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(54) Title: SOLID COMPOSITION IN THE FORM OF WATER-IN-OIL EMULSION

(57) Abstract: It relates to a solid composition in form of a water-in-oil emulsion comprising: (i) at least one silicone oil; (ii) at least two waxes having a melting point of greater than or equal to 65°C including polyethylene wax and jojoba ester wax; (iii) at least 5 wt. % of pigment, relative to the total weight of the composition; and (iv) no less than 40 wt. % of an aqueous phase, relative to the total weight of the composition. It also relates to a cosmetic method for caring for and/or making up the skin.

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SOLID COMPOSITION IN THE FORM OF WATER-IN-OIL EMULSION

TECHNICAL FIELD

The present invention relates to a composition for making up and/or caring for the skin. Particularly, the present invention relates to a solid composition in the form of water-in-oil emulsion. The present invention also relates to a cosmetic method for making up and/or caring for the skin.

BACKGROUND ART

Foundation compositions are commonly used to contribute to an attractive color of the skin, in particular the face, and/or conceal imperfections of the skin, such as red blotches or blemishes. Conventional foundations exist in fluid or solid form, and each possesses its own advantages due to its form.

WO 2012/145862 discloses fluid foundation compositions in the form of a water-in-silicone emulsion, comprising a fatty phase, an aqueous phase and at least one organopolysiloxane elastomer, at least one dimethicone copolyol, and at least one C₈-C₂₂ alkyl dimethicone. The fatty phase comprises at least one volatile oil and/or at least one nonvolatile oil and optionally at least one wax.

However, the foundations of the prior art, due to their fluid form, are found not easy to carry with and apply on the skin.

In general, consumers are attracted by a cosmetic composition, especially a foundation, which is easy to use, provides good makeup finish and brings a good sensory.

By “easy to use” we intend to mean the composition is easy to carry with and to apply on the skin.

By “good makeup finish” we intend to mean the composition has good spreadability.

By “good sensory” we intend to mean a fresh and watery sensation.

Efforts have been made to develop compositions with these properties.

Besides, the stability is still to be improved.

Therefore, there is a need for developing solid compositions which are easy to carry with and to apply on the skin, with the fingers or an applicator by the users.

There is also a need for developing a solid composition having good spreadability when applied on the skin and more particularly on the face and/or neck.

There is also a need for developing a solid composition that, when applied on the skin and more particularly on the face and/or neck, providing a good sensory, i.e.,

fresh and watery sensation, while presenting a good stability over time and temperature variations.

SUMMARY OF THE INVENTION

The present invention aims to provide a solid composition, which is easy to carry with and to apply on the skin, has a good stability over time and temperature variations, and having good spreadability and providing good sensory when applied on the skin.

Thus, according to a first aspect, the present invention provides a solid composition in the form of a water-in-oil emulsion comprising:

- (i) at least one silicone oil;
- (ii) at least two waxes having a melting point of greater than or equal to 65°C including polyethylene wax and jojoba ester wax;
- (iii) at least 5 wt.% of pigment, relative to the total weight of the composition; and
- (iv) no less than 40 wt.% of an aqueous phase, relative to the total weight of the composition.

Surprisingly, the inventors have discovered that the composition according to the present invention is easy to carry with and to apply on the skin, has a good stability over time and temperature variations, and has good spreadability and provides good sensory when applied on the skin.

More surprisingly, it has been observed that, the combination of at least two waxes having a melting point of greater than or equal to 65°C including polyethylene wax and jojoba ester wax, silicone oil and no less than 40 wt.% of aqueous phase provides a good balance of sensory, spreadability and stability.

According to a second aspect, the present invention provides a cosmetic method for caring for and/or making up the skin, comprising application of a composition of the present invention on the skin.

Other subjects and characteristics, aspects and advantages of the present invention will emerge even more clearly on reading the description and the examples that follows.

DETAILED DESCRIPTION OF THE INVENTION

According to the first aspect, the present invention provides a solid composition in the form of a water-in-oil emulsion comprising:

- (i) at least one silicone oil;

(ii) at least two wax having a melting point of greater than or equal to 65°C including polyethylene wax and jojoba ester wax; and

(iii) at least 5 wt.% of pigment, relative to the total weight of the composition;

(iv) no less than 40 wt.% of an aqueous phase, relative to the total weight of the composition.

In that which follows and unless otherwise indicated, the limits of a range of values are included within this range, in particular in the expressions "of between" and "from ... to ...".

For the purposes of the present invention, the term "skin" is intended to mean human skin and cover the face, the neck and the lips.

Moreover, the expression "at least one" used in the present description is equivalent to the expression "one or more".

Throughout the instant application, the term "comprising" is to be interpreted as encompassing all specifically mentioned features as well optional, additional, unspecified ones.

As used herein, the use of the term "comprising" also discloses the embodiment wherein no features other than the specifically mentioned features are present (*i.e.* "consisting of").

For the purpose of the present invention, "*stable*" refers to a composition which, after having been placed in an oven at 45°C, 37°C, and 4°C for two months, does not, when returned to ambient temperature, exhibit grains perceptible to the touch when a fine layer of the composition is sheared between the fingers.

The composition according to the present invention is a foundation preferably under the form of compact emulsion or cast solid products, for example, a stick foundation.

For the purpose of the present invention, the term "solid" means the composition does not flow under its own weight at room temperature (25°C).

The solid nature of the compositions can be determined by determining the hardness of the compositions. The hardness of a composition may, for example, be expressed by force in gram (g). Solid compositions of the present invention may, for example, have a hardness ranging from 20 g to 200 g, such as from 20 g to 90 g, from 20 g to 80 g and further such as from 20 g to 60 g, from 20 g to 40 g.

The hardness of a solid composition is measured according to the following protocol.

The composition the hardness of which is to be determined is stored at 20°C for 24 hours before measuring the hardness.

This hardness is measured according to a method of penetrating a probe into the composition and in particular using a texture analyzer (for example TA-XT PLUS from Stable Micro System) equipped with a probe with a diameter of 3.26 mm. The hardness measurement is carried out at 20°C. at the center of 5 samples of the composition. The cylinder is introduced into each sample of composition at a pre-speed of 1.5 mm/s and then at a speed of 2 mm/s and finally at a post-speed of 10 mm/s, the total displacement being 5 mm. The recorded hardness value is that of the maximum peak observed.

For a measurement at a different temperature, the stick is stored for 24 hours at this new temperature before the measurement.

Silicone oil

According to the first aspect, the composition of the present invention comprises at least one silicone oil.

The term “silicone oil” means an oil comprising at least one silicon atom, and especially comprising Si—O groups. The silicone oil(s) may be volatile or non-volatile.

As used herein, by “oils” or “oil” it means compounds having a melting point of less than about 30° C and generally insoluble in water and including a hydrophobic moiety. The hydrophobic moiety may include linear, cyclic, aromatic, saturated or unsaturated groups. The hydrophobic compound is in certain embodiments not amphiphilic and, as such, in this embodiment does not include hydrophilic moieties, such as anionic, cationic, zwitterionic, or nonionic groups, that are polar, including sulfate, sulfonate, carboxylate, phosphate, phosphonate, ammonium, including mono-, di-, and trialkylammonium species, pyridinium, imidazolinium, amidinium, poly(ethyleneiminium), ammonioalkylsulfonate, ammonioalkylcarboxylate, amphotoacetate, and poly(ethyleneoxy)sulfonyl moieties. In certain embodiments, the oil does not include hydroxyl moieties.

The term “volatile” refers to a compound that can evaporate on contact with the skin in less than one hour, at room temperature and atmospheric pressure. The volatile oil is a volatile cosmetic oil, which is liquid at room temperature, especially having a non-zero vapour pressure, at room temperature and atmospheric pressure, in particular having a vapour pressure ranging from 0.13 Pa to 40 000 Pa (10^{-3} to 300 mmHg), preferably ranging from 1.3 Pa to 13 000 Pa (0.01 to 100 mmHg) and preferentially ranging from 1.3 Pa to 1300 Pa (0.01 to 10 mmHg).

