

【DESCRIPTION】

【TITLE OF INVENTION】

Ultraviolet radiation screening cosmetic composition and preparation method thereof

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【TECHNICAL FIELD】

The present invention relates to an ultraviolet radiation screening cosmetic composition and a preparation method thereof.

10 This application claims priority to and the benefit of Korean Patent Application No. 10-2015-0137946 filed on September 30, 2015 with the Korean Intellectual Property Office, the disclosure of which is incorporated herein by reference in its entirety.

15

【BACKGROUND ART】

Unlike organic ultraviolet radiation screening cosmetics, inorganic ultraviolet radiation screening cosmetics use inorganic powder particles, which have
20 no controversy over harmfulness on the human body, and have been widely used on the market. However, when the particle size of inorganic powder particles is large, there is a problem that a so-called whitening phenomenon occurs, in which the skin floats
25 white.

In addition, inorganic powder particles of inorganic ultraviolet screening cosmetics may preferably have a small particle size in order to minimize a whitening phenomenon. However, when the
5 size of the inorganic powder particles is very small, the inorganic powder particles tend to aggregate in the formulation in accordance with their characteristics, and the aggregated inorganic powder particles have problems that they lead to a whitening
10 phenomenon when applied to the skin and also have reduced ability to block ultraviolet radiation.

Accordingly, there is a need to develop an ultraviolet radiation screening cosmetic composition which can minimize a whitening phenomenon and enhance
15 the ability to block ultraviolet radiation. However, it is difficult to disperse particles in the size of nanometers by conventional mixing method or device used for the dispersion of powder during the preparation of cosmetics. Thus, it is difficult to
20 ensure high dispersibility, and also there exists difficulties in maintaining a stable formulation for a long time while maintaining high dispersibility even when the dispersibility is increased.

25 **【DETAILED DESCRIPTION OF THE INVENTION】**

【Technical Problem】

It is one object of the present invention to provide a method for dispersing a cosmetic composition which can prevent a whitening phenomenon when applied to the skin and can enhance the ability to block ultraviolet radiation by increasing dispersibility of fine inorganic powder particles, and a cosmetic composition prepared by the dispersion method.

【Technical Solution】

The present invention provides an ultraviolet radiation screening cosmetic composition in which a ring-mill base comprising inorganic powder particles is ring-mill-dispersed, wherein the average particle size of the inorganic powder particles is 5 to 2000 nm.

The present invention also provides a method for preparing an ultraviolet radiation screening cosmetic composition comprising ring-mill mixing step of mixing inorganic powder particles having a size of 5 to 2000 nm and ring beads; and ring-mill dispersing step of stirring the ring-mill base.

【ADVANTAGEOUS EFFECTS】

The present invention can provide a cosmetic composition which can increase dispersibility of fine inorganic powder particles, prevent a whitening phenomenon when applied to the skin, and exhibit
5 excellent ability to block ultraviolet radiation by ring-mill dispersion of a ring-mill base containing inorganic powder particles.

【BRIEF DESCRIPTION OF DRAWINGS】

10 FIG. 1 is a photograph showing the dispersion state of after the preparation of Comparative Example 1 prepared in Reference Example 2.

FIG. 2 is a photograph showing the dispersion state after the preparation of Example 4 prepared in
15 Reference Example 2.

【DETAILED DESCRIPTION OF THE EMBODIMENTS】

The ultraviolet radiation screening cosmetic composition and the preparation method thereof
20 according to the present invention will be described in detail below.

Unless otherwise specified, the names of the compounds specified in the present disclosure refer to a compound corresponding to the INCI
25 (International Nomenclature Cosmetic Ingredient) Name

registered in the International Cosmetic Ingredient Dictionary (ICID) published by the Cosmetic, Toiletry and Fragrance Association (CTFA). If the name does not exist among the INCI name, they refer to a
5 compound in accordance with the IUPAC nomenclature established by the International Union of Pure and Applied Chemistry (IUPAC). Further, if no compound in accordance with the IUPAC nomenclature exists, they refer to a compound corresponding to the name of
10 the compound commonly used in the technical field of the present invention.

