shell has a rupture stress comprised between 0.1 and 3MPa.

- 5 Method for determining rupture stress are well-known by the person skilled in the art. A typical method is the following: 2 drops of microcapsules suspension are diluted in 10ml of demineralized water. The dilute solution is applied to a 20x20mm glass microscope slide and then removed. This is repeated once more and then small single droplets of about 1mm are left on the glass slides and allowed to dry at room temperature in controlled relative humidity.
- 10 Sample was left to dry overnight. Once the sample is dried, the glass slide is placed under the microscope and analyzed with the Femto Tools micro-force probe.
- A setup using a micro-force probe/xyz robot/microscope assembly was used for the measurements. The point 'distance zero' of the probe is set at 100µm from the glass slide. The probe is then placed over a microcapsule at about 20µm from the glass. The program is
- 15 initiated and the micro-force probe moves downward applying pressure onto the capsule. The force as a function of distance is recorded. The force curves are processed using an R statistical programming package (<https://www.r-project.org/>) applying an R script developed to extract mechanical properties from the curves.
- Rupture stress is a well-known parameter that can be obtained by dividing the rupture force by
- 20 the average size of microcapsules (both parameters provided by the program defined above).

According to an embodiment, the polyamide-shell comprises the reaction product of:

- an acyl chloride,
 - an amino compound A,
 - 25 • optionally an amino compound B,
 - a protein
- in the presence of a cross-linker, as defined previously.

According to an embodiment, the shell comprises a cross-linked protein.

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

- a core, preferably an oil-based core, comprising a hydrophobic material, preferably a perfume, and
- a polyamide-based shell comprising a reaction product of
 - an acyl chloride in an amount comprised between 5 and 98%, preferably between 20 and 98%, more preferably between 30 and 85% w/w
 - an amino compound A, in an amount comprised between 1% and 50% w/w, preferably between 7 and 40% w/w;
 - optionally an amino compound B, in an amount comprised between 1% and 50% w/w, preferably between 2 and 25% w/w
 - a protein, in an amount comprised between 0.1 and 90%, preferably between 0.1 and 75%, more preferably between 1 and 70%.

The embodiments described above for the process according to the invention also apply to the microcapsule or the microcapsule slurry according to the invention. This particularly applies to the hydrophobic material, the cross-linker, the protein, the acyl chloride, the amino compound(s), the stabilizer.

According to an embodiment, amino-compound A and amino-compound B are different.

According to a particular embodiment, the polyamide microcapsule comprises an inner shell of polyurea.

The composition of the shell can be quantified for example by elemental analysis and identified by solid-state NMR which are two well-known techniques for the person skilled in the art.

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.

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 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%,
5 95% or 98% within 60 days according to OECD301F.

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*.

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In a particular embodiment, the microcapsule has a stability or chemical stability of not more than 50%, preferably not more than 40%, preferably not more than 35%, preferably more than 30%. Typically, the stability or chemical stability of the microcapsules being determined as not more than 50%, preferably not more than 40%, preferably not more than 35%, preferably
15 not more than 30%, of the perfume leaking out of the microcapsules when incorporated in a consumer product for a particular storage time and temperature, with the microcapsules being stable after 15 days storage at 37° C, more preferably after 30 days storage at 37°C preferably in fabric softeners, liquid detergents, body washes, deodorants or antiperspirants, for at least 2 weeks storage at 40° C in body lotions, shampoos or hair conditioners.

Furthermore, the microcapsule shows preferably a rubbing effect detectable on fresh
20 samples, and preferably after 15 days of storage in application at 37°C, even more preferably after 30 days at 37°C.

Another object of the invention is a solid particle comprising:

- a carrier material, preferably a polymeric carrier material chosen in the group consisting
25 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.

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

Optional components

When microcapsules are in the form of a slurry, the microcapsule slurry can comprise auxiliary ingredients selected from the group of thickening agents/rheology modifiers, antimicrobial agents, opacity-building agents, mica particles, salt, pH stabilizers/buffering ingredients, preferably in an amount comprised between 0 and 15% by weight based on the total weight of the slurry.

According to another embodiment, the microcapsule slurry of the invention comprises additional free (i.e non-encapsulated) perfume, preferably in an amount comprised between 5 and 50% by weight based on the total weight of the slurry.

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.

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.

