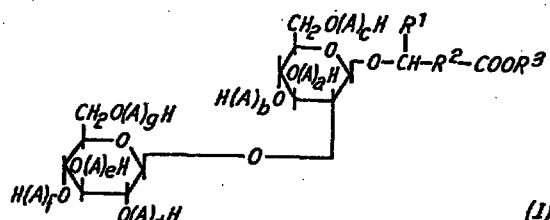


SPECIFICATION

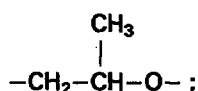
Hydroxypropyl-etherified glycolipid esters in cosmetic materials

5 This invention relates to cosmetic materials for use in skin treatment and more particularly to skin cosmetic materials comprising hydroxypropyl-etherified glycolipid esters.

According to the invention there is provided a skin cosmetic material in the form of a composition including a hydroxypropyl-etherified glycolipid ester of the formula:-



wherein R¹ represents a methyl group or a hydrogen atom; R² represents a saturated or unsaturated acyclic hydrocarbon group having 11 to 15 carbon atoms when R¹ is a methyl group, or a saturated or unsaturated acyclic hydrocarbon group having 12 to 16 carbon atoms when R¹ is a hydrogen atom; A represents the group

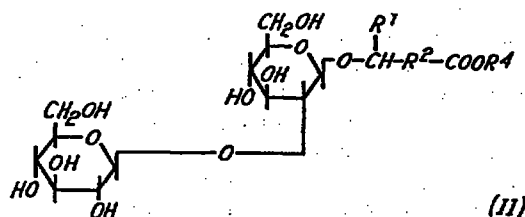


R³ represents a saturated acyclic hydrocarbon group having 1 to 20 carbon atoms, an unsaturated acyclic hydrocarbon group having 2 to 20 carbon atoms, or -(A)_hH; and a, b, c, d, e, f, g and h are integers, whose sum is in the range of from 1 to 60.

The present applicants have made intensive studies of a variety of compounds possessing surface activity and wax-like properties, and have found that the compounds represented by the formula (I) (hereinafter referred to as "POSL" for the sake of brevity) have desirable properties.

Further investigations have been made as to the characteristics and utility of POSL. As a result of these investigations, it has been found that POSL provides skin cosmetic materials with excellent properties resulting from its action as an emulsifying agent and elasticity and lubricity because of its affinity for the skin. The present invention is based on this finding.

POSL to be used in the invention are novel compounds which were first synthesized by the present applicants and which can be produced, for example, by reacting a glycolipid or glycolipid ester represented by the formula (II),



wherein R⁴ represents a saturated hydrocarbon group having 1 to 20 carbon atoms, or an unsaturated hydrocarbon group having 2 to 20 carbon atoms, or a hydrogen atom, with propylene oxide in the presence of an alkali catalyst (see Japanese Patent Application No. 24306/1978).

The total number of carbon atoms of the groups R¹ and R² in POSL is 12 to 16, and the addition mole number of propylene oxide (the sum of a-h) is 1 to 60. Outside these limits, the cleansing ability of the compound is markedly reduced and it no longer acts as an oily or waxy substance.

Preferably the total number of carbon atoms in the groups R¹ and R² is 14 to 16, and the mole number of the added propylene oxide is 5 to 20.

The properties of POSL which are useful and practical in the present invention are shown in Table 1.

Table 1

5	R ¹	R ²	R ³	Addition mole number	Hydroxy value	Acid value	Saponifi- cation value	Appearance	5
10	CH ₃	C ₁₅ H ₂₈	CH ₃	5	420.3	0.2	61.3		10
				7	376.5	0.7	53.5	Viscous	
				15	261.5	0.3	37.0	paste sub-	
15				30	167.2	1.2	21.5	stance	15
	CH ₃	C ₁₅ H ₂₈	C ₁₂ H ₂₅	7	325.3	0.2	47.0	Paste-like	
				15	236.2	0.8	31.5	wax sub-	
20				30	159.3	0.5	20.7	stance	20
	CH ₃	C ₁₅ H ₂₈	-(A) _h H	5	492.0	0.7	60.5		
25				8	413.5	0.0	51.5	Viscous	25
				15	299.0	0.1	37.5	paste sub-	
30				30	185.3	0.0	23.8	stance	30

It should be noted that skin cosmetic materials the properties of which may be improved by addition of POSL include those of the emulsion-solution, compressed powder and stick-shaped types.

