

METHOD FOR PRODUCING HAIR COSMETIC COMPOSITION

Field of the Invention

[0001]

The present invention relates to a method for producing a hair cosmetic composition.

Background of the Invention

[0002]

The most important role of hair-care products which are washed out after being applied to the hair such as a shampoo and a conditioner is to keep the scalp and the hair clean while caring for them. Therefore, it is needed for these hair-care products that the "clean feeling" can be obtained by using them. Among other various properties the products have, the fragrance imparted to the products contributes to providing the clean feeling, thus it is desired that the fragrance will last long so as to make it possible to obtain the clean feeling until the next hair-washing after hair-washing.

[0003]

In regards to this, it is conceivable to let the fragrance last for a long time by adding a large amount of compound perfume materials. However, more volatile components in the compound perfume materials will preferentially volatilize during application of hair-care

products and after drying the washed hair, as a result of which, the aroma of the compound perfume materials will change qualitatively over time. Therefore, even if a combination of the compound perfume materials is devised, it is difficult to let the fragrance last for a long time with retained quality of the original aroma.

[0004]

In regards to this, JP-A-54-59498, JP-A-54-93006, JP-A-58-22063, JP-A-2003-526644, JP-A-2009-197055, and JP-A-2009-529018 disclose the technology of letting the fragrance last by releasing a perfume alcohol gradually through hydrolysis using a silicate ester derived from the perfume alcohol and an organic silicon compound such as methyltriethoxysilane.

[0005]

(Patent document 1) JP-A-54-59498

(Patent document 2) JP-A-54-93006

(Patent document 3) JP-A-58-22063

(Patent document 4) JP-A-2003-526644

(Patent document 5) JP-A-2009-197055

(Patent document 6) JP-A-2009-529018

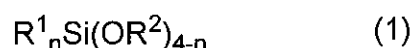
SUMMARY OF THE INVENTION

[0006]

The present invention provides a method for producing a hair cosmetic composition, comprising mixing components other than components (A) and (D) among all of

components including the following components (A) to (E) to be emulsified, followed by adding the components (A) and (D) to the mixture and mixing it at a temperature of 0°C or more and 60°C or less, wherein a total content of the components (A) and (D) is 4 mass% or less, and a mass ratio of the component (A) to a total amount of the components (A) and (D) is more than 0.2 and less than 0.8:

(A) a silicate ester represented by general formula (1)
[0007]



[0008]

wherein R^1 represents an aliphatic hydrocarbon group having 1 to 22 carbon atoms in total which may have a phenyl group, a hydroxy group or an alkoxy group as a substituent; R^2 represents a residue obtained by removing one hydroxy group from a perfume alcohol having 3 to 40 carbon atoms; and n represents an integer of 0 to 3, provided that $(4-n)$ pieces of R^2 may be the same or different;

(B) a quaternary ammonium salt type-cationic surfactant;

(C) an aliphatic alcohol having 16 to 22 carbon atoms;

(D) a perfume compound; and

(E) water.

[0009]

Moreover, the present invention provides a method of using the hair cosmetic obtained by the production method

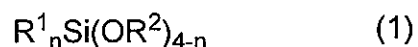
described above, comprising applying the hair cosmetic to the hair and washing the hair cosmetic out.

[0010]

In addition, the present invention provides a hair cosmetic containing the following components (A) to (E), wherein a total content of the components (A) and (D) is 4 mass% or less, and a mass ratio of the component (A) to a total amount of the components (A) and (D) is more than 0.2 and less than 0.8:

(A) a silicate ester represented by general formula (1)

[0011]



[0012]

wherein R^1 represents an aliphatic hydrocarbon group having 1 to 22 carbon atoms in total which may have a phenyl group, a hydroxy group or an alkoxy group as a substituent; R^2 represents a residue obtained by removing one hydroxy group from a perfume alcohol having 3 to 40 carbon atoms; and n represents an integer of 0 to 3, provided that $(4-n)$ pieces of R^2 may be the same or different;

(B) a quaternary ammonium salt type-cationic surfactant;

(C) an aliphatic alcohol having 16 to 22 carbon atoms;

(D) a perfume compound; and

(E) water.

DETAILED DESCRIPTION OF THE INVENTION

[0013]

In products such as hair conditioners, base agents such as an oil agent and a higher alcohol are used in order to care for the hair. These base agents are used by mixing with a surfactant at a high temperature to be emulsified so as to be finely dispersed in water. On the other hand, although perfumes are generally oily, thus preferably emulsified together with the base agent components described above, they volatilize and their fragrance is likely to deteriorate when combined in a high-temperature condition. In some cases, they cause thickening of the formulation. Therefore, it is conceivable to combine the perfumes in a process of cooling down to room temperature after finishing emulsification. If the perfumes being oil agents are combined after finishing emulsification, however, the separation of the formulation and a rapid viscosity decrease will be caused, and how to deal with it has been a concern.

[0014]

Also, when using perfume components composed mainly of a silicate ester as described in JP-A-54-59498, JP-A-54-93006, JP-A-58-22063, JP-A-2003-526644, JP-A-2009-197055, and JP-A-2009-529018, it was a problem that rising of fragrance upon application was inferior.

[0015]

Accordingly, the present invention relates to a method for producing a hair cosmetic composition, wherein an increase or decrease in its viscosity does not occur when combined with perfume components, and its fragrance during and after application can last with appropriate intensity.

[0016]

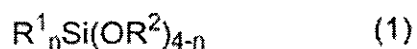
We found that a hair cosmetic composition with suppressed viscosity change due to combining perfumes and excellent sustainability of fragrance during and after application could be produced by using a silicate ester at a fixed amount among perfume components, and emulsifying components other than the perfume components, followed by adding and mixing the perfume components in the process of cooling down to room temperature.

[0017]

(Component (A): A silicate ester represented by general formula (1))

The component (A) is a silicate ester represented by general formula (1) described below and is a perfume precursor which releases perfumes gradually by hydrolysis because it has one or more residue(s) derived from a perfume alcohol in a molecule. Additionally, a perfume alcohol in the present invention refers to an alcohol compound which has volatility and fragrance.