The ultraviolet radiation screening cosmetic composition according to the present invention is prepared by ring-mill dispersion of a ring-mill base,
15 and the ring-mill base will be described in detail below.

The ring-mill dispersion will be described in detail below.

20 **Ring-mill mixing step of mixing inorganic powder particles and ring beads (S10)**

In one embodiment of the present invention, the average diameter of the ring bead is 0.1 mm to 3.0 mm. When the average diameter of the ring bead is less
25 than 0.1 mm, dispersion may not occur sufficiently,

and when the average diameter exceeds 3.0 mm, dispersion may also not occur sufficiently.

In one embodiment of the present invention, the ring bead may be a bead composed zirconia, a bead
5 composed of tungsten carbide, or a combination thereof.

Ring-mill dispersing step of stirring ring-mill base (S20)

10 In one embodiment of the present invention, the ring-mill dispersing step is carried out at a temperature of 20°C to 100°C. When the temperature is less than 20°C, physical dispersion may hardly occur, and when the temperature exceeds 100°C, the
15 components in the ring-milled base may be denatured.

In one embodiment of the present invention, the stirring speed of the ring-mill base is 100 rpm to 1000 rpm. When the stirring speed is less than 100 rpm, dispersion may not occur, and when the stirring
20 speed exceeds 1000 rpm, it may cause deterioration of the performance of the stirring device.

In one embodiment of the present invention, the ring-mill dispersing step is carried out for 30 to 120 minutes. When the run time of the ring-mill
25 dispersing step is less than 30 minutes, dispersion

may not occur sufficiently. When the run time exceeds 120 minutes, the components in the ring-mill base may be denatured due to a temperature rise, and the yield of the ring-milled base may be reduced.

5 Hereinafter, the ring-mill base according to the present invention will be described in detail.

Ring-mill base

1) Inorganic powder particles

10 The ring-mill base according to the present invention includes inorganic powder particles, and the average particle size of the inorganic powder particles is 5 nm to 2000 nm. When the average particle size of the inorganic powder particles is
15 less than 5 nm, the effect of blocking ultraviolet radiation is insignificant, and when the average particle size of the inorganic powder particles exceeds 2000 nm, a light in a wide wavelength range including the visible light region is scattered,
20 which results in a whitening phenomenon due to the scattering of light in the visible light region.

In one embodiment of the present invention, the inorganic powder particle is at least one selected from the group consisting of zinc oxide, titanium
25 dioxide, cerium oxide, and zirconium dioxide.

In one embodiment of the present invention, the inorganic powder particle further includes at least one selected from the group consisting of aluminum hydroxide, stearic acid, triethoxycaprylylsilane, alumina, silica, aluminum stearate, isostearic acid, polyhydroxystearic acid, hydrogen dimethicone, simethicone, methicone, PEG-10 dimethicone, perfluorooctyltriethoxysilane, manganese dioxide, sodium metaphosphate, sodium polyacrylate, and triethoxysilylethyl polydimethylsiloxylethyl hexyl dimethicone.

In one embodiment of the present invention, the amount of the inorganic powder particles is 5% by weight to 80% by weight, preferably 20% by weight to 70% by weight, based on the total weight of the composition.

2) Thickening agent

In one embodiment of the present invention, the ring-mill base further includes at least one thickening agent selected from the group consisting of disteardimonium hectorite, stearylalkonium hectorite, trihydroxystearin, vinyl dimethicone/methicone silsesquioxane crosspolymer, polymethylsilsesquioxane, silica, polymethyl methacrylate, talc, and mica. The thickening agent can appropriately maintain the

viscosity of the ring-mill base, thereby preventing deposition of the inorganic powder particles in the ring-mill base, and thus, the shelf life of the ultraviolet radiation screening cosmetic composition can be extended.

In one embodiment of the present invention, the amount of the thickening agent is 0.1% by weight to 10% by weight, preferably 0.5% by weight to 2% by weight, based on the total weight of the composition.