1) *Emulsion-solution type cosmetic materials*

35 Emulsion-solution type cosmetics are used for preventing the skin from chapping and keeping the skin 35 fresh-looking. For these purposes, there have been proposed hand creams, cold creams, vanishing creams, milky lotions, beauty washes and the like. Such cosmetic materials are, in general, in the form of an emulsion or solution produced by emulsification or solubilization of various oily substances with surface active agents. Suitable oily substances include, for instance, hydrocarbons such as liquid paraffin, petroleum jelly, 40 paraffin wax, squalane, ceresine wax and the like; esters such as bees-wax, spermaceti, carnauba wax, 40 lanolin and synthetic esters of higher alcohols and fatty acids; alcohols such as long-chain aliphatic alcohols, lanolin alcohol and the like; and fatty acids.

The emulsion-solution type cosmetic materials are intended primarily to function in that an oil film formed on the skin after evaporation of the moisture from inside the skin acts as a barrier against stimuli from 45 outside and prevents the moisture in the skin from drying out and hence protects the skin. However, the oily 45 substance itself often damages the skin because the substance itself degenerates, for example, by oxidation, when the oil film remains on the skin over a long period of time.

It is, therefore, preferable that the oily substance should possess the ability to act as a barrier against external stimuli, that is, blocking properties, and at the same time, should be removed easily by washing 50 whenever necessary. The known oily substances have an excellent ability to act as a barrier, but are liable to 50 remain on the skin even after washing, thereby resulting in damaged skin.

Since the POSL compounds represented by the formula (I) possess a skin-protecting ability, coupled with easy removal by washing with water, they permit the production of conventional cosmetics of an emulsion-solution type which are free from the above-noted defects when POSL is wholly or partially 55 substituted for the usual oily substances. 55

The emulsion-solution type cosmetic materials according to the present invention can be produced in the same manner as with conventional cosmetics, except for the whole or partial substitution of POSL for the oily substances used in the known cosmetics. The amounts of POSL so substituted preferably lie in a range of from 0.1 to 100% by weight. Where desirable, other components or additives can be utilized which have 60 already been widely used in the art and which include, for example, various oily substances, surface active 60 agents, alcohols, viscosity modulators, preservatives, drugs, pigments, perfumes, humectants and water.

Typical examples of the oily substances have been given above. Typical examples of surface active agents are polyoxyethylene alkyl ethers, polyoxyethylene fatty acid esters, polyoxyethylene sorbitan fatty acid esters, sorbitan fatty acid esters, glycerine fatty acid esters, polyoxyethylene glycerine fatty acid esters, 65 polyoxyethylene hydrogenated castor oil, polyoxyethylene sorbitol fatty acid esters, and polyoxyethylene- 65

polyoxypropylene condensed products. Typical examples of alcohols are ethanol and isopropanol. Typical examples of viscosity modulators are carboxymethyl cellulose, methyl cellulose, hydroxyethyl cellulose, polyvinyl alcohol, polyvinyl pyrrolidone, tragacanth gum, carrageenan, locust bean gum, dextrin, dextrin fatty acid esters, carboxyvinyl polymer gels, sodium alginate, and acacia gum. Typical examples of humectants are sorbitol, glycerine, propylene glycol, 1,3-butylene glycol, pyrrolidone carbonic acid sodium salt, lactic acid, sodium lactate, and polyethylene glycol. Typical examples of preservatives are p-hydroxybenzoic acid alkyl ester, sodium benzoate, potassium sorbate, and phenoxyethanol. Finally, typical examples of drugs are vitamins, antiinflammatory agents and germicides.

The expression emulsion-solution type cosmetic material as used in this Specification covers all cosmetics for use in skin protection, such as hand creams, cleansing creams, milky lotions, cold creams, vanishing creams, hair creams, foundation creams, beauty washes, facial packs and so on.