[0018]



[0019]

wherein R^1 represents an aliphatic hydrocarbon group having 1 to 22 carbon atoms in total which may have a substituent of a phenyl group, a hydroxy group or an alkoxy group; R^2 represents a residue obtained by removing one hydroxy group from a perfume alcohol having 3 to 40 carbon atoms; and n represents an integer of 0 to 3, provided that (4-n) pieces of R^2 may be the same or different.

[0020]

The aliphatic hydrocarbon group as R^1 includes a straight-chain or branched alkyl group or alkenyl group. Furthermore, the total number of carbon atoms of R^1 is preferably 8 or more, and preferably 20 or less, more preferably 18 or less.

[0021]

The number of carbon atoms of the perfume alcohol which is a source of R^2 is preferably 5 or more, more preferably 9 or more, and preferably 15 or less, more preferably 14 or less. Any of a primary alcohol, a secondary alcohol or a tertiary alcohol can be used as a perfume alcohol.

[0022]

A specific example of the primary alcohol of the perfume alcohol which is a source of R^2 includes allyl alcohol, trans-2-hexenol, cis-3-hexenol, 9-decenol, 10-undecenol, trans-2-cis-6-nonadien-1-ol, geraniol, nerol,

citronellol, lavandulol, tetrahydrogeraniol, hydroxycitronellol, nopol, farnesol, vetiverol, 2,4-dimethyl-3-cyclohexene-1-methanol, 4-isopropylcyclohexanemethanol (Mayol; Firmenich SA), 2,2-dimethyl-3-(3-methylphenyl)-propanol (Majantol; Wacker Chemie AG), benzyl alcohol, phenylethyl alcohol, phenoxyethyl alcohol, anise alcohol, cinnamic alcohol, 3-phenylpropyl alcohol, 3-methyl-5-phenyl-1-pentanol (phenylhexanol; Firmenich SA), 2-ethyl-4-(2,2,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol (Bagdanol; International Flavors & Fragrances Inc.), 2-methyl-4-(2,3,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol (Sandal mysore core; Kao Corporation), 2-methyl-4-(2,3,3-trimethyl-3-cyclopenten-1-yl)-2-butan-1-ol (Brahmanol; Dragoco Inc.) and the like.

[0023]

A specific example of the secondary alcohol of the perfume alcohol which is a source of R^2 includes undecavertol, 1-(2,2,6-trimethylcyclohexan-1-yl)-pentan-2-ol (Timberol), menthol, 1-(2-*t*-butylcyclohexyloxy)-2-butanol (AMBER CORE; Kao Corporation), *p*-*tert*-butylcyclohexanol, *o*-*tert*-butylcyclohexanol, carveol, 3,4,5,6,6-pentamethyl-2-heptanol (Kohinoor; International Flavors & Fragrances Inc.), 3-methyl-5-(2,2,3-trimethylcyclopenten-1-yl)-pentan-2-ol (Sandalore; Givaudan-Roure Corporation), 2-octyl alcohol, 2-nonyl alcohol, oscillol, 4-isopropylcyclohexanol (Folrosia;

Givaudan-Roure Corporation), iso-Pulegol, 3-heptanol, 3-octanol, 2-undecanol, 1-penten-3-ol, 1-octen-3-ol, 1-nonen-3-ol, 4-methyl-3-decen-5-ol, methylsandeol, dihydrocarveol, 1,3,3-trimethylbicyclo(2.2.1)heptan-2-ol (α -fenchyl alcohol), borneol, α ,3,3-trimethylbicyclo(2.2.1)heptan-2-methanol (Camekol ; International Flavors & Fragrances Inc.), 2(or 3),4-dimethyl-3a,4,5,6,7,7a-hexahydro-4,7-methano-1H-inden-5-ol (Dimethylcyclormol; International Flavors & Fragrances Inc.), isocamphylcyclohexanol, β -caryophyllene alcohol, cedrol, p- α -dimethylbenzyl alcohol, α -phenylethyl alcohol, α -propylphenylethyl alcohol, isobutylbenzyl carbinol, decahydro- β -naphthol and the like.

[0024]

A specific example of the tertiary alcohol of the perfume alcohol which is a source of R² includes 2-methyl-6-methylen-7-octen-2-ol (myrcenol), terpeneol, linalool, 2,6-dimethylheptanol, 2-methyl-3-buten-2-ol, ambrinol, dihydro linalool, tetrahydro linalool, 2,6-dimethyl-2-octanol and 3,7-dimethyl-3-octanol (Tetrahydro muguol), 2,6-dimethyl-3,5-octadien-2-ol and 3,7-dimethyl-4,6-octadien-3-ol (Muguol), 2,6-dimethyl-7-octen-2-ol (dihydromyrcenol), 2,6-dimethyl-2-octanol (tetrahydromyrcenol), ocimenol, 3,6-dimethyl-3-octanol, ethyl linalool, terpeneol, dihydro terpeneol, 4-Thujanol, Nerolidol, bisabolol, Patchouli alcohol, 3,7,11,15-tetramethyl-1-hexadecen-3-ol (isophytol), Geranyl

linalool, sclareol, α,α -dimethylphenylethyl alcohol, p-methyldimethylbenzyl carbinol, dimethylphenylethyl carbinol, 3-methyl-1-phenyl-3-pentanol, 2-isobutyl-4-methyltetrahydro-2H-pyran-4-ol (Florol) and the like.

[0025]

As a silicate ester represented by general formula (1), from the perspective of synthesis easiness, a compound in which at least one of the perfume alcohols for R^2 in general formula (1) is a primary alcohol is preferred, and furthermore, from the perspective of favorability of fragrance quality, it is preferred that the primary alcohol is geraniol.

[0026]

Other preferred perfume alcohols include, from the perspective of favorability of fragrance, 2-methyl-4-(2,3,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol (Sandal mysore core; Kao Corporation), undecavertol, menthol, Folrosia, o-tert-butylcyclohexanol, p-tert-butylcyclohexanol, allyl alcohol, benzyl alcohol, 2-ethyl-4-(2,2,3-trimethyl-3-cyclopentenyl)-2-buten-1-ol (Bagdanol; International Flavors & Fragrances Inc.), nerol, cinnamic alcohol, and from the perspective of improvement in storage stability, undecavertol, menthol, Folrosia, o-tert-butylcyclohexanol, p-tert-butylcyclohexanol are preferred.