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, polysiloxane, 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 comprises 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, polysiloxane, 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-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 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 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 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 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-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;
- 2) dispersing or dissolving into water an aminoplast resin and optionally a stabilizer to form a water 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 water phase;
- 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

- 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;
 - 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;

- 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;
 - 5 c. at least an oligomeric composition as obtained in step 1;
 - 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
 - 10 ii. a di- or tri-oxiran compounds of formula:

$$A-(\text{oxiran-2-ylmethyl})_n$$

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;
 - e. optionally a C₁-C₄ compounds comprising two NH₂ functional groups;
 - 15 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,
- 20 - 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 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 thereof.

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

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

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%.

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,
 - 5 • 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 mixture thereof.
 - a second amino-compound chosen in the group consisting of ethylene diamine, diethylene triamine, cystamine and/or mixture thereof, and
 - 10 • a biopolymer chosen in the group consisting of casein, sodium caseinate, bovin serum albumin, whey protein, and/or mixture thereof.

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:

- 20 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 water phase;
- c) Adding the oil phase to the water 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
- 25 d) Applying conditions sufficient to induce interfacial polymerisation and form microcapsules in form of a slurry.

30 **PERFUMING COMPOSITION AND CONSUMER PRODUCTS**

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

- (i) microcapsules as defined above;

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

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

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

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

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

The nature and type of the perfuming co-ingredients present in the perfuming composition do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled person being able to select them on the basis of his general knowledge and according to the 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 sulfurous heterocyclic compounds and essential oils, and said perfuming co-ingredients can be of natural or synthetic origin. Many of these co-ingredients are in any case listed in reference texts such as the book

5 by S. Arctander, *Perfume and Flavor Chemicals*, 1969, Montclair, New Jersey, USA, or its more recent versions, or in other works of a similar nature, as well as in the abundant patent literature in the field of perfumery. It is also understood that said co-ingredients may also be compounds known to release in a controlled manner various types of perfuming compounds. Co-ingredients may be chosen in the group consisting of 4-(dodecylthio)-4-(2,6,6-trimethyl-

10 2-cyclohexen-1-yl)-2-butanone, 4-(dodecylthio)-4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-butanone, trans-3-(dodecylthio)-1-(2,6,6-trimethyl-3-cyclohexen-1-yl)-1-butanone, 2-(dodecylthio)octan-4-one, 2-phenylethyl oxo(phenyl)acetate, 3,7-dimethylocta-2,6-dien-1-yl oxo(phenyl)acetate, (Z)-hex-3-en-1-yl oxo(phenyl)acetate, 3,7-dimethyl-2,6-octadien-1-yl hexadecanoate, bis(3,7-dimethylocta-2,6-dien-1-yl) succinate, (2-((2-methylundec-1-en-1-

15 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,

20 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-

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

By "perfumery adjuvant" we mean here an ingredient capable of imparting additional added benefit such as a color, a particular light resistance, chemical stability, etc. A detailed description of the nature and type of adjuvant commonly used in perfuming bases cannot be

30 exhaustive, but it has to be mentioned that said ingredients are well known to a person skilled in the art.

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

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

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

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

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

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

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

In particular a liquid consumer product comprising:

- a) from 2 to 65% by weight, relative to the total weight of the consumer product, of at least one surfactant;
- 30 b) water or a water-miscible hydrophilic organic solvent; and
- c) a perfuming composition as defined above is another object of the invention.

Also a powder consumer product comprising

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

(b) a perfuming composition as defined above is part of the invention.

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

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

The nature and type of the other constituents of the perfumed consumer product do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled person being able to select them on the basis of his general knowledge and according to the nature and the desired effect of said product. Base formulations of consumer products in which the 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.

Non-limiting examples of suitable perfumed consumer products can be a perfume, such as a fine perfume, a cologne, an after-shave lotion, a body-splash; a fabric care product, such as a liquid or solid detergent, tablets and unit dose (single or multi-chambers), a fabric softener, a dryer sheet, a fabric refresher, an ironing water, or a bleach; a personal-care product, such as a hair-care product (e.g. a shampoo, hair conditioner, a coloring preparation or a hair spray), a cosmetic preparation (e.g. a vanishing cream, body lotion or a deodorant or antiperspirant), or a skin-care product (e.g. a perfumed soap, shower or bath mousse, body wash, oil or gel, bath salts, or a hygiene product); an air care product, such as an air freshener or a "ready to use" powdered air freshener; or a home care product, such all-purpose cleaners, liquid or power or tablet dishwashing products, toilet cleaners or products for cleaning various surfaces, for example sprays & wipes intended for the treatment / refreshment of textiles or hard surfaces (floors, tiles, stone-floors etc.); a hygiene product such as sanitary napkins, diapers, toilet paper.