2) Compressed-powder type cosmetic materials

Known compressed powder cosmetics are usually produced by compressing and moulding under high pressure a mixture of a major component such as talc or stericite, and a combining agent such as a paste, for instance, carboxymethyl cellulose, or an emulsifying agent, for instance lanolin. However, these known cosmetics when produced by compression to such extent that it is sufficiently hard to be applied, are easily subject to cracking when dried or dropped by mistake whilst being carried by the consumer, and eventually become unsuitable for use.

The addition of POSL of the formula (I) makes it possible to produce excellent compressed powder cosmetics which do not suffer from the above noted defects.

The compressed powder cosmetic materials according to the invention can be produced by any conventional method, either dry or wet, the only difference being that POSL is added in amounts of 0.1 to 10%, preferably 3 to 7%, by weight of the cosmetic material.

The expression "compressed powder cosmetic material" as used herein, includes the so-called pressed face powders, cheek rouge, highlight, eye-shadow and the like, for use with a compact, which are produced by mixing a major component such as talc, kaolin, stericite, silk powder, nylon powder, metal soap, or starch, with a combining agent such as a paste, oil or emulsion, for instance, squalane, liquid paraffin, isopropyl myristate, oleyl alcohol, or lanolin; combining an inorganic or organic pigment with the mixture; and subsequently compressing the resulting mixture.

3) Stick-shaped type cosmetic materials

Known stick-shaped cosmetics are usually produced by combining castor oil, plant oil, liquid paraffin, a synthetic ester oil, beeswax, carnauba wax, microcrystalline wax and other fats and oils as major or adjunct components. However, it is difficult to make the mixed system homogeneous because of the combination of a wide variety of components. The lapse of time and changes in, for example, temperature and humidity, cause unbalanced miscibility of the components and induce sweating, blooming and varied compressive strengths, thereby resulting in unfavourable appearance and feeling during application of the stick-shaped cosmetics.

The addition of POSL of the formula (I) makes it possible to produce excellent stick-shaped cosmetics which do not suffer from the above-noted defects.

The stick-shaped cosmetic material according to the present invention can be produced by any conventional method, the only difference being that POSL is added in amounts of 0.1 to 10%, preferably 0.5 to 8%, by weight of the cosmetic material.

The expression "stick-shaped cosmetic material" includes stick-shaped rouge, lip creams, stick-shaped eye-shadow and cosmetic pencils which are produced by fusing the fats, oils, and waxes described above, mixing the fused product with a pigment, perfume and a drug to make a homogeneous mixture, pouring the mixture into a mould, and cooling, solidifying and moulding the resulting mass into a stick-shape.

The invention will now be further described with reference to certain specific Examples. The following Reference Example is illustrative of the preparation of POSL for use in the invention. All percentages are by weight unless otherwise indicated.

Reference Example

1) To a mixture of 1500 g of glucose, 75 g of a yeast extract and 15 g of urea was added water in an amount sufficient to adjust the whole volume to 15 l, and the resulting mixture was sterilized and used as a fermentation liquid. This fermentation liquid was inoculated with *Torulopsis bombicola* which had been cultured at 30°C for 48 hours in a culture medium having the same composition as the fermentation liquid.

The fermentation was started under the following conditions: temperature, 20°C; stirring, 300 rpm; and aeration, 0.33 VVM. The fermentation was conducted for 24 hours after inoculation of the micro-organisms, and 150 g of beef tallow was added, followed by adding the same amount of beef tallow at intervals of 24 hours. The added beef tallow totalled 900 g. After the final addition, the fermentation was carried on for further 24 hours. The fermentation time amounted to 168 hours. After completion of the fermentation, the sophorolipid layer which had precipitated at the bottom of the fermentor was collected by decantation to give 1300 g of sophorolipid which was in the form of a paste having a water content of about 50%.

2) In a 200 ml round bottom flask equipped with a stirrer and a Liebig condenser was placed 100 g of the thus obtained sophorolipid so obtained, together with 2.5 g of polypropylene glycol having an average

molecular weight of 200. The mixture was heated in an oil bath at 80°C with stirring under a reduced pressure of 250 mm Hg to eliminate water. The distillation was completed in about 2 hours, and the water content at that time was found to be less than 1%.