In certain embodiments, the volatile silicone oils that may be mentioned include cyclopolydimethylsiloxanes (INCI name: cyclomethicone), such as cyclopentasiloxane, cyclohexasiloxane, octylmethylcyclotetrasiloxane, decamethylcyclopentasiloxane, dodecamethylcyclohexasiloxane; linear silicones such as heptamethylhexyltrisiloxane, heptamethyloctyltrisiloxane, hexamethyldisiloxane, octamethyltrisiloxane, decamethyltetrasiloxane and dodecamethylpentasiloxane.

In certain embodiments, the silicone oil may be selected from linear or cyclic silicone oils, such as linear or cyclic polydimethylsiloxanes (PDMSs), which are liquid or pasty at room temperature, especially cyclopolydimethylsiloxanes (cyclomethicones) such as cyclohexasiloxane; polymethylsiloxanes comprising alkyl, alkoxy or phenyl groups, which are pendent or at the end of a silicone chain, these groups containing from 2 to 24 carbon atoms, phenyl silicones, for instance phenyl trimethicones, phenyl dimethicones, phenyltrimethylsiloxydiphenyl-siloxanes, diphenyl dimethicones, diphenylmethyl-diphenyltrisiloxanes or 2-phenylethyl trimethylsiloxysilicates, and polymethylphenylsiloxanes, combinations thereof.

Preferably, the silicone oil is selected from polydimethylsiloxane.

Advantageously, the silicone oil is present in an amount ranging from 20 wt.% to 45 wt.%, preferably from 20 wt.% to 40 wt.%, more preferably from 25 wt.% to 35 wt.%, relative to the total amount of the composition.

Waxes

According to the first aspect, the composition of the present invention comprises at least two waxes having a melting point of greater than or equal to 65°C including polyethylene wax and jojoba ester wax and at least one solid fatty substance.

The wax under consideration in the context of the present invention is generally a lipophilic compound that is solid at room temperature (25°C), with a solid/liquid reversible change of state.

According to the present invention, the composition comprises a combination of polyethylene wax and jojoba ester wax, both of them having a melting point of greater than or equal to 65°C.

Polyethylene wax

The polyethylene wax with a melting point of greater than or equal to 65°C used according to the invention is an ethylene homopolymer or a copolymer of

ethylene and of another copolymerizable monomer corresponding to the following formula (I):



wherein

R represents a linear or branched alkyl chain which can be interrupted by mono-or polyoxyalkylene units, an aryl or aralkyl radical or $-\text{CH}_2\text{COOH}$ or $-\text{CH}_2\text{CH}_2\text{OH}$ radical.

The alkyl radicals more particularly denote the methyl, ethyl, propyl, isopropyl, decyl, dodecyl and octadecyl radicals.

The mono- or polyoxyalkylene units preferably denote mono- or polyoxyethylene groups or mono-or polyoxypropylene groups.

The aryl radical is preferably a phenyl or tolyl radical.

The aralkyl radical is, for example, a benzyl or phenethyl radical.

The weight-average molar mass of the polyethylene wax with a high melting point according to the invention is preferably between approximately 400 and 1000, more particularly between approximately 400 and 700 and is preferably about 500.

According to a preferred embodiment, the wax as defined above is selected from ethylene homopolymers, copolymers of ethylene and of propylene, copolymers of ethylene and of maleic anhydride or acid, or oxidized or ethoxylated polyethylenes.

Mention may in particular be made, among the ethylene homopolymers which can be used according to the invention, of those sold under the names of Polywax 500, Polywax 655 and Polywax 1000 by the company Petrolite.

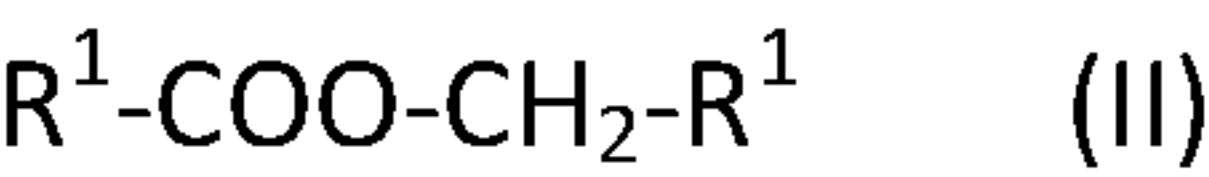
Mention may be made, among the ethylene copolymers which can be used according to the invention, of the copolymers of ethylene and of propylene sold under the names Petrolite® by the company Petrolite, the copolymers of ethylene and of maleic anhydride sold under the names Ceramer® by the company Petrolite, the oxidized polyethylenes sold under the names Unilin® and Unicid® by the company Petrolite, and the ethoxylated polyethylenes sold under the names Unithox® by the company Petrolite.

According to a particularly preferred embodiment of the invention, the polyethylene wax is an ethylene homopolymer wax. Mentions may be made of polyethylene which, for example, is sold under the tradename Performalene 500-L Polyethylene by the company New Phase Technologies.

Preferably, the polyethylene wax is present in an amount ranging from ranging from 2 wt.% to 10 wt.%, preferably from 2 wt.% to 8 wt.%, more preferably from 2 wt.% to 5 wt.%, relative to the total amount of the composition.

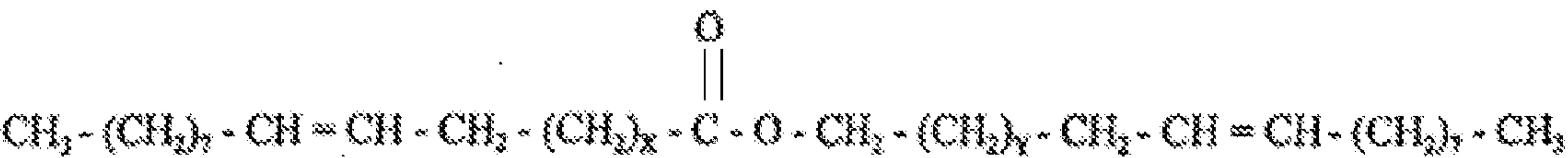
Jojoba ester wax

Jojoba ester wax with a melting point of greater than or equal to 65°C used according to the invention can be saturated form of jojoba esters, which can be fully hydrogenated jojoba ester of formula (II):



wherein R¹ comprises CH₃-(CH₂)_y-, y is 16, 18, 20 or 22.

The jojoba ester wax can be obtained by hydrogenation of jojoba wax ester (jojoba oil) of the following formula:



wherein x and y are 6, 8, 10 or 12.

The jojoba wax esters are composed of straight chain monounsaturated fatty alcohols and monounsaturated fatty acids. The single double bond is located in the middle (n-9 position), counting from the terminal methyl group, (-CH₃) of respective fatty acid or alcohol chain. Such wax esters are composed of fatty alcohols and fatty acids of even number of carbon atoms, primarily 20 and 22 carbons. The resulting esters have chain lengths of 38, 40, 42 and 44 with a small amount of esters of 36- and 46-carbon atoms being present. The typical composition of the wax esters is set out below.

Wax esters		
(X, Y)	Wax Ester Chain Length	Typical % (area by GC)
(6, 6)	36	1
(6, 8) (8, 6)	38	8
(6, 10) (8, 8) (10, 6)	40	39
(10, 8) (8, 10)	42	38
(10, 10)	44	13
(12, 10) (10, 12)	46	1

The jojoba wax ester can be derived from the seed of the jojoba plant (*Simmondsia chinensis*).

As examples of Jojoba ester wax with a melting point of greater than or equal to 65°C, mention can be made to Floraesters® 70 sold by International Flora Technologies Ltd; Jojoba Esters-70 sold by the company vantage.

Preferably, the jojoba ester wax is present in an amount ranging from ranging from 1 wt.% to 10 wt.%, preferably from 1.5 wt.% to 8 wt.%, more preferably from 2 wt.% to 5 wt.%, relative to the total amount of the composition.