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,
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.

- 5
- 10 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), 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);

- 15 Another object of the invention is a consumer product comprising:

- 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.

- 20 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 his general knowledge and of the available literature.
- 25

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

- 30 An object of the invention is a consumer product, preferably a home care or a fabric care consumer product comprising the microcapsules or the microcapsule slurry as defined above, wherein the consumer product has a pH less than 7.

An object of the invention is a consumer product, preferably a home care or a fabric care consumer product comprising the microcapsules or the microcapsule slurry as defined above, wherein the consumer product has a pH equals or greater than 7.

Another object of the invention is a consumer product, preferably a home care or a fabric care consumer product comprising microcapsules, wherein the consumer product has a pH equals or greater than 7, and wherein the microcapsule comprises

- a core, preferably an oil-based core, comprising a hydrophobic material, preferably a perfume, and
- a polyamide-based shell comprising a reaction product of:

- an acyl chloride,
- an amino compound A,
- optionally an amino compound B,
- a protein,

preferably in the presence of a cross-linker as defined above, and

wherein 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.

Another object of the invention is a consumer product, preferably a home care or a fabric care consumer product comprising microcapsules, wherein the consumer product has a pH less than 7, and wherein the microcapsule comprises

- a core, preferably an oil-based core, comprising a hydrophobic material, preferably a perfume, and
- a polyamide-based shell comprising a reaction product of:
 - an acyl chloride,
 - an amino compound A,
 - optionally an amino compound B,
 - a protein,

preferably in the presence of a cross-linker as defined above, and

wherein 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

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

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

Fabric softener

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

- 10 - 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,
- 15 - a microcapsule slurry or microcapsules 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,
- 20 - optionally free perfume oil.

Liquid detergent

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

- 25 - 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,
- 30

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

Shampoo/shower gel

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

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

- optionally free perfume oil.

Rinse-Off Conditioner

An object of the invention is a consumer product in the form of a rinse-off conditioner
5 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
10 on the total weight of the composition,
- a microcapsule slurry or microcapsules 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.

15

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,
20 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
25 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,
- a microcapsule slurry or microcapsules as defined above, in a powdered form, preferably in an amount comprised between 0.05 to 15 wt%, more preferably between
30 0.1 and 5 wt% by weight based on the total weight of the composition,
- optionally free perfume oil.

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,
- 5 - 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,
- 10 alkyl polyglucosides, 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 than 10 and mixtures thereof, and
- 15 - a microcapsule slurry or microcapsules 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.

20 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 comprising an alkakine agent, a dye precursor and a coupling compound; wherein said dye precursor
- 25 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
- 30 based on the total weight of the composition,
- optionally free perfume oil

Perfuming composition

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

- 5 - 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.

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

EXAMPLES

15 General protocol:

Process A: Cross-linker (TG) added in the water phase before the emulsification step

Sodium Caseinate is dispersed in Neobee (inert organic solvent) for 30min at 60°C under magnetic stir in a water bath. This pre-mixture and the acyl chloride compound were added to the perfume (see Table 2) to form an oil phase. Aqueous phase was formed of L-lysine, at a concentration of 2.5 wt% and was kept at 45°C. Right before emulsion step, the enzyme (e.g transglutaminase) was added to the aqueous phase at a concentration of 1.5%. Oil phase was mixed with aqueous solution and stirred with an Ultra Turrax at 25,000 rpm for 30 s to afford an emulsion. A solution of ethylene diamine and a base, such as sodium hydroxide, was added dropwise to the suspension. The reaction mixture was stirred at 45°C for 3 h to afford a white dispersion and then for 1hours at 70°C for the enzyme deactivation.