[0027]

If there are a plurality of perfume alcohols which are sources of R^2 , the source is preferably a mixture of two or more of the perfume alcohols described above, more preferably, at least one of the perfume alcohols is a primary alcohol and even more preferably, two or more, three or more, and four of them are primary alcohols. Furthermore, at least one primary alcohol is preferably geraniol.

[0028]

Although n represents an integer of 0 to 3, n is preferably 0 or 1, more preferably 0 from the perspective of aroma.

[0029]

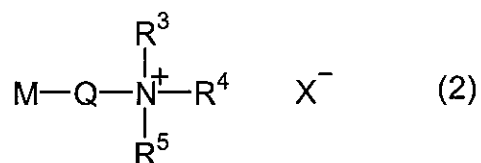
A silicate ester, the component (A), can be used singly or in combination or two or more. The content of the component (A) in the hair cosmetic composition of the present invention is, from the perspective of comfortability of fragrance after drying the hair, preferably 0.08 mass% or more, more preferably 0.2 mass% or more, even more preferably 0.3 mass% or more and preferably 3.2 mass% or less, more preferably 2 mass% or less, even more preferably 1 mass% or less.

[0030]

(Component (B): A quaternary ammonium salt type-cationic surfactant)

A quaternary ammonium salt type-cationic surfactant, the component (B), includes a compound represented by the following general formula (2):

[0031]



[0032]

wherein M represents a hydrogen atom, or a straight-chain or branched, and saturated or unsaturated amide group, N-hydrocarbon carbamoyl group, acyloxy group or hydrocarbonoxy group having 12 to 24 carbon atoms in total; Q represents a divalent straight-chain or branched, and saturated or unsaturated hydrocarbon group having 1 to 25 carbon atoms which may have a hydroxy group; at least one of R³, R⁴ and R⁵ represents a straight-chain or branched alkyl group or alkenyl group having 1 to 25 carbon atoms in total and the rest represents an alkyl group having 1 to 3 carbon atoms; X⁻ represents a halide ion or an organic anion.

[0033]

A quaternary ammonium salt represented by general formula (2) includes a mono-long-chain alkyl (having 12 to 25 carbon atoms) quaternary ammonium salt, a di-long-chain alkyl or alkenyl (having 12 to 25 carbon atoms) quaternary ammonium salt, a branched alkyl (having 12 to 25 carbon atoms) quaternary ammonium salt, an alkylamide

(having 12 to 24 carbon atoms) alkyl (having 1 to 5 carbon atoms) quaternary ammonium salt, an N-hydrocarbon carbamoyl (having 12 to 24 carbon atoms) alkyl (having 1 to 5 carbon atoms) quaternary ammonium salt, an acyl (having 12 to 24 carbon atoms) oxyalkyl (having 1 to 5 carbon atoms) quaternary ammonium salt, an alkyl or alkenyl (having 12 to 24 carbon atoms) oxyalkyl (having 1 to 5 carbon atoms) quaternary ammonium salt.

[0034]

A mono-long-chain alkyl (having 12 to 25 carbon atoms) quaternary ammonium salt includes stearyltrimethylammonium chloride, myristyltrimethylammonium chloride, cetyltrimethylammonium chloride, arachyltrimethylammonium chloride, behenyltrimethylammonium chloride, lauryltrimethylammonium chloride, behenyltrimethylammonium methosulfate and the like.

[0035]

A di-long-chain alkyl or alkenyl (having 12 to 25 carbon atoms) quaternary ammonium salt includes distearyldimethylammonium chloride, dioleyldimethylammonium chloride, diisostearyldimethylammonium methosulfate, di [(2-dodecanoylamino) ethyl] dimethylammonium chloride, di [(2-stearoylamino) propyl] dimethylammonium ethosulfate and the like.

[0036]

A branched alkyl (having 12 to 25 carbon atoms) quaternary ammonium salt includes 2-decyltetradecyltrimethylammonium chloride, 2-dodecylhexadecyltrimethylammonium chloride, di-2-hexyldecyldimethylammonium chloride, di-2-octyldodecyldimethylammonium chloride and the like.

[0037]

An alkylamide (having 12 to 24 carbon atoms) alkyl (having 1 to 5 carbon atoms) quaternary ammonium salt includes stearamidopropyl quaternary ammonium salt. An N-hydrocarbon carbamoyl (having 12 to 24 carbon atoms) alkyl (having 1 to 5 carbon atoms) quaternary ammonium salt includes N-stearylcarbamoylpropyl quaternary ammonium salt. An acyl (having 12 to 24 carbon atoms) oxyalkyl (having 1 to 5 carbon atoms) quaternary ammonium salt includes stearyloxypylpropyl quaternary ammonium salt. A hydrocarbon (having 12 to 24 carbon atoms) oxyalkyl (having 1 to 5 carbon atoms) quaternary ammonium salt includes octadecyloxypylpropyltrimethylammonium chloride.

[0038]

Among these quaternary ammonium salts represented by general formula (2), a quaternary ammonium salt in which M is a hydrogen atom; Q is a divalent saturated hydrocarbon group having 8 to 25 carbon atoms; R³ is a methyl group or an alkyl group having 8 to 25 carbon atoms; and R⁴ and R⁵ are methyl groups is preferred.

[0039]

A quaternary ammonium salt type-cationic surfactant, as the component (B) can be used singly or in combination or two or more. The content of the component (B) in the hair cosmetic composition is, from the perspective of softening the hair in application, rinsing and drying with a towel, preferably 0.05 mass% or more, more preferably 0.1 mass% or more, even more preferably 0.5 mass% or more and preferably 20 mass% or less, more preferably 10 mass% or less, even more preferably 5 mass% or less.

[0040]

(Component (C): An aliphatic alcohol having 12 to 30 carbon atoms)

An aliphatic alcohol having 12 to 30 carbon atoms, as the component (C), includes any of straight-chain or branched, and saturated or unsaturated alcohols. As the component (C), an aliphatic alcohol having 16 to 22 carbon atoms is preferred, and a straight-chain saturated aliphatic alcohol is preferred. A specific example includes cetyl alcohol, stearyl alcohol, oleyl alcohol, arachyl alcohol, behenyl alcohol, octyldodecyl alcohol and the like.