3) 150 g of methanol was added to the thus prepared polypropylene solution of sophorolipid, and to the resulting mixture was added 2.5 g of sulfuric acid. The mixture was reacted at $40 \pm 2^\circ\text{C}$ for 90 minutes. The reaction was regarded as having been completed when many spots shown by the raw material or sophorolipid converted on one spot corresponding to a glycolipid methyl ester in thin-layer chromatography on silica gel [developing solvent: chloroform-methanol-acetic acid (75:20:5)].

After completion of the reaction, the mixture was neutralised with a given amount of potassium hydroxide and then filtered. The filtrate was placed again in a round bottom flask equipped with a Liebig condenser, methanol and methyl acetate were removed by distillation to obtain 48 g of a mixture containing 94% of a [(2'-O- β -D-glucopyranosyl- β -D-glucopyranosyl)oxy]alkanic acid and alkenic acid methyl ester as a brown paste and coexisting polypropylene glucose. This mixture was purified by column chromatography on silica gel to obtain a pure [(2'-O- β -D-glucopyranosyl- β -D-glucopyranosyl)oxy]alkanic acid and alkenic acid methyl ester.

IR (cm^{-1}):	1740(>C=O ester)
	1380 - 3200 (-OH sugar)
	900 - 750 (glucopyranose ring)
NMR [δ (pyridine)]:	1.1 - 1.6 (-CH ₂ -CH ₂ -)
	3.6 (-O-CH ₃)
	3.5 - 5.0 (sugar)
	5.5 (-CH=CH- unsaturated fatty acid)

Oil Characterization Analyses:	Acid value:	0
	Hydroxy value:	615
	Saponification value:	88
	Ester value:	87

4) In an autoclave was placed 100 g of the thus obtained mixture of the [(2'-O- β -D-glucopyranosyl- β -D-glucopyranosyl)oxy]alkanic acid and alkenic acid methyl ester and coexisting polypropylene glycol together with 0.25 g of potassium hydroxide, and propylene oxide gas was bubbled into the mixture in an amount corresponding to a given addition mole number of propylene oxide. The mixture was reacted at 100 to 120°C for 6 hours. After completion of the reaction, the mixture was neutralized with phosphoric acid and filtered under high pressure to obtain a crude product in the form of brown paste. This product was purified by chromatography on silica gel to obtain a pure polyoxypropylene [(2'-O- β -D-glucopyranosyl- β -D-glucopyranosyl)oxy]alkanic acid and alkenic acid methyl ester as a pale yellow paste.

Example 1

POSL to be used in the present invention and comparative compounds were applied to the skin, and the resistant properties of these oil substances to keratolysis were examined by the Scrub method (Note 1). The results obtained are shown in Table 2.

Table 2

Test compounds		R ¹	R ²	R ³	a-h	Corneocyte counts (number/cm ²)		
						1st	2nd	3rd
Present compounds	POSL	CH ₃	C ₁₅ H ₃₀	CH ₃	12	1.6×10 ³	7.2×10 ³	1.5×10 ⁴
	POSL	CH ₃	C ₁₁ H ₂₂	CH ₃	7	1.7×10 ³	1.0×10 ³	1.8×10 ³
	POSL	H	C ₁₆ H ₃₀	C ₁₂ H ₂₅	15	1.3×10 ³	1.4×10 ³	1.2×10 ⁴
	POSL	CH ₃	C ₁₅ H ₂₈	-(A) _h H	30	1.1×10 ³	1.1×10 ³	1.3×10 ³
Vaseline						1.6×10 ³	1.8×10 ³	1.1×10 ³
Comparative compounds	Liquid paraffin					2.3×10 ³	1.9×10 ³	1.0×10 ³
	Lanolin					1.9×10 ³	1.7×10 ³	1.6×10 ³
Control						8.5×10 ⁴	1.1×10 ⁴	3.2×10 ³

(Note 1) Scrub Method: Each oil substance was applied to the anterbrachial part of a healthy subject in an amount of 0.1 g/4 cm × 4 cm, and a glass cup having a diameter of 2.5 cm was fixed thereon. In the glass cup was placed 2 ml of a solution containing 0.1 % polyoxy-ethylene sorbitan monolaurate, and the skin was scrubbed with a rod for 60 seconds. Thereafter, each test solution was dyed with Crystal Violet/Fuchsine Base, and the number of corneocyte exfoliated from the skin was counted which was regarded as being the first Scrub number. The second and third Scrub numbers were counted by repeating the same procedure.