Process B: Cross-linker (TG) added in the slurry after the curing step

Sodium Caseinate is dispersed in Neobee for 30min at 60°C under magnetic stir in a water bath. This pre-mixture and the acyl chloride compound were added to the perfume to form an oil phase. Aqueous phase was formed of L-lysine, at a concentration of 2.5 wt% and was kept at 45°C. Oil phase was mixed with aqueous solution and stirred with an Ultra Turrax at 25,000 rpm for 30 s to afford an emulsion. A solution of ethylene diamine and a base, such as sodium hydroxide, was added dropwise to the suspension. The reaction mixture was stirred at 60°C for

4 h to afford a white dispersion. This suspension was adjusted to pH7. A solution of CaCl_2 (0.5g in 2g of water) and a solution of Transglutaminase (1g in 5g of water) were added successively to the dispersion. Mixture is then left under stir at 45°C for 3hours and then for 1hour at 70°C for the enzyme deactivation.

5

Table 1: Ingredients

Ingredients	Effective Amount of Ingredient (% wt)
BTC ¹⁾	1.2
EDA ²⁾	0.2
Sodium Caseinate ³⁾	1.4
Sodium Hydroxide ⁴⁾	0.3
L-Lysine ⁵⁾	1.8
Transglutaminase ⁶⁾	1.0

1) 1,3,5-benzene tricarbonyl chloride; origin : Aldrich, Switzerland

2) Ethylene Diamine; origin : Aldrich, Switzerland

3) origin: Aldrich, Switzerland

10 4) origin: Aldrich, Switzerland

5) origin: Aldrich, Switzerland

6) Activa TI® origin: Ajinomoto

Perfume:

Table 2 - Formulation of the perfume oil

Ingredients	% in oil
Ethyl 2-methyl-pentanoate	3.20%
Eucalyptol	7.80%
2,4-Dimethyl-3-cyclohexene-1-carbaldehyde	0.75%
Aldehyde C10	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 C12	1.30%
Dicyclopentadiene acetate	9.85%
Ionone beta	3.30%
Undecalactone gamma	18.75%
Hexyl Salicylate	15.90%
Benzyl Salicylate	16.20%

5

Example 1Preparation of microcapsules according to the invention (process A)

- Preparation of **capsules A** with 1,3,5-benzene tricarbonyl chloride (BTC – acyl chloride), Sodium Caseinate (NaCas - stabilizer), Ethylene diamine (EDA – Amino-compound A), L-
 10 Lysine (Amino-compound B), Sodium Hydroxide (NaOH - base), Transglutaminase (Tg – cross-linker), Calcium Chloride (salt) and perfume oil (see Table 2).

15

Table 3: Capsule A compositions

Capsules	NaCas (g)	BTC (g)	EDA (g)	Perfume (g)	Tg (g)	L- lysine (g)	Base (g)	Inert organic Solvent (g)	Water (g)	Calcium Chloride (g)
A1	2	1.77	0.3	25	1.5	2.5	0.35	10	95	0
A2	2	1.77	0.3	25	1.5	2.5	0.35	10	95	0.8
A3	1	0.88	0.18	25	1.5	1.25	0.15	10	95	0
A4	2	1.77	0.3	25	1.5	2	0.5	10	95	0
A5	1.25	0.88	0.18	25	1.5	1.25	0.15	10	95	0
A6	2	1.5	0.25	25	1.5	2.15	0.35	10	95	0

5

Example 2Preparation of microcapsules according to the invention (process B)

Preparation of **capsules B** with 1,3,5-benzene tricarbonyl chloride (BTC – acyl chloride), Sodium Caseinate (NaCas - stabilizer), Ethylene diamine (EDA – Amino-compound A), L-Lysine (Amino-compound B), Sodium Hydroxide (NaOH - base), Transglutaminase (Tg – cross-linker), Calcium Chloride (salt) and perfume oil (see Table 2)

Table 4: Capsule compositions:

Capsules	NaCas (g)	BTC (g)	EDA (g)	Perfume (g)	Tg (g)	L- lysine (g)	Base (g)	Inert organic Solvent (g)	Water (g)	Calcium Chloride (g)
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B1	2	1.77	0.3	25	1.5	2	0.4	10	95	0.75
B2	2	1.77	0.3	25	1.5	2.5	0.3	10	95	0.75
B3	2	1.5	0.3	25	1.5	2.1	0.3	10	95	0.75
B4	2	0.88	0.18	25	1.5	1.25	0.15	10	95	0.75
B5	1	1.33	0.2	25	0.75	1.8	0.4	10	95	0.75
B6	2	1.33	0.22	25	1.5	0	0.65	10	95	0.75
B7	2	1.77	0.3	25	1.5	2	0.5	10	95	0.75

Example 3

Stability performance

5

Microcapsules of the present invention are dispersed in a fabric softener composition described in Table 5 or in a liquid detergent composition described in Table 6 to obtain a concentration of encapsulated perfume oil at 0.116%.