3) Dispersion medium

In one embodiment of the present invention, the ring-mill base further includes at least one dispersion medium selected from the group consisting of alcohol benzoate having 12 to 15 carbon atoms, cyclopentasiloxane, caprylic/capric triglyceride, dimethicone, dicaprylyl carbonate, isopropyl myristate, and octyldodecanol.

In one embodiment of the present invention, the amount of the dispersion medium is 20% by weight to 90% by weight based on the total weight of the composition.

4) Dispersant

In one embodiment of the present invention, the ring-mill base further includes at least one dispersant selected from the group consisting of

acrylate/ethylhexyl acrylate/dimethicone methacrylate
copolymer, polyglyceryl-3 polyricinoleate,
polyhydroxystearic acid, polyglyceryl-3
polydimethylsiloxylethyl dimethicone, cetyl PEG/PPG-
5 10/1 dimethicone, PEG-10 dimethicone, lauryl PEG-9
polydimethylsiloxylethyl dimethicone, bis-PEG/PPG-
14/14 dimethicone, PEG-30 dipolyhydroxystearate, and
polyglyceryl-2 dipolyhydroxystearate.

In one embodiment of the present invention, the
10 amount of the dispersant is 0.5% by weight to 20% by
weight based on the total weight of the composition.

Hereinafter, the ultraviolet radiation screening
cosmetic composition in which the ring-mill base
described above is dispersed will be described in
15 detail.

Ultraviolet radiation screening cosmetic composition

In one embodiment of the present invention, the
20 viscosity of the cosmetic composition is 3000 to
100000 cps.

In one embodiment of the present invention, the
SPF of the cosmetic composition is 10 to 60.

In the present disclosure, the "SPF" is
25 determined in accordance with the COLIPA

International Sun Protection Factor Test Method (COLIPA, May, 2006), and herein, an increase in skin erythema threshold (minimal erythema dose (MED)) after standardized application of a light blocking preparation is determined as a function of irradiation time.

In addition to the above-mentioned substances, the composition according to the present invention may include other components within a range that does not impair the main effect, preferably which can exert a synergistic effect on the main effect. The composition according to the present invention may further include coloring agents, moisturizers, emollients, ultraviolet absorbers, preservatives, disinfectants, antioxidants, pH adjusters, flavoring agents, cooling agents or antiperspirants widely used in the technical field of the present invention. The mixing amount of the above components can be easily selected by a person skilled in the art within a range that does not impair the objects and effects of the present invention.

In one embodiment of the present invention, the composition may be formulated by containing a cosmetically or dermatologically acceptable medium or base.

In one embodiment of the present invention, the formulation of the composition is any one selected from the group consisting of a water-in-oil type, an oil-in-water type and an oil-dispersed type.

5 In one embodiment of the present invention, the formulation of the composition is impregnated into an expanded polymer foam. More preferably, the expanded polymer foam is an expanded urethane foam.

10 When the composition is impregnated into the expanded polymer foam, the dispersion stability is enhanced such that a water phase component and an oil phase component are not separated.

Hereinafter, the present invention will be described in more detail by way of specific Examples.
15 However, these Examples are given for illustrative purposes only, and the scope of the invention is not intended to be limited by these Examples.

20 **<Reference Example 1> Preparation of ring-mill base**

A ring-mill base was prepared by mixing Preparation Examples 1 to 3 with the composition shown in Table 1 below in accordance with the following procedures.

25 In the case of Preparation Examples 1 and 2, the

oil-type components 3 to 6 of Table 1 were mixed, then the powder-type components 1 and 2 of Table 1 were wetted and mixed with the previously mixed components 3 to 6 to prepare a ring-mill base.

5 In the case of Preparation Example 3, the oil-type components 3 to 6 of Table 1 were mixed and then dispersed with the component 7 of Table 1 by an agi-mixer. The powder-type components 1 and 2 were wetted and mixed with the dispersed components 3 to 7
10 to prepare a ring-mill base.