[0041]

An aliphatic alcohol, as the component (C), can be used singly or in combination or two or more. The content of the component (C) in the hair cosmetic composition of the present invention is, from the

perspective of improvement of feel and safety, preferably 0.5 mass% or more, more preferably 1 mass% or more, even more preferably 2 mass% or more and preferably 20 mass% or less, more preferably 15 mass% or less, even more preferably 10 mass% or less.

[0042]

Furthermore, the content of the components (C) and (B) is, from the perspective of improvement of feel and safety, preferably so that the molar ratio of them, (C)/(B), is in a certain range. From the above point of view, the molar ratio of the component (C) and the component (B) is preferably 1.0 or more, more preferably 2.0 or more, even more preferably 3.0 or more and preferably 10.0 or less, more preferably 9.0 or less, even more preferably 8.0 or less.

[0043]

(Component (D): A perfume compound)

A perfume compound, the component (D), is not particularly limited and other synthetic perfumes and natural perfumes can be used. For example, the perfume materials described in "Basic Knowledge of Perfume and Flavor Blending", (written and edited by Mototaka Nakajima, SANGYO-TOSHO CO., LTD, June 21, 1995, the first edition), "Synthetic Perfume, Chemistry and Knowledge on products", (written by Motoichi Indo, THE CHEMICAL DAILY CO., LTD, March 25, 2005, the enlarged and revised edition) can be mentioned.

[0044]

The total content of the perfume components in the hair cosmetic composition of the present invention, that is, the total content of the components (A) and (D), is, from the perspective of lasting of fragrance with the appropriate intensity, preferably 0.4 mass% or more, more preferably 0.5 mass% or more, even more preferably 0.6 mass% or more and 4 mass% or less, preferably 3 mass% or less, more preferably 2 mass% or less.

[0045]

Furthermore, the mass ratio of the component (A) to the total amount of the components (A) and (D), $(A)/[(A)+(D)]$, is, from the perspective of lasting of fragrance with the appropriate intensity, more than 0.2, preferably 0.3 or more, more preferably 0.4 or more and less than 0.8, preferably 0.7 or less, more preferably 0.6 or less.

[0046]

((E): Water)

The hair cosmetic composition produced in accordance with the production method of the present invention contains water as a medium. A water content is the balance except for the content of the components (A) to (D) and optional components.

[0047]

(Other optional components)

The hair cosmetic composition produced in accordance with the production method of the present invention may further be combined with other components commonly used in hair cosmetic compositions depending on the purpose. For example, silicones such as dimethylpolysiloxane, cyclic silicone, amino-modified silicone, dimethiconol, polyether-modified silicone, polyglycidol-modified silicone, methylphenylpolysiloxane, fatty acid-modified silicone, alcohol-modified silicone, alkoxy-modified silicone, epoxy-modified silicone, fluorine-modified silicone, alkyl-modified silicone; polymer compounds such as cationized cellulose, hydroxylated cellulose, highly-polymerized polyethylene oxide; non-ionic surfactants such as polyoxyethylene alkyl ether, polyoxyethylene sorbitan fatty acid ester, glycerol monofatty acid ester, polyglycerol fatty acid ester, polyoxyethylene hydrogenated castor oil, sucrose fatty acid ester, polyglycerol alkyl ether, fatty acid alkanol amide, alkyl glycoside; hydrocarbons such as squalene, squalane, liquid paraffin, liquid isoparaffin, cycloparaffin; glycerides such as castor oil, cacao-seed oil, mink oil, avocado oil, olive oil; waxes such as beeswax, spermaceti, lanolin, carnauba wax; esters such as isopropyl palmitate, isopropyl myristate, octyldodecyl myristate, hexyl laurate, cetyl lactate, propylene glycol monostearate, oleyl oleate, hexadecyl 2-ethylhexanoate, isononyl isononanoate, tridecyl isononanoate; higher fatty acids

such as capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, behenic acid, oleic acid, coconut oil fatty acid, isostearic acid, isopalmitic acid; oil agents such as isostearyl glyceryl ether, polyoxypropylene butyl ether; alcohols such as ethanol, 1-propanol, 2-propanol, butanol, ethylene glycol, propylene glycol, benzyl alcohol, phenoxy ethanol, 2-benzyloxy ethanol, methyl carbitol, ethyl carbitol, propyl carbitol, butyl carbitol, triethylene glycol monoethyl ether, triethylene glycol monobutyl ether, glycerol; antidandruff agents such as zinc pyrithione, benzalkonium chloride; pH regulators; vitamins; bacteriocides; anti-inflammatory agent; preservatives; chelating agents; moisturizing agents such as panthenol; coloring agents such as dyes and pigments; extracts such as polar solvent extracts of Eucalyptus, proteins or their hydrolysates obtained from shells having pearl layers or pearls, proteins obtained from silk or their hydrolysates, protein containing extracts obtained from seeds of legumes, Panax ginseng extracts, rice embryo extracts, Fucus extracts, Camellia japonica extracts, aloe extracts, Alpinia speciosa leaf extracts, chlorella extracts; pearl powders such as mica titanium; refreshing agents such as menthol; coloring matters; ultraviolet absorbers; antioxidants; other components described in ENCYCLOPEDIA OF SHAMPOO INGREDIENTS (MICELLE PRESS) can be mentioned.

[0048]

(pH)

The pH of the hair cosmetic composition produced in accordance with the production method of the present invention is preferably 2.0 or more, more preferably 2.5 or more and preferably 7.5 or less, more preferably 6.5 or less, even more preferably 5.5 or less. In the present invention, the pH of the hair cosmetic composition refers to the value at 25°C when it is 20-fold mass diluted with water.

[0049]

(Viscosity)

The viscosity of the hair cosmetic composition produced in accordance with the production method of the present invention at 30°C is, from the perspective of manageability and performance upon application, preferably 1,000 mPa·s or more, more preferably 5,000 mPa·s or more, even more preferably 10,000 mPa·s or more, even more preferably 20,000 mPa·s or more, even more preferably 30,000 mPa·s or more, even more preferably 35,000 mPa·s or more and preferably 120,000 mPa·s or less, more preferably 100,000 mPa·s or less, even more preferably 70,000 mPa·s or less, even more preferably 60,000 mPa·s or less. The viscosity of the hair cosmetic composition at 30°C can be measured by a B8R-type viscometer (equipped with a helical stand, Rotor No. T-C, 10 rpm, 1 minute).