Additional wax

In addition to polyethylene wax and jojoba ester wax mentioned above, the composition according to the present invention can comprise an additional wax.

As illustrations of waxes that are suitable for the present invention, mention may be made especially of hydrocarbon-based waxes, for instance beeswax, lanolin wax, Chinese insect waxes, rice bran wax, carnauba wax, candelilla wax, ouricury wax, esparto grass wax, berry wax, shellac wax, Japan wax and sumach wax; montan wax, orange wax and lemon wax, microcrystalline waxes, paraffins, and ozokerite; the waxes obtained by Fischer-Tropsch synthesis and waxy copolymers, and also esters thereof, fatty acids or esters obtained by catalytic hydrogenation of animal or plant oils containing linear or branched C₈-C₃₂ fatty chains, preferably C₁₆ to C₁₈ chains, silicone waxes and fluoro waxes, or a mixture thereof.

Mention may also be made of fatty acids obtained by catalytic hydrogenation of animal or plant oils containing linear or branched C₈-C₃₂ fatty chains, preferably C₁₆ to C₁₈ chains. Among these compounds, mention may be made especially of stearic acid, palmitic acid, or a mixture thereof. The fatty acids used in the present invention are commercially available under the trade names, for example, AEC Stearic Acid sold by A & E Connock (Perfumery & Cosmetics) Ltd., Emersol sold by Emery Oleochemical LLC, Palmitic Acid PC sold by Protameen Chemicals, Inc.

As for esters obtained by catalytic hydrogenation of animal or plant oils, mention may be made to the waxes obtained by hydrogenation of castor oil esterified with cetyl alcohol, sold under the names Phytowax ricin 16L64[®] and 22L73[®] by the company Sophim, may also be used. Such waxes are described in patent application FR-A-2 792 190.

The waxes obtained by hydrogenation of olive oil esterified with C₁₂ to C₁₈ chain fatty alcohols such as those sold by the company SOPHIM under the brand names Phytowax Olive 12L44, 14L48, 16L55 and 18L57, are also convenient.

A wax that may be used is a C₂₀-C₄₀ alkyl (hydroxystearyloxy) stearate (the alkyl group containing from 20 to 40 carbon atoms), alone or as a mixture. Such a wax is especially sold under the names Kester Wax K 82 P[®], Hydroxypolyester K 82 P[®] and Kester Wax K 80 P[®] by the company Koster Keunen.

Mentions may be made of candelilla (*Euphorbia cerifera* wax), such as the one sold under the reference "NC 1630" sold by Cera Rica Noda,

Mentions may also be made of ozokerite, such as the commercial reference "Ozokerite Wax SP 1020 P" sold by Strahl & Pitsch,

If presents, the additional waxes can be present in an amount ranging from 0.1% to 10% by weight, preferably of 0.5% to 5% by weight and especially of 1% to 4% by weight, relative to the total weight of the composition.

Pigments

According to the first aspect, the composition of the present invention comprises at least 5.0 wt.% of pigment, relative to the total weight of the composition.

"Pigments" are white or coloured, organic or inorganic, hydrophilic or hydrophobic, non-interference particles which are insoluble in aqueous and non-aqueous media and are intended for colouring the composition.

Inorganic pigments can be used in the present invention include oxides or dioxides of titanium, zirconium or cerium, and also oxides of zinc, iron or chromium, mica, Prussian blue, manganese violet, ultramarine blue and chromium hydrate, and mixtures thereof.

The organic pigments can be used in the present invention include D & C pigments, lakes based on cochineal carmine, and on barium, strontium, calcium and aluminium, or else the diketopyrrolopyrroles (DPP) which are described in documents EP-A-542 669, EP-A-787 730, EP-A-787 731 and WO-A-96/08537.

Advantageously, the pigments can be used in the present invention is a hydrophobic pigment, treated or untreated.

Use will be made of inorganic pigments, selected more particularly from titanium oxides and iron oxides (especially yellow, black and red), mica, untreated or treated with at least one hydrophobic agent.

The pigments can be used in the present invention are preferably wholly or partly surface-treated with a hydrophobic agent, more particularly with a fluoro, fatty acid or amino acid, or silicone compound, or a mixture thereof.

By way of example, the hydrophobic treatment agent may be selected from fatty acids such as stearic acid; metal soaps such as aluminium dimyristate and the aluminium salt of hydrogenated tallow glutamate; perfluoroalkyl phosphates and polyhexafluoropropylene oxides; perfluoropolyethers; amino acids; N-acylamino acids or salts thereof; lecithin, isopropyl trisostearyl titanate, isostearyl sebacate,

and silicone compounds such as dimethicones or polydimethylsiloxanes, and mixtures thereof.

The hydrophobic treatment agent is preferably selected from perfluoroalkyl phosphates, polyhexafluoropropylene oxides, perfluoropolyethers, amino acids, N-acylamino acids or salts thereof, isopropyl trisostearyl titanate, and mixtures thereof.

More preferably, the hydrophobic agent is selected from perfluoroalkyl phosphates, N-acylamino acids or salts thereof, isopropyl trisostearyl titanate, and mixtures thereof.

The surface-treated pigments may be prepared according to chemical, electronic, chemomechanical or mechanical surface treatment techniques that are well known to the skilled person. It is also possible to use commercial products.

The surface agent may be absorbed or adsorbed on the pigments by solvent evaporation, chemical reaction and creation of a covalent bond.

According to one version, the surface treatment comprises a coating of the pigments.

The coating may represent from 0.1 to 10% by weight and more particularly from 1% to 5% by weight, of the total weight of the coated pigments.

Coating may be carried out, for example, by adsorption of a liquid surface agent on the surface of the pigments, by simple mixing with stirring of the pigments and of said surface agent, optionally under hot conditions, prior to the incorporation of the pigments into the other ingredients of the care or makeup composition.

Coating may be carried out, for example, by chemical reaction of a surface agent with the surface of the pigments and creation of a covalent bond between the surface agent and the pigments. This method is described especially in patent US 4,578,266.

The hydrophobic treatment agent may be selected from fatty acids, such as stearic acid; metal soaps, such as aluminium dimyristate and the aluminium salt of hydrogenated tallow glutamate; amino acids; N-acylamino acids or salts thereof; lecithin, isopropyl trisostearyltitanate (or alternatively called ITT), and mixtures thereof.

The N-acylamino acids may comprise an acyl group having from 8 to 22 carbon atoms, such as, for example, a 2-ethylhexanoyl, caproyl, lauroyl, myristoyl, palmitoyl, stearyl or cocoyl group. The salts of these compounds may be the aluminium, magnesium, calcium, zirconium, zinc, sodium or potassium salts. The amino acid may be, for example, lysine, glutamic acid or alanine.

The fatty acids in the present invention are more particularly acids with hydrocarbon chains having from 1 to 30 carbon atoms, preferably having from 5 to 18 carbon atoms. The hydrocarbon chain may be saturated, monounsaturated or polyunsaturated.

Examples of pigments coated with fatty acids include those containing the disodium stearylglutamate/aluminium hydroxide pairing, these being sold in particular under the trade name NAI-TAO-77891, NAI-C33-8073-10, NAI-C33-8075, NAI-C47-051-10, NAI-C33-115, NAI-C33-134, NAI-C33-8001-10, NAI-C33-7001-10, NAI-C33-9001-10 from the company Miyoshi Kasei.

Examples of pigments treated with isopropyltitanium triisostearate (ITT) include those sold under the trade name BWBO-12 (Iron oxide CI77499 and isopropyl titanium triisostearate), BWYO-12 (Iron oxide CI77492 and isopropyl titanium triisostearate) and BWRO-12 (Iron oxide CI77491 and isopropyl titanium triisostearate) by the company Kobo.

Preferably, pigments coated with fatty acids, such as those containing disodium stearylglutamate/aluminium hydroxide pairing are used in the present invention.