【Table 1】

Component	Name of component	Composition (Unit: g)		
		Preparation Example 1	Preparation Example 2	Preparation Example 3
1	Titanium oxide particles*aluminum hydroxide*stearic acid (titanium dioxide* aluminum hydroxide*stearic acid)	30	33	30
2	Zinc oxide*triethoxycaprylylsilane	25	27	25
3	C ₁₂₋₁₅ alcohol benzoate	26.5	21.5	25.7
4	PEG-10 dimethicone	2.5	2.5	2.5
5	Acrylate/ethylhexyl	8	8	8

	acrylate/dimethicone methacrylate copolymer* cyclopentasiloxane			
6	Polyhydroxystearic acid*lecithin* ethylhexyl palmitate * isopropyl palmitate *isostearic acid*polyglyceryl-3 polyricinoleate	8	8	8
7	Disteardimonium hectorite	-	-	0.8

< Experimental Example 1> Dispersion of ring-mill base and measurement of viscosity thereof

Each of the ring-mill bases of Preparation Examples 1 to 3 was subjected to ring-mill dispersion by rotating them in a vessel containing zirconia beads having an average diameter of 230 nm at a temperature of 30°C to 80°C for 1 hour with a 500 rpm agitator to prepare compositions of Examples 1 to 3.

The viscosity of the compositions of Examples 1 to 3 prepared above was measured using BROOKFIELD viscometer (BROOKFIELD RVDV-III ULTRA (Serial No. RY6521152)), which runs with spindle No. 64 and No. 29 at a spindle speed of 12 rpm, and the results are shown in Table 2 below.

【Table 2】

Category	Example 1	Example 2	Example 3
Viscosity (cps)	6000	60000	45000

As shown in Table 2, Example 1 containing about

55% by weight of the inorganic ultraviolet radiation blocking agent showed low viscosity of 6000 cps, confirming that dispersion was well achieved. Example 2 also showed that dispersion was well achieved with an appropriate viscosity. In the case of Example 3, it was confirmed that decomposition of the inorganic ultraviolet radiation blocking agent having a high density could be prevented over time by including a thickening agent.

In addition, in the case of Example 1, since the viscosity was low, it was difficult to suspend the inorganic powder particles in the formulation, and deposition phenomenon, which is sinking of inorganic powder particles, and caking phenomenon, which is an aggregation phenomenon caused by deposition, occurred in some parts. In contrast, in the case of Example 3, even though the viscosity was lower than that in Example 2, due to disteardimonium hectorite contained as a component, a network was formed in the oil phase to suspend the inorganic powder, confirming that the formulation was stably maintained without sinking of the inorganic powder particles.

<Reference Example 2> Preparation of ultraviolet radiation screening composition

【Table 3】

Compo nents	Name of component	Example and Comparative Example 1 (Unit: g)	4
1	C ₁₂₋₁₅ alcohol benzoate	11.65	
2	Titanium oxide particles*aluminum hydroxide*stearic acid (titanium dioxide* aluminum hydroxide*stearic acid)	12	
3	Zinc oxide* triethoxycaprylylsilane	10	
4	PEG-10 dimethicone	1	
5	Acrylate/ethylhexyl acrylate/dimethicone methacrylate copolymer* cyclopentasiloxane	3	
6	Polyhydroxystearic acid*lecithin* ethylhexyl palmitate * isopropyl palmitate *isostearic acid*polyglyceryl-3 polyricinoleate	3	
7	Disteardimonium hectorite	0.35	
8	Cyclopentasiloxane*cyclohexasil oxane	26.5	
9	lauryl PEG-9 polydimethylsiloxylethyl dimethicone	1.5	
10	Disteardimonium hectorite	1	

11	Distilled water	24.05
12	Disodium EDTA	0.05
13	Sodium chloride (ICID), sodium chloride (KP)	0.5
14	Butylene glycol	5
15	Phenoxyethanol	0.4

In the case of Example 4, an oil-phase was prepared by mixing the ring-mill base, in which the components 1 to 7 of Table 3 were dispersed, with the components 8 to 10. The thus-prepared oil phase was gradually mixed with a water phase in which the components 11 to 15 of Table 3 were mixed, and emulsified for 10 minutes at 7000 rpm to prepare an ultraviolet radiation screening composition.