[0050]

The hair cosmetic composition produced in accordance with the production method of the present invention is used by being applied to the hair, followed by washed out and includes products used in a bathroom such as a shampoo, a hair rinse, a hair conditioner, a hair treatment, a hair pack.

[0051]

(Production Method)

In the present invention, a hair cosmetic composition is produced by mixing the components other than the components (A) and (D) among all of the components including the components (A) to (E) to be emulsified, followed by adding the components (A) and (D) to the mixture and mixing it at a temperature of 0°C or more and 60°C or less.

[0052]

The method for mixing all of the components other than perfume components to be emulsified is not particularly limited and may follow commonly used methods. That is, water as the component (E), is heated to a temperature of 50°C or more and 100°C or less, the component (B) and the component (C) which is heated to its melting point or more are then added to it, and the resultant is stirred to be emulsified. If the melting point of the component (B) is higher than that of the component (C), the components (B) and (C) which are heated to their melting points or more may be added, and

the resultant is stirred to be emulsified. At that time, as other components, an oil agent, a surfactant and the like, may be added to them.

The temperature upon stirring to be emulsified after adding the components (B) and (C) to the component (E) which is heated to a temperature of 50°C or more and 100°C or less is, from the perspective of smoothness upon application and rinsing and styling ease after drying and easy emulsification, preferably 50°C or more, more preferably 53°C or more, even more preferably 55°C or more, even more preferably 58°C or more and preferably 100°C or less, more preferably 90°C or less, even more preferably 85°C or less, even more preferably 80°C or less, even more preferably 75°C or less.

[0053]

After emulsifying the components other than the components (A) and (D) in this way, the perfume components are added and mixed at a temperature of 0°C or more and 60°C or less, and this temperature upon adding and mixing is preferably 20°C or more, more preferably 30°C or more and preferably 50°C or less, more preferably 45°C or less. Subsequently, it may be cooled down to a temperature in the range of 20 to 35°C.

[0054]

In regard to the embodiments described above, preferred aspects of the present invention will be further disclosed below.

[0055]

<1> A method for producing a hair cosmetic composition, comprising mixing components other than components (A) and (D) among all of components including the following components (A) to (E) to be emulsified, followed by adding the components (A) and (D) to a resultant emulsion and mixing a resultant mixture at a temperature of 0°C or more and 60°C or less, wherein a total content of the components (A) and (D) is 4 mass% or less, and a mass ratio of the component (A) to a total amount of the components (A) and (D) is more than 0.2 and less than 0.8:

(A) a silicate ester represented by general formula (1)

[0056]



[0057]

wherein R^1 represents an aliphatic hydrocarbon group having 1 to 22 carbon atoms in total which may have a phenyl group, a hydroxy group or an alkoxy group as a substituent; R^2 represents a residue obtained by removing one hydroxy group from a perfume alcohol having 3 to 40 carbon atoms; and n represents an integer of 0 to 3, provided that $(4-n)$ pieces of R^2 may be the same or different;

(B) a quaternary ammonium salt type-cationic surfactant;

(C) an aliphatic alcohol having 16 to 22 carbon atoms;

(D) a perfume compound; and

(E) water.

[0058]

<2> The method according to <1>, wherein the aliphatic hydrocarbon group of R^1 in general formula (1) is preferably a straight-chain or branched alkyl group or alkenyl group.

[0059]

<3> The method according to <1> or <2>, wherein the total number of carbon atoms of R^1 in general formula (1) is preferably 8 or more and preferably 20 or less, more preferably 18 or less.

[0060]

<4> The method according to any one of <1> to <3>, wherein the number of carbon atoms of R^2 in general formula (1) is preferably 5 or more, more preferably 9 or more and preferably 15 or less, more preferably 14 or less.

[0061]

<5> The method according to any one of <1> to <4>, wherein at least one of R^2 in general formula (1) is preferably derived from a perfume alcohol being a primary alcohol.

[0062]

<6> The method according to <5>, wherein the primary alcohol is preferably geraniol.

[0063]

<7> The method according to any one of <1> to <6>, wherein n in general formula (1) is preferably 0 or 1, more preferably 0.

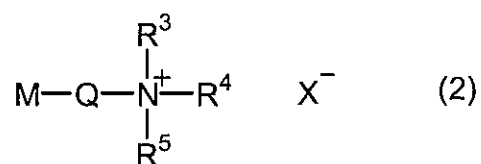
[0064]

<8> The method according to any one of <1> to <7>, wherein a content of the component (A) is preferably 0.08 mass% or more, more preferably 0.2 mass% or more, even more preferably 0.3 mass% or more and preferably 3.2 mass% or less, more preferably 2 mass% or less, even more preferably 1 mass% or less.

[0065]

<9> The method according to any one of <1> to <8>, wherein the component (B) is preferably a compound represented by the following general formula (2):

[0066]



[0067]

wherein M represents a hydrogen atom, or a straight-chain or branched, saturated or unsaturated amide group, N-hydrocarbon carbamoyl group, acyloxy group or hydrocarbonoxy group having 12 to 24 carbon atoms in total; Q represents a divalent straight-chain or branched, and saturated or unsaturated hydrocarbon group having 1 to 25 carbon atoms which may have a hydroxy group; at least one of R³, R⁴ and R⁵ represents a straight-chain or

branched alkyl group or alkenyl group having 1 to 25 carbon atoms in total and the rest represents an alkyl group having 1 to 3 carbon atoms; X^- represents a halide ion or an organic anion.

[0068]

<10> The method according to <9>, wherein the quaternary ammonium salt represented by general formula (2) is preferably selected from the group consisting of a mono-long-chain alkyl (having 12 to 25 carbon atoms) quaternary ammonium salt, a di-long-chain alkyl or alkenyl (having 12 to 25 carbon atoms) quaternary ammonium salt, a branched alkyl (having 12 to 25 carbon atoms) quaternary ammonium salt, an alkylamide (having 12 to 24 carbon atoms) alkyl (having 1 to 5 carbon atoms) quaternary ammonium salt, an N-hydrocarbon carbamoyl (having 12 to 24 carbon atoms) alkyl (having 1 to 5 carbon atoms) quaternary ammonium salt, an acyl (having 12 to 24 carbon atoms) oxyalkyl (having 1 to 5 carbon atoms) quaternary ammonium salt, and an alkyl or alkenyl (having 12 to 24 carbon atoms) oxyalkyl (having 1 to 5 carbon atoms) quaternary ammonium salt.