Preferably, the pigment of the present invention is selected from mica, titanium oxides and iron oxides, untreated or coated with the disodium stearylglutamate/aluminium hydroxide pairing, or a mixture thereof.

Advantageously, the pigment is present in an amount up to 18 wt.%, relative to the total weight of the composition.

Aqueous phase

The composition according to the present invention comprises an aqueous phase.

The aqueous phase is present in an amount equal or above 40% by weight, in particular from 40% to 50%, especially between 40% and 45 % by weight, relative to the total weight of the composition.

In one embodiment, this aqueous phase can comprise alcohol(s), in particular polyethylene glycols having from 6 to 80 ethylene oxide units; polyols, such as propylene glycol, isoprene glycol, butylene glycol, pentylene glycol, glycerin, maltitol, sorbitol, dipropylene glycol or diethylene glycol, glycol ethers, such as mono-, di- or tripropylene glycol or mono-, di- or triethylene glycol (C_1 - C_4) alkyl ether, and any mixture thereof.

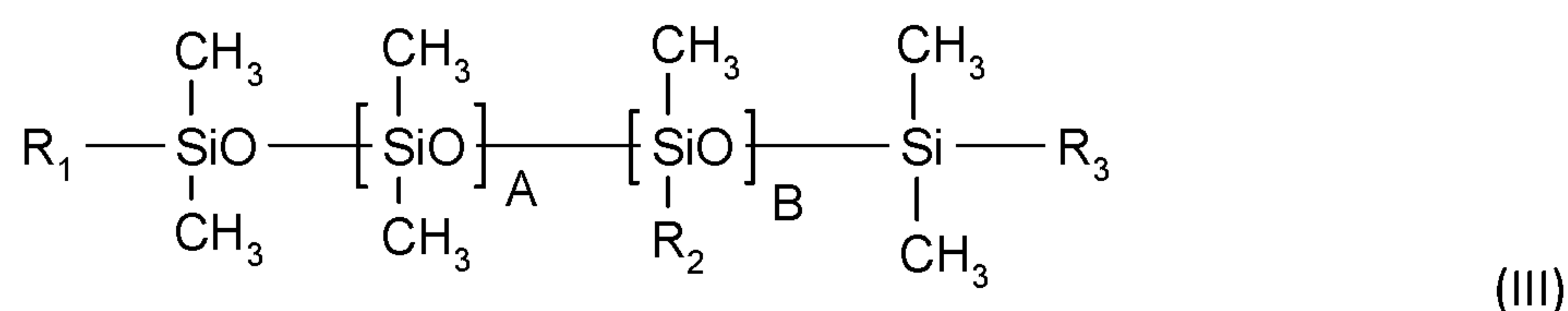
In one preferred embodiment, the aqueous phase of the present invention comprises at least one polyol. Preferably, the polyol is selected from propylene glycol, dipropylene glycol, pentylene glycol, glycerin and any mixture thereof.

The aqueous phase can additionally comprise stabilizing agents such as sodium chloride, magnesium dichloride, magnesium sulphate, preferably sodium chloride.

Oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane

According to a preferred embodiment, the composition according to the present invention comprises at least one oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane as an emulsifier.

Use may be made, as oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane, of those corresponding to the following formula (III):



in which:

R_1 , R_2 and R_3 represent, independently of one another, a C_1 - C_6 alkyl radical or a $-(\text{CH}_2)_x - (\text{OCH}_2\text{CH}_2)_y - (\text{OCH}_2\text{CH}_2\text{CH}_2)_z - \text{OR}_4$ radical, at least one R_1 , R_2 or R_3 radical not being an alkyl radical; R_4 being a hydrogen, a C_1 - C_3 alkyl radical or a C_2 - C_4 acyl radical;

A is an integer ranging from 0 to 200;

B is an integer ranging from 0 to 50; provided that A and B are not equal to zero at the same time;

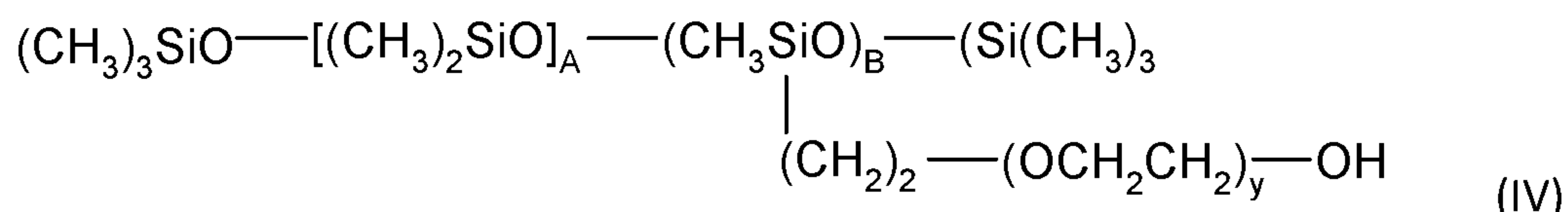
x is an integer ranging from 1 to 6;

y is an integer ranging from 1 to 30;

z is an integer ranging from 0 to 5.

According to a preferred embodiment of the present invention, in the compound of formula (III), $\text{R}_1 = \text{R}_3 = \text{methyl radical}$, x is an integer ranging from 2 to 6 and y is an integer ranging from 4 to 30, R_4 is in particular hydrogen.

Mention may be made, as examples of compounds of formula (II), of the compounds of formula (IV):



in which A is an integer ranging from 20 to 105, B is an integer ranging from 2 to 10 and y is an integer ranging from 5 to 20.

Mention may also be made, as examples of compounds of formula (II), of the compounds of formula (V):



in which A' and y are integers ranging from 5 to 20, preferably from 8 to 20.

As Examples of oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane, mention can be made to PEG-7 dimethicone, PEG-8 dimethicone, PEG-9 dimethicone, PEG-10 dimethicone, PEG-12 dimethicone, PEG-14 dimethicone, PEG-17 dimethicone and mixtures thereof.

Use may be made, as oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane, of those sold under the names DC 5329, DC 7439-146, DC 2-5695 and Q4-3667 by Dow Corning; and KF-6013, KF-6015, KF-6016, KF-6017 and KF-6028 by Shin-Etsu.

In a preferred embodiment, the composition of the present invention comprises as oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane one of those sold under the names KF-6013, KF-6015, KF-6016, KF-6017 and KF-6028 by Shin-Etsu, and more particularly KF-6017. The INCI name of KF-6017 is PEG-10 dimethicone.

Preferably, the composition according to the present invention may comprise the oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane in an amount ranging from 0.1% to 2% by weight, preferably from 0.2% to 1.5% by weight, and preferentially from 0.5% to 1% by weight, relative to the total weight of the composition.

In a more preferred embodiment, the compositions of the present invention comprise PEG-10 dimethicone as oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane, in an amount ranging from 0.1 % to 2% by weight, preferably from 0.2% to 1.5% by weight, and more preferably from 0.5% to 1% by weight, relative to the total weight of the composition.

C₈-C₂₂ alkyl dimethicone copolyol

According to a preferred embodiment, the composition of the present invention comprises at least one C₈-C₂₂ alkyl dimethicone copolyol as an emulsifier.