10 Table 5: Fabric Conditioner composition

Product	Wt %
Stepantex VL 90A	8.88
Calcium Chloride Sol. 10%	0.36
Proxel GXL	0.04
Perfume	1.00
Water	89.72
TOTAL	100

Table 6: 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

10 Weigh 2g of sample (base with capsules) in a 20mL vial. Add to the vial 10 mL of the extraction solvent isooctane containing the internal standard 1,4-dibromobenzene at a precisely known concentration around 90 ng/uL. Shake for 45 min at 40RPM to extract the free perfume. Remove the solvent phase.

To measure the leakage in the base the Agilent GCFID7890A is use, the injector is set at 250°C, helium is used as the carrier gas at a flow rate of 1 mL/min, the oven temperature is programmed from 120°C, held 5 minutes, increased to 170°C at 10°C/min, increased to 220°C at 25°C/min and then increased to 260 at 25°C/min. To finish a post run is apply at 260°C to

5 finish the measure.

Calibration solutions are prepared at 100, 300 and 600 ng/uL of fragrance oil in the isooctane. It is important that the fragrance oil used to prepare the calibration curve comes from the same batch used to produce the microcapsules.

10 Table 7: Stability

Perfume Leakage at 37°C (3days)	Fabric Conditioner	Liquid Detergent
A1	30%	21%

Table 8: Stability

Perfume Leakage at 37°C (3days/1month)	Fabric Conditioner	Liquid Detergent
B1	24%/34%	21%/35%
B2	20%/31%	20%/40%
B3	12%/22%	19%/36%
B4	26%/46%	26%/46%
B5	NM	22%/43%

NM: not measured

15 One can conclude that microcapsules prepared by the process of the present invention show satisfactory stability in consumer product.

Example 4

Mechanical Properties Measurements

A. Samples description

5

Microcapsules were prepared according to Process A.

Table 9: Capsule compositions

Sample	BTC	Perfume ¹⁾	Inert Organic Solvent	Sodium Caseinate	EDA	L-Lysine	Water	Tg	Sodium Hydroxide
Microcapsules C	1.33	25	10	2	0.22	1.8	95	1.5	0.35

1) See Table 2

10

B. Protocol

2 drops of microcapsules suspension are diluted in 10ml of demineralized water. The dilute solution is applied to a 20x20mm glass microscope slide and then removed. This is repeated once more and then small single droplets of about 1mm are left on the glass slides and allowed to dry at room temperature in controlled relative humidity. Sample was left to dry overnight. Once the sample is dried, the glass slide is placed under the microscope and analyzed with the Femto Tools micro-force probe.

A setup using a micro-force probe/xyz robot/microscope assembly was used for the measurements. The point 'distance zero' of the probe is set at 100µm from the glass slide. The probe is then placed over a microcapsule at about 20µm from the glass. The program is initiated and the micro-force probe moves downward applying pressure onto the capsule. The force as a function of distance is recorded. The force curves are processed using an R statistical programming package (version 3.1.2; <http://www.R-project.org>) applying an R script developed to extract mechanical properties from the curves.

Those parameters have also been described in *Hybrid microcapsules with tunable properties via Pickering emulsions templates for the encapsulation of bioactives volatiles* (RCS Adv., 2016, 6, 102595).

C. Summary

Table 10: Mechanical properties

Sample	Percentage Broken Capsules	Stiffness (N/m)	Adhesion (μ N)	Elasticity (J)	Rupture Stress (MPa)
Microcapsules C	36.86%	9.24	-22.27	8.4×10^{-7}	2.11

5

Microcapsules C (with enzymatic reaction) shows a high percentage of broken microcapsules which is favorable for sensory performance.

Sample B showed a high adhesion which can also be in favor of the sensory performance by improving deposition when in customer application.

10

D. Conclusion

It can be underlined that the enzymatic reaction confers good microcapsule's shell mechanical properties.