[0069]

<11> The method according to <9>, wherein preferably, M is a hydrogen atom; Q is a divalent saturated hydrocarbon group having 8 to 25 carbon atoms; R^3 is a methyl group or an alkyl group having 8 to 25 carbon atoms; and R^4 and R^5 are methyl groups.

[0070]

<12> The method according to any one of <9> to <11>, wherein a content of the component (B) is preferably 0.05 mass% or more, more preferably 0.1 mass% or more, even more preferably 0.5 mass% or more and preferably 20 mass% or less, more preferably 10 mass% or less, even more preferably 5 mass% or less.

[0071]

<13> The method according to any one of <1> to <12>, wherein the component (C) is preferably an aliphatic alcohol having 16 to 22 carbon atoms.

[0072]

<14> The method according to any one of <1> to <13>, wherein the component (C) is preferably a straight-chain saturated aliphatic alcohol.

[0073]

<15> The method according to any one of <1> to <14>, wherein a content of the component (C) is preferably 0.5 mass% or more, more preferably 1 mass% or more, even more preferably 2 mass% or more and preferably 20 mass% or less, more preferably 15 mass% or less, even more preferably 10 mass% or less.

[0074]

<16> The method according to any one of <1> to <15>, wherein a molar ratio of the component (C) and the component (B), $(C)/(B)$, is preferably 1.0 or more, more preferably 2.0 or more, even more preferably 3.0 or more

and preferably 10.0 or less, more preferably 9.0 or less, even more preferably 8.0 or less.

[0075]

<17> The method according to any one of <1> to <16>, wherein the total content of the components (A) and (D) is preferably 0.4 mass% or more, more preferably 0.5 mass% or more, even more preferably 0.6 mass% or more and preferably 4 mass% or less, more preferably 3 mass% or less, even more preferably 2 mass% or less.

[0076]

<18> The method according to any one of <1> to <17>, wherein the mass ratio of the component (A) to the total amount of the components (A) and (D) is preferably more than 0.2, more preferably 0.3 or more, even more preferably 0.4 or more and preferably less than 0.8, more preferably 0.7 or less, even more preferably 0.6 or less.

[0077]

<19> The method according to any one of <1> to <18>, wherein the pH of the hair cosmetic composition at 25°C when it is 20-fold mass diluted with water is preferably 2.0 or more, more preferably 2.5 or more and preferably 7.5 or less, more preferably 6.5 or less, even more preferably 5.5 or less.

[0078]

<20> The method according to any one of <1> to <19>, wherein the hair cosmetic composition has a viscosity at 30°C of preferably 1,000 mPa·s or more, more preferably

5,000 mPa·s or more, even more preferably 10,000 mPa·s or more, even more preferably 20,000 mPa·s or more, even more preferably 30,000 mPa·s or more, even more preferably 35,000 mPa·s or more and preferably 120,000 mPa·s or less, more preferably 100,000 mPa·s or less, even more preferably 70,000 mPa·s or less, even more preferably 60,000 mPa·s or less.

[0079]

<21> The method according to any one of <1> to <20>, wherein the temperature upon stirring to be emulsified after adding the components (B) and (C) to the component (E) which is heated to a temperature of 50°C or more and 100°C or less is preferably 50°C or more, more preferably 53°C or more, even more preferably 55°C or more, even more preferably 58°C or more and preferably 100°C or less, more preferably 90°C or less, even more preferably 85°C or less, even more preferably 80°C or less, even more preferably 75°C or less.

[0080]

<22> The method according to any one of <1> to <21>, wherein the hair cosmetic composition is preferably used by being applied to the hair, followed by washed out.

[0081]

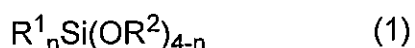
<23> A method of using the hair cosmetic composition obtained by the method according to any one of <1> to <22>, comprising applying the hair cosmetic composition to the hair and washing the hair cosmetic composition out.

[0082]

<24> A hair cosmetic composition comprising the following components (A) to (E), wherein the total content of the components (A) and (D) is 4 mass% or less, and the mass ratio of the component (A) to the total amount of the components (A) and (D) is more than 0.2 and less than 0.8:

(A) a silicate ester represented by general formula (1)

[0083]



[0084]

wherein R^1 represents an aliphatic hydrocarbon group having 1 to 22 carbon atoms in total which may have a phenyl group, a hydroxy group or an alkoxy group as a substituent; R^2 represents a residue obtained by removing one hydroxy group from a perfume alcohol having 3 to 40 carbon atoms; and n represents an integer of 0 to 3, provided that $(4-n)$ pieces of R^2 may be the same or different;

(B) a quaternary ammonium salt type-cationic surfactant;

(C) an aliphatic alcohol having 16 to 22 carbon atoms;

(D) a perfume compound; and

(E) water.

[0085]

<25> The hair cosmetic composition according to <24>, wherein the hair cosmetic composition has a viscosity at 30°C of preferably 1,000 mPa·s or more, more preferably

5,000 mPa·s or more, even more preferably 10,000 mPa·s or more, even more preferably 20,000 mPa·s or more, even more preferably 30,000 mPa·s or more, even more preferably 35,000 mPa·s or more and preferably 120,000 mPa·s or less, more preferably 100,000 mPa·s or less, even more preferably 70,000 mPa·s or less, even more preferably 60,000 mPa·s or less.

[Examples]

[0086]

(Materials)

In Examples and Comparative Examples, the followings were used as the materials. The water content of the materials was measured by the Karl Fischer method.

(1) Geraniol

International Flavors & Fragrances Inc., "GERANIOL 980",
water content: 1000 μ g/g

(2) 2-methyl-4-(2,3,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol

Kao Corporation, "Sandal mysore core" (abbreviated as "SMC" hereinafter), water content: 1000 μ g/g

(3) Undecavertol

Givaudan-Roure Corporation, "UNDECAVERTOL", water
content: 1000 μ g/g

(4) Tetraethoxy silane

Momentive Performance Materials, water content: below the
measuring lower limit (10 μ g/g)

(5) 20 mass% sodium ethoxide ethanol solution

NIPPON SODA CO., LTD.