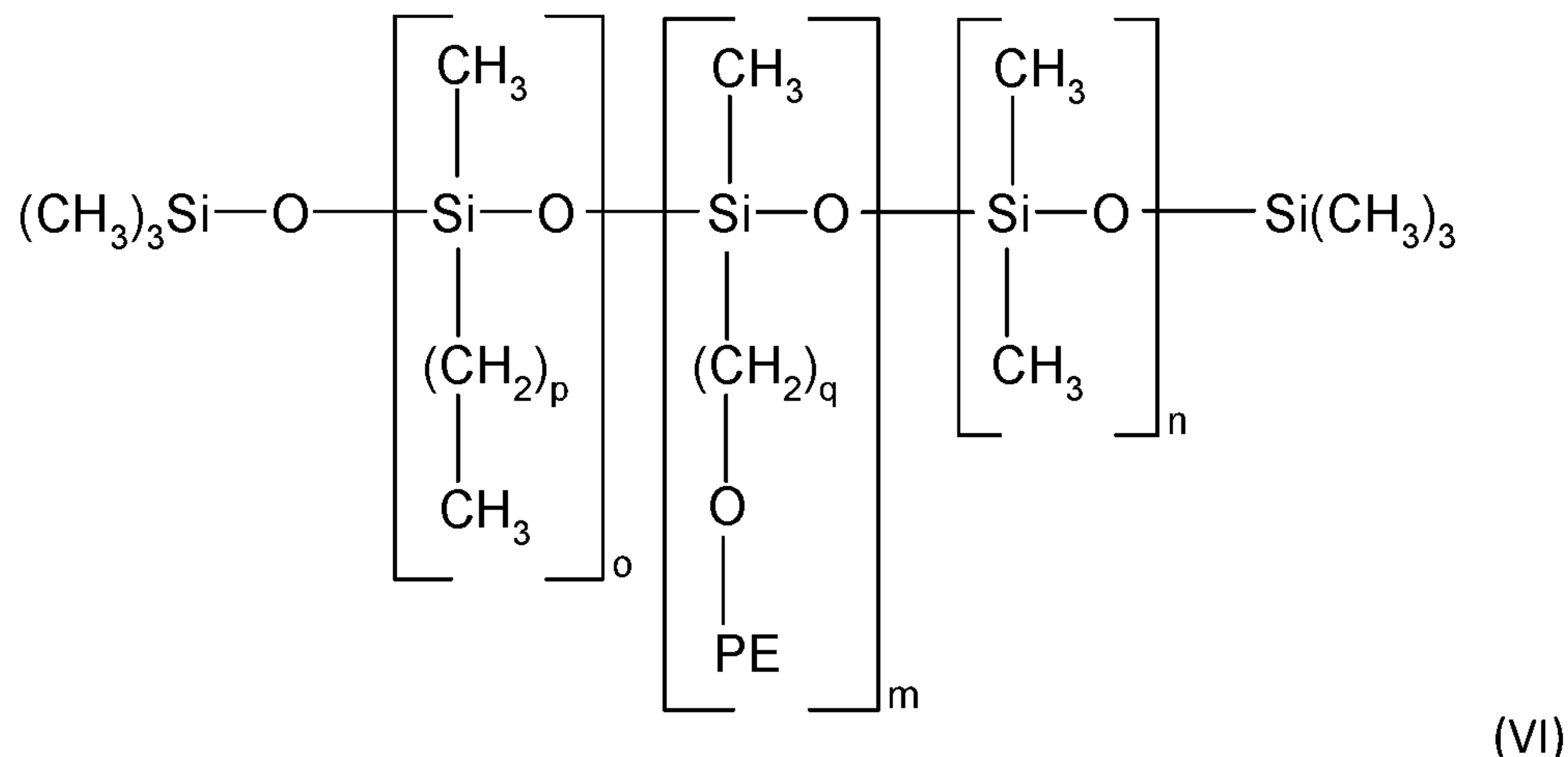
For the purpose of the present invention, C₈-C₂₂ alkyl dimethicone copolyol is different from oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane as defined above.

Advantageously, the presence of C₈-C₂₂ alkyl dimethicone copolyol renders the compositions according to the present invention a good stability at ambient

temperature (25°C). The composition according to the present invention also exhibits a good stability at 37°C or 45°C, in particular after storing for 2 months.

This C₈-C₂₂ alkyl dimethicone copolyol of the present invention is more particularly an oxypropylenated and/or oxyethylenated polymethyl (C₈-C₂₂) alkyl dimethyl methyl siloxane.

The C₈-C₂₂ alkyl dimethicone copolyol is advantageously a compound of the following formula (VI):



wherein:

- PE represents from group $(-\text{C}_2\text{H}_4\text{O})_x(-\text{C}_3\text{H}_6\text{O})_y-\text{R}$, wherein R is selected from a hydrogen atom and an alkyl radical comprising from 1 to 4 carbon atoms, x is an integer ranging from 0 to 100, and y is an integer ranging from 0 to 80, provided that x and y are not simultaneously equal to 0; and

- m is an integer ranging from 1 to 40, n is an integer ranging from 10 to 200, o is an integer ranging from 1 to 100, p is an integer ranging from 7 to 21, and q is an integer ranging from 0 to 4.

Preferably, R is a hydrogen atom, m is an integer ranging from 1 to 10, n is an integer ranging from 10 to 100, o is an integer ranging from 1 to 30, p is 15, and q is 3.

In a preferred embodiment, the at least one C₈-C₂₂ alkyl dimethicone copolyol of the present invention is selected from cetyl dimethicone copolyols such as the product marketed under the name Abil EM-90 by the company Goldschmidt.

The composition according to the present invention may comprise C₈-C₂₂ alkyl dimethicone copolyol in an amount ranging from 0.1% to 1.5% by weight, preferably from 0.1% to 1% by weight, and more particularly from 0.1% to 0.8% by weight, relative to the total weight of the composition.

Abil EM-90 is a cetyl PEG/PPG-10/1 dimethicone.

In a more preferred embodiment, the composition of the present invention comprises cetyl PEG/PPG-10/1 dimethicone as C₈-C₂₂ alkyl dimethicone copolyol, in an amount ranging from 0.1% to 1.5% by weight, preferably from 0.2% to 1% by weight, and more particularly from 0.2% to 0.8% by weight, relative to the total weight of the composition.

In another preferred embodiment, the composition of the present invention comprises PEG-10 dimethicone as dimethicone copolyol, and cetyl PEG/PPG-10/1 dimethicone as C₈-C₂₂ alkyl dimethicone copolyol.

In another preferred embodiment, the composition of the present invention comprises from 0.5% to 1% by weight of PEG-10 dimethicone, and 0.2% to 0.8% by weight of cetyl PEG/PPG-10/1 dimethicone, relative to the total weight of the composition.

Active ingredients

Optionally, the composition according to the present invention can further comprise active ingredients.

Mention may be made, as active ingredient which can be used in the composition of the present invention, for example, of moisturising agents, such as trehalose; vitamins, such as vitamin A (retinol), vitamin E (tocopherol), vitamin C (ascorbic acid), vitamin B5 (panthenol), vitamin B3 (niacinamide), the derivatives of these vitamins (in particular esters) and their mixtures; urea; caffeine; salicylic acid and its derivatives; alpha-hydroxy acids, such as lactic acid and glycolic acid, and their derivatives; retinoids, such as carotenoid and vitamin A derivatives; sunscreen; essential oils of mint, aloe vera or ginseng and their mixtures, emollients, such as hydrogenated polyisobutene, sensory adjusting agent, such as diphenyl dimethicone/vinyl diphenyl dimethicone/silsesquioxane crosspolymer.

In a preferred embodiment, the composition of the present invention can further comprise at least one active ingredient is ethyl menthane carboxamide.

In a preferred embodiment, the composition of the present invention can moreover comprise a chemical sunscreen such as any UV-A or UV-B screening agent which can be used in the cosmetics field. The sunscreens (or UV screening agents) can be selected from organic screening agents, physical screening agents and their mixtures, and can more particularly be selected from ethylhexyl methoxycinnamate, titanium dioxide or their mixture.

Additives

In a known way, the composition of the present invention can comprise one or more of the adjuvants usual in the cosmetic and dermatological fields: hydrophilic or lipophilic gelling and/or thickening agents, such as sodium hyaluronate and aluminum starch octenylsuccinate; fillers; sequestering agents; antioxidants; preservatives such as phenoxyethanol and chlorphenesin; basifying or acidifying agents; fragrances; emulsifiers, such as sorbitan sesquioleate and their mixtures.

The amounts of various adjuvants are those conventionally used in foundations.