In the case of Comparative Example 1, the components 3 to 15 were mixed, and then the powder-type components 1 and 2 were wetted and mixed thereto. The mixture was dispersed for 10 minutes at 7000 rpm using a homomixer to prepare an oil phase. Then, the thus-prepared oil phase was gradually mixed with a water phase in which the components 11 to 15 were mixed, and emulsified for 10 minutes at 7000 rpm to prepare an ultraviolet radiation screening composition.

<Experimental Example 2> Measurement of

viscosity and formulation stability

Comparative Example 1 and Example 4 prepared in Reference Example 2 were photographed after their preparation, and the results are shown in FIG. 1 and
5 FIG. 2, respectively.

In FIG. 1, unlike the formulation of Comparative Example 1 in which atypical inorganic powder particles were observed which existed in an aggregated form having a diameter of 20 μm or more,
10 in FIG. 2, it can be confirmed that the formulation of Example 4 showed that the inorganic powder particles were dispersed in a fine size.

In addition, the viscosity of Example 4 and Comparative Example 1 prepared in Reference Example 2
15 was measured under the same conditions as those of Experimental Example 1, and the period during which the formulation remained stable under each measurement condition was measured and the results are shown in Table 4. The criterion for judging
20 whether the formulation remained stable was based on no aggregation and oil separation in the formulation.

1. Aggregation: Reduced glossiness on the exterior, lumpy particles were confirmed on the surface when ground on a glass plate, foreign
25 substance sensation confirmed when applied to the

skin

2. Oil separation: A clear oil band was formed on the top of the formulation when taken out of a 45°C thermostatic chamber (manufactured by JISCO) and a cycle apparatus (LTI-1001SD manufactured by EYELA) for repeatedly changing the temperature range of -10°C to 45°C and recovered to room temperature (can be distinguished since the formulation has a white color).

【Table 4】

		Comparative Example 1	Example 4
Viscosity(cps)		80000	17000
Stability	45°C thermostatic chamber	7 days	60 days
	Cycle apparatus	14 days	90 days

As can be seen from Table 4, it was confirmed that, in the case of Comparing Example 1 in which homomixing was applied under the same composition, the inorganic ultraviolet radiation blocking agent was not properly dispersed as compared with Example 4 in which the ring-mill dispersion was applied, which accelerated the aggregation in the formulation, and also, separation was observed as it did not mix well

with oil on the exterior.

In contrast, in the case of Example 4 in which the ring-mill dispersion was applied, it was confirmed that the stability was excellent and that the viscosity was 1/5 the viscosity of Comparative Example 1 in which homomixing was applied, and thus an excellent application thereof can be expected when applied to the skin.

10 **<Reference Example 3> Preparation of ultraviolet radiation screening composition**

Example 5 was prepared in the same manner as in Example 4 of Reference Example 2, and comparative Example 2 was prepared in the same manner as in Comparative Example 1 of Reference Example 2 except that the composition shown in Table 5 was used (Components 1 to 4 were used as the ring-mill base).

In the case of Example 5, an oil phase was prepared by mixing the ring-mill base, in which the components 1 to 4 of Table 5 were dispersed, with the components 8 to 10. The thus-prepared oil phase was gradually mixed with a water phase in which the components 11 to 15 of Table 5 were mixed, and emulsified for 10 minutes at 7000 rpm to prepare an ultraviolet radiation screening composition.

In the case of Comparative Example 2, the components 4 and 8 to 15 were mixed, and then the powder-type components 1 and 2 were wetted and mixed thereto. The mixture was dispersed for 10 minutes at 7000 rpm using a homomixer to prepare an oil phase. Then, the thus-prepared oil phase was gradually mixed with a water phase in which the components 11 to 15 were mixed, and emulsified for 10 minutes at 7000 rpm to prepare an ultraviolet radiation screening composition.