[0087]

(Production of a silicate ester composition)

Production Example 1

(Synthesis of a silicate ester composition comprising a silicate ester represented by formula (1))

To a 5000 mL 4-neck flask equipped with a condenser was added 2,700 g of geraniol, and a temperature inside the bath was heated to 100 to 110°C, a pressure in the system was gradually decreased and from a point of time the pressure reached to 1.0 kPa, dehydration treatment was carried out for 2 hours. 2 hours later, sampling from the flask was conducted and after confirming by the Karl Fischer that the water content was 200µg/g, the flask was cooled down to room temperature.

Next, a 5000 mL 4-neck flask equipped with a condenser was charged with 2,488.3 g (16.13 mol) of geraniol subjected to dehydration treatment above, 800.2 g (3.83 mol) of tetraethoxy silane and 0.96 g (2.82 mmol) of 20 mass% sodium ethoxide ethanol solution, and the content in the flask was stirred at 130°C for 2 hours with distilling ethanol under the nitrogen gas stream. Then, a pressure inside the bath was gradually decreased to 4 kPa, and the mixture was stirred at 130°C for another 3 hours with distilling ethanol. Then, it was cooled and after the pressure was brought to the atmospheric pressure, the product was filtered to produce

2,555.8 kg of a yellow oily product comprising a silicate ester represented by formula (1). The obtained oily product was analyzed by gas chromatography. The obtained oily product was a silicate ester substituted with four geraniols.

[0088]

Production Example 2

(Synthesis of a silicate ester composition comprising a silicate ester represented by formula (1))

To a 5000 mL 4-neck flask equipped with a condenser was added 2,046.3 g of geraniol and 859.3 g of SMC, and a temperature inside the bath was heated to 100 to 110°C, a pressure in the system was gradually decreased and from a point of time the pressure reached to 1.0 kPa, dehydration treatment was carried out for 2 hours. 2 hours later, sampling from the flask was conducted and after confirming by the Karl Fischer that the water content was 200µg/g, the flask was cooled to room temperature.

Next, a 5000 mL 4-neck flask equipped with a condenser was charged with 2,641.5 g of a mixture of geraniol and SMC subjected to dehydration treatment above (geraniol 12.06 mol, undecavertol 4.02 mol), 800.2 g (3.83 mol) of tetraethoxy silane and 0.96 g (2.82 mmol) of a 20 mass% sodium ethoxide ethanol solution, and the content in the flask was stirred at 130°C for 2 hours with distilling ethanol under the nitrogen gas stream.

Then, a pressure inside the bath was gradually decreased to 4 kPa, and the mixture was stirred at 130°C for another 3 hours with distilling ethanol. Then, it was cooled and after the pressure was brought to the atmospheric pressure, the product was filtered to produce 2,758.3 kg of a yellow oily product comprising a silicate ester represented by formula (1). The obtained oily product was analyzed by gas chromatography. The obtained oily product was a silicate ester substituted with three geraniols and one SMC.

[0089]

Examples 1 to 5, Comparative Examples 1 to 4

The hair conditioners shown in Table 1 were prepared according to the following method.

These hair conditioners were evaluated concerning storage stability and smell in accordance with the following method and criteria.

[0090]

(Preparation Method)

Production methods of Examples 1 to 5 and Comparative Examples 2 to 4

(1) Using a 500 mL beaker, dipropylene glycol was added to ion exchanged water heated to 55°C to provide an aqueous phase.

(2) Separately, the component (B) and the component (C) were mixed and dissolved at 80°C to provide an oily phase.

(3) To the aqueous phase obtained in (1) above was added the oily phase of (2) above while keeping the temperature at 55°C, which was stirred for about 20 minutes (HEIDON Three One Motor BL600, rate 250 r/min) to be emulsified, followed by adding silicone with stirring and cooling it to 45 to 50°C.

(4) The components (A) and (D) were added to the resultant with stirring at 45°C and cooled to 30°C while stirring to produce a hair conditioner.

[0091]

Production method of Comparative Example 1

(1) Using a 500 mL beaker, dipropylene glycol was added to ion exchanged water heated to 55°C to prepare an aqueous phase.

(2) Separately, the components (A), (B), (C) and (D) were mixed and dissolved at 80°C to prepare an oily phase.

(3) To the aqueous phase obtained in (1) above was added the oily phase of (2) above while keeping the temperature at 55°C, which was stirred for about 20 minutes (HEIDON Three One Motor BL600, rate 200 r/min) to be emulsified, followed by adding silicone with stirring, and cooling and stirring to 30°C to prepare a hair conditioner.

[0092]

The hair conditioner having the formulation shown in Table 1 (perfumed sample) was prepared according to the preparation method described before.

The perfumed sample prepared was degassed and placed in a 110 mL glass vial, which was immersed in an incubator at 30°C. At a point of time when the temperature inside the sample reached 30°C, the viscosity was measured by a B8R viscometer (equipped with a helical stand, Rotor No. T-C, 10 rpm, 1 minute).

[0093]

(Viscosity change rate)

The hair conditioner having the formulation shown in Table 1 (perfumed sample) and a hair conditioner from which formulation the perfumes (the components (A) and (D)) were excluded (non-perfumed sample) were prepared according to the preparation method described before, respectively.

The perfumed sample and the non-perfumed sample prepared were degassed and placed in a 110 mL glass vial, which were immersed in an incubator at 30°C. At a point of time when the temperature inside the sample reached 30°C, the viscosity was measured by a B8R viscometer (equipped with a helical stand, Rotor No. T-C, 10 rpm, 1 minute). From these measurements, the viscosity change rate between before and after perfuming was calculated based on the following equation and is shown in Table 1.

$$\text{Viscosity Change Rate} = (\text{Viscosity of perfumed sample}) / (\text{Viscosity of non-perfumed sample})$$

[0094]

(Comfortability of smell)

The following evaluation was carried out in each sample immediately after production.

20 g of a hair tress of Chinese human hair (no chemical treatment) was rinsed with water for 1 minute after cleansing with the non-perfumed shampoo shown below for 1 minute. 1.5 g of the hair conditioner shown in Table 1 or 2 was applied to the tress, followed by spreading it with fingers for 1 minute and at that time, the smell intensity was evaluated.