Of course, a person skilled in the art will take care to choose the optional adjuvants added to the composition according to the present invention so that the advantageous properties intrinsically attached to the composition according to the present invention are not, or not substantially, detrimentally affected by the envisaged addition.

In a preferred embodiment, the present invention provides a solid cosmetic composition in form of a water-in-oil emulsion comprising, relative to the total weight of the composition:

(i) from 25 wt.% to 35 wt.% of at least one silicone oil selected from polydimethylsiloxane;

(ii) from 2 wt.% to 5 wt.% of ethylene homopolymer wax and from 2 wt.% to 5 wt.% of jojoba ester wax, both of them having a melt point having a melting point of greater than or equal to 65°C;

(iii) at least 5 wt.% of pigment selected from mica, titanium oxides and iron oxides, untreated or coated with the disodium stearylglutamate/aluminium hydroxide pairing, or a mixture thereof; and

(iv) no less than 40 wt.% of an aqueous phase including at least one polyol selected from propylene glycol, dipropylene glycol, pentylene glycol, glycerin and any mixture thereof.

Galenic form

The composition of the present invention is suitable to be used as a skin care, make up or cosmetic treatment product.

More particularly, the composition of the present invention is in the form of compact powder or cast solid product, for example, a stick foundation.

Method and use

The composition according to the present invention can be used for caring for/making up the skin and particularly the face and/or neck.

The composition according to the present invention can be applied on the skin, with the fingers or an applicator by the users.

Thus, according to another aspect, the present invention provides a cosmetic method for caring and/or making up the skin, comprising application of a composition of the present invention on the skin.

Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which the present invention pertains.

The examples that follow are given as non-limiting illustrations of the present invention.

Examples

Example 1

Stick Foundations in the form of water-in-oil emulsion of invention formula (Inv.1) and comparative formulas (Comp.) 1-2 were prepared (the contents are expressed as weight percentages of active material with regard to the total weight of each composition, unless otherwise indicated):

Phase	INCI Name (trade name and supplier)	Inv.1	Comp.1	Comp.2
		Wt.%		
A1	POLYETHYLENE(PERFORMALENE 500-L POLYETHYLENE from NEW PHASE TECHNOLOGIES)	3.50	-	7.00
	PARAFFIN(PARAFFINE PLASTIQUE 46/50 from AIGLON)	2.00	2.00	2.00
	JOJOBA ESTERS (JOJOBA ESTERS-70 from VANTAGE SPECIALTY CHEMICALS)	3.50	7.00	-
	CETYL PEG/PPG-10/1 DIMETHICONE (ABIL EM 90 from EVONIK GOLDSCHMIDT)	0.43	0.43	0.43
	PEG-10 DIMETHICONE (KF-6017 from SHIN ETSU)	0.86	0.86	0.86
	SORBITAN SESQUIOLEATE(SPAN 83-LQ-(MV) from CRODA)	0.21	0.21	0.21
	HYDROGENATED POLYISOBUTENE(PARLEAM from NOF CORPORATION)	3.50	3.50	3.50
	DIMETHICONE(KF-96L-2CS from SHIN ETSU)	QS 100	QS 100	QS 100
A2	DIMETHICONE(KF-96L-2CS from SHIN ETSU)	4.50	4.50	4.50
	TITANIUM DIOXIDE (and) DISODIUM STEAROYL GLUTAMATE (and) ALUMINUM HYDROXIDE (NAI-TAO-77891 from MIYOSHI KASEI)	11.7	11.7	11.7
	IRON OXIDES (and) DISODIUM STEAROYL GLUTAMATE (and) ALUMINUM HYDROXIDE (AMS YELLOW NO.602P from DAITO KASEI KOGYO)	1.16	1.16	1.16
	IRON OXIDES (and) DISODIUM STEAROYL GLUTAMATE (and) ALUMINUM HYDROXIDE (AMS RED NO.211P from DAITO KASEI KOGYO)	0.36	0.36	0.36
	IRON OXIDES (and) DISODIUM STEAROYL GLUTAMATE (and) ALUMINUM HYDROXIDE (AMS BLACK NO.710P from DAITO KASEI KOGYO)	0.27	0.27	0.27
A3	DIPHENYL DIMETHICONE/VINYL DIPHENYL DIMETHICONE/SILSESQUIOXANE CROSSPOLYMER (KSP 300 from SHIN ETSU)	1.00	1.00	1.00
	MICA (and) TITANIUM DIOXIDE(TIMICA TERRA WHITE MN4501 from BASF)	0.50	0.50	0.50
	ALUMINUM STARCH OCTENYLSUCCINATE (DRY FLO PLUS from AKZO NOBEL)	0.30	0.30	0.30
A4	DIMETHICONE(KF-96L-2CS from SHIN ETSU)	3.00	3.00	3.00
B	WATER	38	38	38
	PHENOXYETHANOL	0.70	0.70	0.70
	SODIUM CHLORIDE	0.70	0.70	0.70
	PENTYLENE GLYCOL	1.50	1.50	1.50
	CHLORPHENESIN	0.30	0.30	0.30
	SODIUM HYALURONATE	0.02	0.02	0.02

The stick foundation of comparative formula does not comprise polyethylene wax with a melting point of greater than or equal to 65°C.

The stick foundation of comparative formula does not comprise jojoba ester wax with a melting point of greater than or equal to 65°C.

The stick foundations with formulas listed above were prepared according to the following steps:

1. Ingredients of A1 were added into a main beaker, heated to 90°C and mixed until uniform;
2. Ingredients of A2 were ground until uniform and added into the main beaker, mixed for 5 minutes;
3. Ingredients of A3 were added into the main beaker and mixed for 5 minutes, then cooled to 80°C, thereto ingredients of A4 were added and mixed for 5 minutes;
4. Ingredients of B were premixed in a small beaker at 80°C until uniform, and transferred into the main beaker and homogenized for 10 minutes at 80°C.

Example 2

The stability and hardness of the stick foundations of invention formula 1 and comparative formulas 1-2, and the spreadability and skin sensory upon application of them were evaluated.

The stability of each stick foundation was evaluated at 45°C, 37°C, and 4°C for 8 weeks.

The hardness of each stick foundation was evaluated at room temperature with a TA-XT PLUS type texture analyzer from Stable Micro System as mentioned previously.

The spreadability of each stick foundation was evaluated with a MCR 301 type Rheometer from Anton Paar as follows.

A stick foundation to be tested was put on a plate and the value of shear stress at the cross point of G' (storage modulus) and G'' (loss modulus) was measured with PP25 probe. The lower value means the better spreadability. Generally, a shear stress of no more than 500 Pa indicates good spreadability.

The skin sensory (including fresh and watery sensation) of each stick foundation was scored by 10 sensorial experts based on the following standard and averaged:

- 5: very good;
- 4: basically good;
- 3: acceptable;
- 2: slightly poor and not acceptable;
- 1: poor, not acceptable.

The results of stability, hardness, spreadability and skin sensory were summarized in the following table.

		Inv. 1	Comp. 1	Comp. 2
stability	45°C	PASS	*	PASS
	37°C	PASS	*	PASS
	4°C	PASS	*	PASS
Hardness (g)		29.1	16.8	14.4
Spreadability (Pa)		162.9	15.7	59.2
Skin sensory		5	4	3

*not acceptable

It was observed that, the foundation of invention formula 1 was easily casted as a stick foundation, which enabled the consumers to use with either single hand or applicators for making up the skin. It was very convenient for the consumers to supplement the makeups of the skin during the day.

CLAIMS

1. Solid composition in form of a water-in-oil emulsion comprising:

- (i) at least one silicone oil;
- (ii) at least two waxes having a melting point of greater than or equal to 65°C including polyethylene wax and jojoba ester wax;
- (iii) at least 5 wt.% of pigment, relative to the total weight of the composition; and
- (iv) no less than 40 wt.% of an aqueous phase, relative to the total weight of the composition.