15

Example 5Spray-dried microcapsules preparation

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

Table 11: 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 C 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 C	0%	0%	0%	28.1%	0%
Microcapsules I-L	12.%	24.1%	36.1%	9.8%	48.4%
Silica	3.0	3.0%	2.9%	2.0%	2%
Fragrance loading in powder after spray-drying	10.1%	20.1%	30%	35.8%	40.2%

1) Capsul™, Ingredient

2) Maltodextrin 10DE origin: Roquette

3) Maltose, Lehmann & Voss

4) Silica, Evonik

5) see table 12

5

Table 12: Composition of Perfume C

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

10 3) 1-(octahydro-2,3,8,8-tetramethyl-2-naphthalenyl)-1-ethanone, International Flavors & Fragrances, USA

- 4) Firmenich SA, Switzerland
- 5) Methyl dihydrojasmonate, Firmenich SA, Switzerland
- 6) Firmenich SA, Switzerland
- 5 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 C 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).
- 10 Granulated powder A-E are prepared by spray-drying Emulsion A-E using a Sodeva 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 6

15 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.

20 Table 13: 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 : KKK 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.

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.

15

Example 7

Liquid detergent composition

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

Table 14: 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

6) Hostapur SAS 60; Origin: Clariant

7) Edenor K 12-18; Origin: Cognis

8) Genapol LA 070; Origin: Clariant

9) Origin: Genencor International

5 10) Aculyn 88; Origin: Dow Chemical

Example 8

Unit dose formulation

- 10 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.

Table 15: 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 9

Powder detergent composition

5

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

Table 16: Powder detergent composition

10

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%

Example 10Concentrated All Purpose Cleaner composition

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

Table 17: concentrated all-purpose cleaner composition

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

10 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

All ingredients are mixed together and then the mixture was diluted with water to 100%.

15

Example 11Solid scent booster composition

The following compositions are prepared.

20 Table 18: Salt-based solid scent booster compositions

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

Table 19: Urea-based solid scent booster compositions

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

Example 12

5

Shampoo composition

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

10 Table 20: Shampoo composition

	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
- 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 mixed for 5 min. Then

- 5 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.

Example 13

10

Shampoo composition

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

- 15 Table 21: 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
	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
	T O T A L:	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
- 5 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.

Example 14Rinse-off hair composition

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

Table 22: rinse-off composition

	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.
	T O T A L:	100

- 1) Genamin KDMP, Clariant
- 10 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
- 15 7) Xiameter MEM-949, Dow Corning
- 8) Alfa Aesar

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 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.

Example 15

Antiperspirant spray anhydrous composition

10

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

Table 23: antiperspirant spray anhydrous composition

15

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

2) Aerosil® 200 ; trademark and origin : Evonik

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

4) Micro Dry Ultrafine; origin : Reheis

20

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).

25

Example 16Antiperspirant spray emulsion composition

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

Table 24: antiperspirant spray emulsion composition

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

- 10 1) Tween 65; trademark and origin : CRODA
 2) Dehymuls PGPH; trademark and origin : BASF
 3) Abil EM-90; trademark and origin : BASF
 4) Dow Corning 345 fluid; trademark and origin : Dow Corning
 5) Crodamol ipis; trademark and origin : CRODA
 15 6) Phenoxyethanol; trademark and origin : LANXESS
 7) Sensiva sc 50; trademark and origin : KRAFT

- 8) Tegosoft TN; trademark and origin : Evonik
- 9) Aerosil R 812; trademark and origin : Evonik
- 10) Nipagin mna; trademark and origin : CLARIANT
- 11) Locron L; trademark and origin : CLARIANT

5

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

10 into the aerosol cans. The propellant is crimped and added.

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

Example 17

15

Deodorant spray composition

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

20 Table 25: deodorant spray composition

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

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

25 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: 40% active solution 60% Propane / Butane 2.5 bar).

Example 18Antiperspirant roll-on emulsion composition

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

Table 26: 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
 10 2) BRIJ 721; origin : ICI
 3) ARLAMOL E; origin : UNIQEMA-CRODA
 4) LOCRON L; origin : CLARIAN

Part A and B are heated separately to 75°C; Part A is added to part B under stirring and the
 15 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.

Example 1920 Antiperspirant roll-on composition

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

Table 27: 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
Cetareth-12 ²⁾ (Part C)	2
Cetareth-30 ³⁾ (Part C)	2
Fragrance (Part D)	1

1) LOCRON L; origin: CLARIANT

2) EUMULGIN B-1; origin : BASF

5 3) EUMULGIN B-3; origin : BASF

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 20

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 28: 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

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 limp gel. Then, Part B is poured little by little in Part A whilst continuing stirring until the whole is homogeneous. Part C is added.