【Table 5】

co mp on en t	Name of component	Example 5 and Comparative Example 2 (unit: g)
1	Titanium oxide particles*aluminum hydroxide*stearic acid (titanium dioxide*aluminum hydroxide*stearic acid)	8
2	Zinc oxide*triethoxycaprylylsilane	8
3	Ethylhexyl methoxycinnamate	7
4	Ethylhexyl salicylate	4
8	Cyclopentasiloxane*cyclohexasiloxane	26.5
9	lauryl PEG-9 polydimethylsiloxylethyl dimethicone	1.5

10	Disteardimonium hectorite	1
11	Distilled water	24.05
12	Disodium EDTA	0.05
13	Sodium chloride (ICID), sodium chloride (KP)	0.5
14	Butylene glycol	5
15	Phenoxyethanol	0.4

<Experimental Example 3> Measurement of UV blocking ability

To measure the UV blocking index, the intensity of the ultraviolet radiation emitted to the left and right test areas except for the vertebrae of 12 subjects consisting of 6 men and 6 women was adjusted to 20 to 100 mJ/cm² using an IL1733 Radiometer and a SED 240 probe and irradiated with a solar simulator lamp (Solar simulator; 81293(Xenon UV Lamp 1KW, unfiltered)). Minimal erythema dose (MED) was confirmed when 24 hours has been elapsed after UV irradiation. In order to confirm the UV blocking effect of the sample, the sample of Example 5 and Comparative Example 2 of Reference Example 3 was uniformly applied at a thickness of 2 μl/cm² to the same test areas of the subjects, allowed to stand for 15 minutes and dried. An ultraviolet irradiation window was attached and fixed to the application

sites of the sample of Comparative Example 2 and Example 5, and ultraviolet rays were irradiated to other parts except for the irradiation window by blocking ultraviolet rays using leather and a towel.

5 The experiment was conducted by gradually increasing the intensity of the ultraviolet irradiation. The minimum erythema dose was determined after 24 hours of ultraviolet irradiation, and the UV blocking effect was calculated according to Calculation
10 Formula 1 below and the results are shown in Table 6.

[Calculation Formula 1]

UV blocking effect of sample (SPF) = (Minimal erythema dose of area to which the sample was applied) / (Minimal erythema dose of area to which no sample
15 was applied)

【Table 6】

	Comparative Example 2	Example 5
Measured value of SPF	38	57.5

As can be seen from Table 6, under the same composition, the measured value of SPF of Example 5 in which the ring-mill dispersion was applied was
20 increased by 1.5 times compared to that of Comparative Example 2 in which homomixing was applied.

【CLAIMS】

【Claim 1】

An ultraviolet radiation screening cosmetic composition in which a ring-mill base comprising
5 inorganic powder particles is ring-mill-dispersed, wherein the average particle size of the inorganic powder particles is 5 to 2000 nm.

【Claim 2】

The ultraviolet radiation screening cosmetic
10 composition of claim 1, wherein the inorganic powder particle is at least one selected from the group consisting of zinc oxide, titanium dioxide, cerium oxide, and zirconium dioxide.

【Claim 3】

15 The ultraviolet radiation screening cosmetic composition of claim 1, wherein the ring-mill base further comprises at least one thickening agent selected from the group consisting of disteardimonium hectorite, stearalkonium hectorite, trihydroxystearin,
20 vinyl dimethicone/methicone silsesquioxane crosspolymer, polymethylsilsesquioxane, silica, polymethyl methacrylate, talc, and mica.

【Claim 4】

The ultraviolet radiation screening cosmetic
25 composition of claim 3, wherein the amount of the

thickening agent is 0.1% by weight to 10% by weight based on the total weight of the composition.

【Claim 5】

5 A method for preparing an ultraviolet radiation screening cosmetic composition comprising the steps of:

preparing a ring-mill base by ring-mill mixing inorganic powder particles having a size of 5 nm to 2000 nm and ring beads; and

10 ring-mill dispersing by stirring the ring-mill base.