2. Composition according to claim 1, wherein the silicone oil is selected from linear or cyclic silicone oils, such as linear or cyclic polydimethylsiloxanes (PDMSs); polymethylsiloxanes comprising alkyl, alkoxy or phenyl groups, which are pendent or at the end of a silicone chain, these groups containing from 2 to 24 carbon atoms, phenyl silicones, for instance phenyl trimethicones, phenyl dimethicones, phenyltrimethylsiloxydiphenylsiloxanes, diphenyl dimethicones, diphenylmethyl-diphenyltrisiloxanes or 2-phenylethyl trimethylsiloxo silicates, and polymethylphenylsiloxanes, combinations thereof.

3. Composition according to claim 1 or 2, wherein the silicone oil is present in an amount ranging from 20 wt.% to 45 wt.%, preferably from 20 wt.% to 40 wt.%, more preferably 25 wt.% to 35 wt.%, relative to the total amount of the composition.

4. Composition according to any of claims 1 to 3, wherein the polyethylene wax is an ethylene homopolymer or a copolymer of ethylene and of another copolymerizable monomer corresponding to the following formula (I):

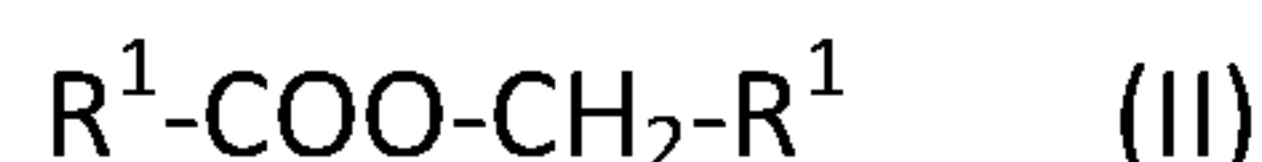


wherein

R represents a linear or branched alkyl chain which can be interrupted by mono-or polyoxyalkylene units, an aryl or aralkyl radical or -CH₂COOH or -CH₂CH₂OH radical.

5. Composition according to any of claims 1 to 4, wherein the polyethylene wax is present in an amount ranging from ranging from 2 wt.% to 10 wt.%, preferably from 2 wt.% to 8 wt.%, more preferably 2 wt.% to 5 wt.%, relative to the total amount of the composition.

6. Composition according to any of claims 1 to 5, wherein the jojoba ester wax is selected from fully hydrogenated jojoba ester of formula (II):



wherein R¹ comprises CH₃-(CH₂)_y-, y is 16, 18, 20 or 22.

7. Composition according to any of claims 1 to 6, wherein the jojoba ester wax is present in an amount ranging from ranging from 1 wt.% to 10 wt.%, preferably from 1.5 wt.% to 8 wt.%, more preferably 2 wt.% to 5 wt.%, relative to the total amount of the composition.

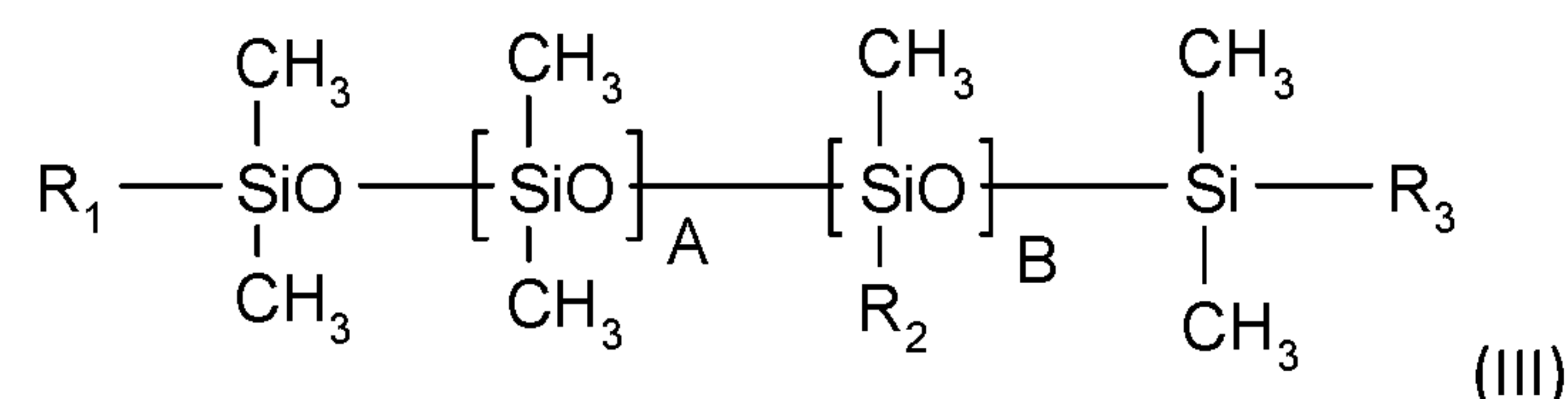
8. Composition according to any of claims 1 to 7, wherein the pigment is selected from oxides or dioxides of titanium, zirconium or cerium, and also oxides of zinc, iron or

chromium, mica, Prussian blue, manganese violet, ultramarine blue and chromium hydrate, and mixtures thereof.

9. Composition according to any of claims 1 to 8, wherein the pigment is present in an amount up to 18 wt.%, relative to the total weight of the composition.

10. Composition according to any of claims 1 to 9, wherein the aqueous phase comprises at least one polyol selected from propylene glycol, dipropylene glycol, pentylene glycol, glycerin and any mixture thereof.

11. Composition according to any of claims 1 to 10, further comprising at least one oxypropylenated and/or oxyethylenated polydimethyl(methyl)siloxane of formula (III):



in which:

R_1 , R_2 and R_3 represent, independently of one another, a C_1 - C_6 alkyl radical or a $-(\text{CH}_2)_x$ - $(\text{OCH}_2\text{CH}_2)_y$ - $(\text{OCH}_2\text{CH}_2\text{CH}_2)_z$ - OR_4 radical, at least one R_1 , R_2 or R_3 radical not being an alkyl radical; R_4 being a hydrogen, a C_1 - C_3 alkyl radical or a C_2 - C_4 acyl radical;

A is an integer ranging from 0 to 200;

B is an integer ranging from 0 to 50; provided that A and B are not equal to zero at the same time;