Then, the above tress was rinsed with tap water at 40°C for 1 minute, and after drying with a towel, it was left to stand at room temperature for 24 hours to be dried, and the smell intensity was evaluated.

The evaluation of the smell intensity was carried out as follows: Seven specialized panelists directly sniffed the smell of the hair tress and alternatively selected any of "Too Strong", "Moderate", "Too weak". The number of people who selected each choice is shown in Table 1 in turn.

[0095]

Formulation of non-perfumed shampoo

Component	Content (mass%)
Sodium laureth sulfate	15.5
Lauramide DEA	1.5
Sodium benzoate	0.5
EDTA-2Na	0.3
Phosphoric acid	pH7

Ion exchanged water	balance
Total	100

onium	-	-	12.0	-	-	-	-	-	-
bl(*5)	5.07	5.07	5.07	5.07	5.07	5.07	5.07	5.07	5.07
	0.60	0.60	0.60	0.15	0.20	0.60	0.00	1.20	2.50
ycol	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00
	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.
of (°C)	45	45	45	45	45	80	45	45	45
	0.50	0.50	0.50	0.50	0.50	0.50	1.00	0.00	0.50
	1.20	1.20	1.20	0.30	0.40	1.20	0.60	0.60	5.00
	47,000	53,000	40,000	47,000	49,000	67,000	49,000	40,000	31,000
(-)	1.07	1.05	1.08	1.05	1.10	1.39	1.10	0.89	0.69
ation	1/6/0	1/6/0	1/6/0	0/4/3	0/5/2	0/7/0	0/2/5	0/6/1	3/4/0
urs	0/7/0	0/7/0	0/7/0	0/3/4	0/4/3	0/7/0	0/5/2	0/2/5	5/2/0

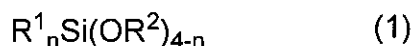
[0097]

- *1: Silicate ester synthesized in Production Example 1
- *2: Behentrimonium chloride (58 wt%) (Kao Corporation, Q2285E-E)
- *3: Stearoxyparyl trimonium chloride (45 wt%) (Kao Corporation, Q-E80K)
- *4: Behentrimonium methosulfate (25 wt%) (Croda Japan, INCROQUAT BEHENYL TMS-PA-MH)
- *5: Kao Corporation, KALCOL 8098
- *6: Mixed perfume of Geraniol and Limonene (5:1)
- *7: Dimethyl siloxane (Shin-Etsu Chemical Co., Ltd., KHS-3)

What is claimed is:

1. A method for producing a hair cosmetic composition, comprising mixing components other than components (A) and (D) among all of components including the following components (A) to (E) to be emulsified, followed by adding the components (A) and (D) to a resultant emulsion and mixing a resultant mixture at a temperature of 0°C or more and 60°C or less, wherein a total content of the components (A) and (D) is 4 mass% or less, and a mass ratio of the component (A) to a total amount of the components (A) and (D) is more than 0.2 and less than 0.8:

(A) a silicate ester represented by general formula (1)



wherein R^1 represents an aliphatic hydrocarbon group having 1 to 22 carbon atoms in total which may have a phenyl group, a hydroxy group or an alkoxy group as a substituent; R^2 represents a residue obtained by removing one hydroxy group from a perfume alcohol having 3 to 40 carbon atoms; and n represents an integer of 0 to 3, provided that $(4-n)$ pieces of R^2 may be the same or different;

(B) a quaternary ammonium salt type-cationic surfactant;

(C) an aliphatic alcohol having 16 to 22 carbon atoms;

(D) a perfume compound; and

(E) water.

2. The method according to claim 1, wherein the total content of the components (A) and (D) in the hair cosmetic composition is 0.4 mass% or more.
3. The method according to claim 1 or 2, wherein the number of carbon atoms of R^2 in general formula (1) is 9 or more and 14 or less.
4. The method according to any one of claims 1 to 3, wherein at least one of R^2 in general formula (1) is derived from a perfume alcohol being a primary alcohol.
5. The method according to claim 4, wherein the primary alcohol is geraniol.
6. The method according to any one of claims 1 to 5, wherein a content of the component (A) is 0.08 mass% or more and 3.2 mass% or less.
7. The method according to any one of claims 1 to 6, wherein a content of the component (B) is 0.05 mass% or more and 20 mass% or less.
8. The method according to any one of claims 1 to 7, wherein a content of the component (C) is 0.5 mass% or more and 20 mass% or less.

9. The method according to any one of claims 1 to 8, wherein a molar ratio of the component (C) and the component (B), $(C)/(B)$, is 1.0 or more and 10.0 or less.

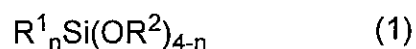
10. The method according to any one of claims 1 to 9, wherein the hair cosmetic composition has a viscosity at 30°C of 1,000 mPa·s or more and 120,000 mPa·s or less.

11. The method according to any one of claims 1 to 10, comprising heating the component (E) to 50°C or more and 100°C or less, mixing the component (B) and the component (C) which is heated to its melting point or more with it to be emulsified.

12. A method of using the hair cosmetic composition obtained by the method according to any one of claims 1 to 11, comprising applying the hair cosmetic to the hair and washing the hair cosmetic composition out.

13. A hair cosmetic composition comprising the following components (A) to (E), wherein a total content of the components (A) and (D) is 4 mass% or less, and a mass ratio of the component (A) to the total amount of the components (A) and (D) is more than 0.2 and less than 0.8:

(A) a silicate ester represented by general formula (1)



wherein R^1 represents an aliphatic hydrocarbon group having 1 to 22 carbon atoms in total which may have a phenyl group, a hydroxy group or an alkoxy group as a substituent; R^2 represents a residue obtained by removing one hydroxy group from a perfume alcohol having 3 to 40 carbon atoms; and n represents an integer of 0 to 3, provided that $(4-n)$ pieces of R^2 may be the same or different;

- (B) a quaternary ammonium salt type-cationic surfactant;
- (C) an aliphatic alcohol having 16 to 22 carbon atoms;
- (D) a perfume compound; and
- (E) water.

14. The hair cosmetic composition according to claim 13, having a viscosity at 30°C of 1,000 mPa·s or more and 120,000 mPa·s or less.