Example 21

10 Deodorant pump without alcohol formulation

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

15 Table 29: 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

1) Ceraphyl 41; trademark and origin ASHLAND

2) DOW CORNING 200 FLUID 0.65cs; trademark and origin DOW CORNING CORPORATION

20 3) Ceraphyl 28; trademark and origin ASHLAND

4) Eutanol G; trademark and origin BASF

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.

Example 22Deodorant pump with alcohol formulation

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

Table 30: 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

- 10 2) Cremophor[®] RH 40; trademark and origin : BASF

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.

15

Example 23Talc formulation

- 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
 20 equivalent of 0.2% perfume.

Example 24Shower-gel Reference

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

Table 31: shower gel composition

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

1) EDETA B POWDER; trademark and origin: BASF

5 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)).

Example 25

Shower-gel composition

15

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

Table 32: shower gel composition

Ingredients	Amount (% wt)	Function
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)).

Example 26

10

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 33: shower gel composition

Ingredients	Amount (% wt)	Function
WATER deionized	50.950	Solvent
Tetrasodium EDTA ¹⁾	0.050	Chelating agent
Sodium Benzoate	0.500	Preservative
Glycerin 86%	3.500	Solvent
Sodium Laureth Sulfate ²⁾	27.000	Surfactant
Polyquaternium-7 ³⁾	1.000	Conditioning Agent
Coco-Betaine ⁴⁾	6.000	Surfactant
PEG-120 Methyl Glucose trioleate ⁵⁾	1.000	Thickener
Citric Acid (40%)	1.000	pH adjuster
Glycol Distearate & Laureth-4 & Cocamidopropyl Betaine ⁶⁾	3.000	Pearlizing agent
Sodium Chloride 20%	5.000	Viscosity adjuster
PEG-40 Hydrogenated Castor Oil ⁷⁾	1.000	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 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
10 / Spindle#4 / 20RPM))

Example 27

Hand Dishwash

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

Table 34: 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

- 20 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

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.

5

Example 28Toothpaste 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.

10

Table 35: 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

2) Tixosil 43

15

Example 29Dicalcium 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 mixed in the following composition to add the equivalent of 0.2% flavor.

5 Table 36: 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

Example 30

Mouthwash alcohol free formulation

10

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 37: 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 31Mouthwash 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.

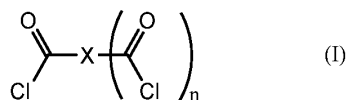
10 Table 38: Mouthwash formulation

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 process for preparing a polyamide-based core-shell microcapsule slurry comprising
5 the following steps:
a) Dissolving at least one acyl chloride in a hydrophobic material, preferably a perfume
to form an oil phase;
b) Dispersing the oil phase obtained in step a) into a water phase to form an oil-in-water
emulsion;
10 c) Performing a curing step to form polyamide-based microcapsules in the form of a
slurry;
wherein at least one amino-compound A is added in the water phase before the
formation of the oil- in-water emulsion and/or in the oil-in water emulsion obtained
after step b),
15 wherein a protein is added in the oil phase and/or in the water phase, and
wherein a cross-linker is added in step b) and/or in step c) and/or after step c).
- 2- The process according to claim 1, wherein the cross-linker is an enzymatic cross-
linker preferably selected from the group consisting of transglutaminase or wherein
20 the cross-linker is a non-enzymatic cross-linker preferably selected from the group
consisting of glutaraldehyde, genipin and mixtures thereof.
- 3- The process according to claim 2, wherein the cross-linker is transglutaminase.
- 25 4- The process according to anyone of the preceding claims, wherein the protein is
selected from 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, bovine serum albumin, hydrolyzed soy protein, hydrolyzed
30 sericin, pseudocollagen, silk protein, sericin powder, gelatin and mixtures thereof.