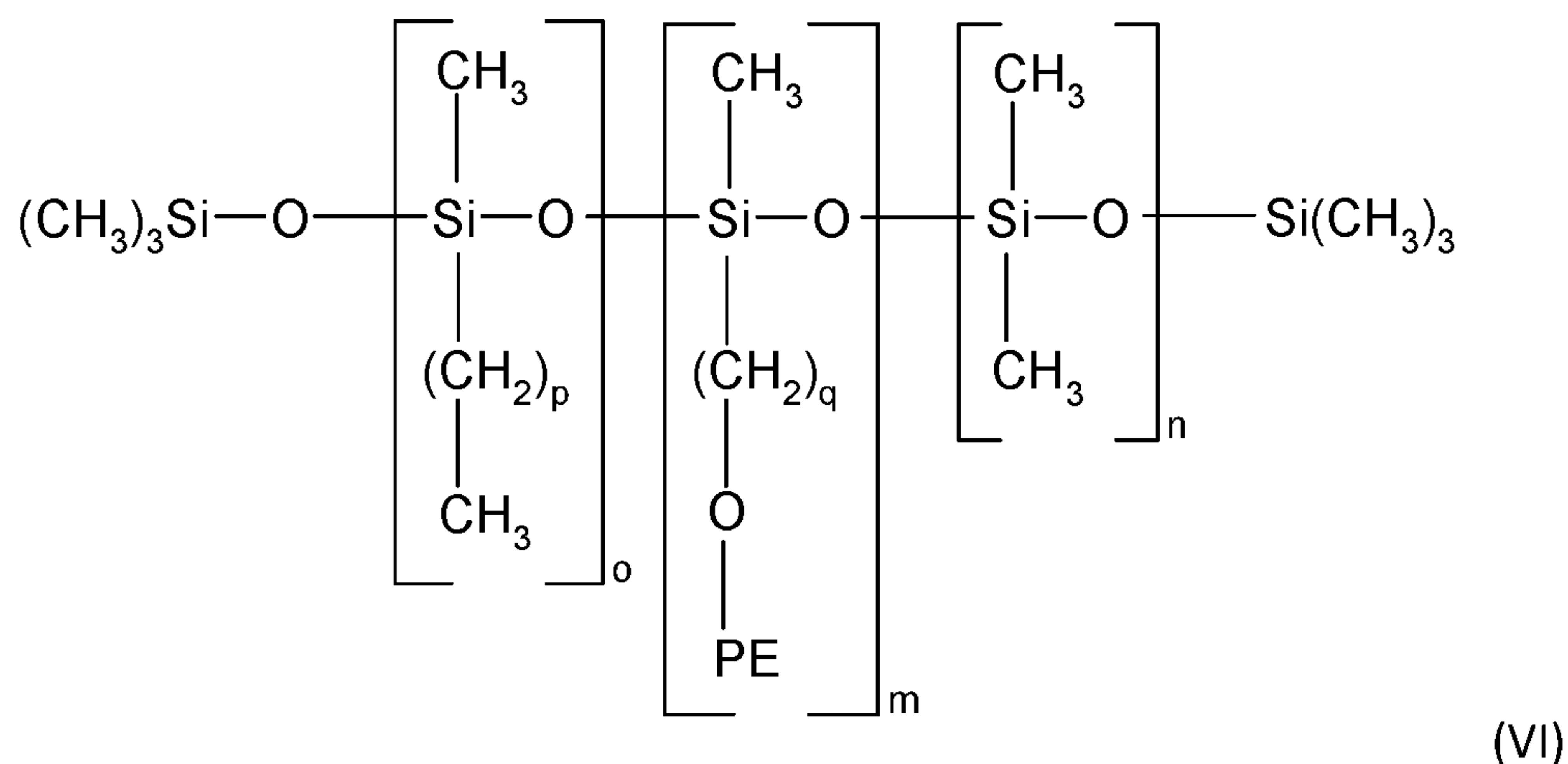
x is an integer ranging from 1 to 6;

y is an integer ranging from 1 to 30;

z is an integer ranging from 0 to 5;

preferably, $\text{R}_1 = \text{R}_3 =$ methyl radical, x is an integer ranging from 2 to 6 and y is an integer ranging from 4 to 30 in the dimethicone copolyol of formula (III).

12. Composition according to any of claims 1 to 11, further comprising at least one (C_8 - C_{22})alkyl dimethicone copolyol of formula (VI):



wherein:

- PE represents from group $(-\text{C}_2\text{H}_4\text{O})_x(-\text{C}_3\text{H}_6\text{O})_y-\text{R}$, wherein R is selected from a hydrogen atom and an alkyl radical comprising from 1 to 4 carbon atoms, x is an integer ranging from 0 to 100, and y is an integer ranging from 0 to 80, provided that x and y are not simultaneously equal to 0; and

- m is an integer ranging from 1 to 40, n is an integer ranging from 10 to 200, o is an integer ranging from 1 to 100, p is an integer ranging from 7 to 21, and q is an integer ranging from 0 to 4; preferably, the $(\text{C}_8\text{-C}_{22})$ alkyl dimethicone copolyol is cetyl PEG/PPG-10/1 dimethicone.

13. Composition according to claim 1 comprising, relative to the total weight of the composition:

(i) from 25 wt.% to 35 wt.% of at least one silicone oil selected from polydimethylsiloxane;

(ii) from 2 wt.% to 5 wt.% of ethylene homopolymer wax and from 2 wt.% to 5 wt.% of jojoba ester wax, both of which having a melt point of greater than or equal to 65°C;

(iii) at least 5 wt.% of pigment selected from mica, titanium oxides and iron oxides, untreated or coated with the disodium stearylglutamate/aluminium hydroxide pairing, or a mixture thereof; and

(iv) no less than 40 wt.% of an aqueous phase including at least one polyol selected from propylene glycol, dipropylene glycol, pentylene glycol, glycerin and any mixture thereof.

14. A cosmetic method for caring for and/or making up the skin, comprising the application of a composition according to any of claims 1 to 13 on the skin.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2019/126522

A. CLASSIFICATION OF SUBJECT MATTER

A61K 8/891(2006.01)i; A61K 8/89(2006.01)i; A61K 8/92(2006.01)i; A61Q 1/02(2006.01)i; A61K 8/06(2006.01)i; A61Q 1/04(2006.01)i; A61Q 1/06(2006.01)i; A61K 8/02(2006.01)i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A61K; A61Q

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DWPI, SIPOABS, CNABS, CNTXT, EPTXT, USTXT, WOTXT, CATXT, CNKI: L'oreal, OREA, ZHAO Yujie, Water 2W in 2W oil, W/O, emuls+, polyethylene, polyethylene wax, Performalene500-L, New Phase Technologies, PE wax, ethylene S homopolymer, jojoba, jojoba ester , Floraesters 70, Jojoba Esters-70, Floratech, Flora Technologies, silicone, pigment, color+, colour+, polydimethyl S methyl S siloxane, alkyl dimethicone copolyol, PEG/PPG-10/1 dimethicone, disodium stearylglutamate, aluminum hydroxide, lip?, makeup?, cosmetic, skin

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 110225741 A (CHANEL PARFUMS BEAUTE) 10 September 2019 (2019-09-10) description paragraphs [0165], [0192], [0197], [0207], [0244], [0253], example 5	1-14
X	US 2005008592 A1 (L'OREAL) 13 January 2005 (2005-01-13) claims 1-99	1-14
X	WO 2016007519 A1 (MARY KAY INC.) 14 January 2016 (2016-01-14) claims 8, 20, description paragraphs [0005]-[0006], [0056], [0058], [0067], example 1	1-14
X	EP 1647268 A1 (L'OREAL) 19 April 2006 (2006-04-19) claims 31, 33, 35, 38, example 2	1-14
A	FR 2911496 A1 (L'OREAL) 25 July 2008 (2008-07-25) the whole document	1-14

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

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“O” document referring to an oral disclosure, use, exhibition or other means

“P” document published prior to the international filing date but later than the priority date claimed

“T” later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

“X” document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

“&” document member of the same patent family

Date of the actual completion of the international search

14 August 2020

Date of mailing of the international search report

24 September 2020

Name and mailing address of the ISA/CN

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2019/126522

Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
CN	110225741	A	10 September 2019	EP	3576713	A1	11 December 2019
				US	2019388306	A1	26 December 2019
				FR	3062301	A1	03 August 2018
				KR	20190109434	A	25 September 2019
				JP	2020514322	A	21 May 2020
				FR	3062301	B1	13 March 2020
				WO	2018142076	A1	09 August 2018
US	2005008592	A1	13 January 2005	US	7776348	B2	17 August 2010
				EP	1374835	B1	24 August 2005
				DE	60301372	T2	22 June 2006
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				JP	2004026833	A	29 January 2004
				CN	1237956	C	25 January 2006
				EP	1374835	A1	02 January 2004
				KR	20040002712	A	07 January 2004
				DE	60301372	D1	29 September 2005
				CN	1471903	A	04 February 2004
				KR	542066	B1	10 January 2006
				JP	4373141	B2	25 November 2009
				FR	2841464	A1	02 January 2004
WO	2016007519	A1	14 January 2016	US	2016008263	A1	14 January 2016
				EP	3166574	A4	14 March 2018
				KR	20170020538	A	22 February 2017
				DE	202015004867	U1	23 December 2015
				CN	105250154	A	20 January 2016
				EP	3166574	A1	17 May 2017
EP	1647268	A1	19 April 2006	US	2006083696	A1	20 April 2006
				JP	4167256	B2	15 October 2008
				US	2008138300	A2	12 June 2008
				JP	2006111629	A	27 April 2006
FR	2911496	A1	25 July 2008	FR	2911496	B1	04 January 2013