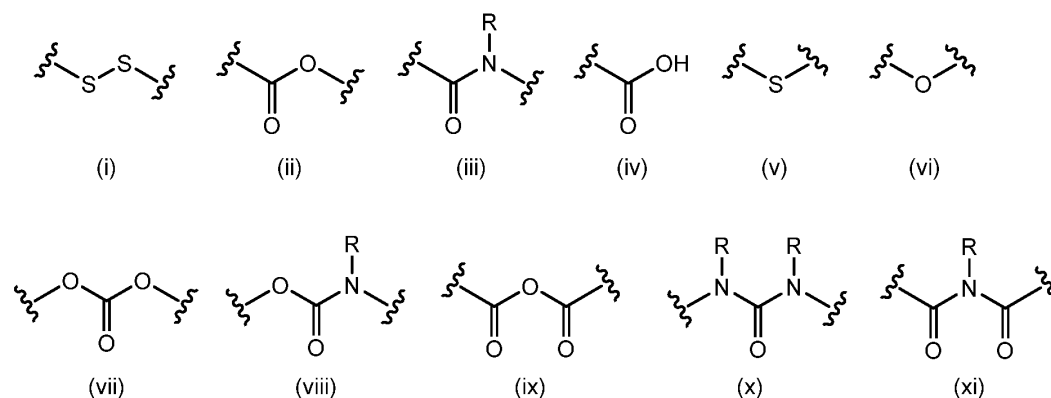
- 5- The process according to anyone of the preceding claims, wherein the amino compound A is chosen in the group consisting of xylylene diamine, 1,2-diaminocyclohexane, 1,4-diaminocyclohexane, L-lysine, L-Lysine ethyl ester, polyetheramines, ethylene diamine, diethylene triamine, spermine, spermidine, polyamidoamine (PAMAM), guanidine carbonate, chitosan, tris-(2-aminoethyl)amine, 3-aminopropyltriethoxysilane, L-arginine, 1,4 diaminobutane, 2,2 Dimethyl-1,3-propanediamine, 1,3-Diaminopentane, 1,2 diaminopropane, cystamine, cystamine hydrochloride, cystine, cystine hydrochloride, cystine dialkyl ester, cystine dialkyl ester hydrochloride, 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 and mixtures thereof.
- 6- The process according to anyone of the preceding claims, wherein at least one amino-compound B is added in the water phase before the formation of the oil-in-water emulsion and/or in the oil-in water emulsion obtained after step b).
- 7- The process according to claim 6, wherein the amino compound B is an amino-acid, preferably chosen in the group consisting of L-Lysine, L-Arginine, L-Leucine, L-Histidine, L-Tryptophane, L-Serin, L-Glutamine, L-Threonine and mixtures thereof.
- 8- The process according to anyone of the preceding claims, wherein the water phase comprises a base, preferably chosen in the group consisting of sodium carbonate, sodium bicarbonate, sodium hydroxide, guanidine carbonate, triethanolamine, and mixtures thereof.
- 9- The process according to anyone of the preceding claims, wherein the acyl chloride is a compound of formula (I)



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

preferably between 1 and 4, and

wherein X is an (n+1)-valent C₂ to C₄₅ hydrocarbon group optionally comprising at least one group selected from (i) to (xi)



wherein R is a hydrogen atom or an alkyl group such as a methyl or an ethyl group, preferably a hydrogen atom.

10- A polyamide-based core-shell microcapsule slurry obtainable by the process according to anyone of the preceding claims.

11- A polyamide-based core-shell microcapsule comprising:

- a core, preferably an oil-based core, comprising a hydrophobic material, preferably a perfume, and
- a polyamide-based shell comprising a reaction product of:
 - an acyl chloride,
 - an amino compound A,
 - optionally an amino compound B,
 - a protein,

wherein the shell has a rupture stress less than 10 MPa.

12- The polyamide-based core-shell microcapsule according to claim 11, wherein the polyamide-based shell comprising a reaction product of:

- an acyl chloride,
- an amino compound A,
- optionally an amino compound B,
- a protein,

5 in the presence of a cross-linker.

13- A perfuming composition comprising

- (i) Perfume microcapsule as defined in claims 11-12, wherein the hydrophobic material comprises a perfume,
- 10 (ii) At least one ingredient selected from the group consisting of a perfumery carrier and a perfumery base
- (iii) Optionally at least one perfumery adjuvant.

14- A consumer product comprising:

- 15 - a personal care active base, and
- microcapsules as defined in claims 11-12 or the perfuming composition as defined in claim 13,

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

20 **15-** A consumer product comprising:

- a home care or a fabric care active base, and
- microcapsules as defined in claims 11-12 or the perfuming composition as defined in claim 13,

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

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