From the above results, it can be seen that POSL possesses a skin-protecting ability equal to that of the usual oils of known skin-protecting creams.

Example 2

POSL and comparative compounds were examined as to their obwashed properties by the method described in Note 2. The results obtained are shown in Table 3.

Table 3

Test compounds		R ¹	R ²	R ³	a-h	Obwashed property (%)
Present compounds	POSL	CH ₃	C ₁₅ H ₃₀	CH ₃	12	96.4
	POSL	CH ₃	C ₁₁ H ₂₂	CH ₃	7	98.2
	POSL	H	C ₁₆ H ₃₀	C ₁₂ H ₂₅	15	95.5
	POSL	CH ₃	C ₁₅ H ₂₈	-(A) _h H	30	82.5
Comparative compounds	Vaseline					17.5
	Liquid Paraffin					34.2
	Lanolin					21.3

(Note 2) Obwashed Property: 120 ± 2 mg of each test compound was coated homogeneously on a glass plate of $2.5\text{ cm} \times 3.5\text{ cm}$ with the use of chloroform. The plate was immersed in 2 ℓ of an aqueous solution containing 0.5% sodium lauryl sulfate and having a temperature of 30°C . In this solution was placed a stirrer having a diameter of 5 cm, and the solution was stirred at 300 rpm for 2 minutes. Thereafter, the glass plate was placed in 2 ℓ of water having a temperature of 30°C , and the water was stirred with the stirrer at 300 rpm for 1 minute. The plate was rinsed and dried. The weight of the glass plate was measured, and the change in weight was calculated. The obwashed property was calculated according to the following equation.

Obwashed property (%)

10

$$= 1 - \left(\frac{\text{Weight of residual oil on glass plate after washing}}{\text{Weight of oil adherent to glass plate before washing}} \right) \times 100$$

10

From the above results, it can be seen that POSL possesses an extremely high obwashed property as compared with the usual oil substances and can be easily removed by washing.

Example 3

Various creams were prepared which had the composition indicated below. The feeling of application of each test cream was determined by a panel of ten experts. The results obtained are shown in Table 4.

Cream Composition:

25	Test compound	5 (%)	25
	Liquid paraffin	10	
	Beeswax	3	
	Cetanol	2	
30	Stearic acid monoglyceride	3	30
	Polyoxyethylene sorbitan monostearate	2	
	Methylparaben	0.1	
	Butylparaben	0.1	
35	Purified water	balance	35
		(%) by weight)	

Table 4

40	Test compounds		Stick- iness	Moist- ness	Refresh- ingness	Spread- ability	Hardness	40
	Present	POSL	-0.3	+1.7	+0.1	+0.1	0	
45	compound	$R^1 = \text{CH}_3$ $R^2 = \text{C}_{15}\text{H}_{28}$ $R^3 = \text{CH}_3$ $a-h=12$						45
50	Compara- tive compounds	Vaseline	+1.8	-0.8	-1.3	+0.7	-0.2	50
		Lanolin	+1.3	+0.2	-1.5	+0.3	-0.1	

Note: Every item was adjudged according to the following evaluation standards. The figures in Table 4 indicate the average values of the ten experts' scores.

- 60
- +2 : Severe

+1 : Fair

0 : Moderate

-1 : Lacking

-2 : Nothing
- 60

From the above results, it can be seen that the cream containing POSL gives a less sticky and more moist feeling, without destroying the other properties, in comparison with the creams comprising the usual oil substances.

Example 4

**Hand Cream
(Materials)**

5	1)	POSL ($R^1=CH_3$, $R^2=C_{15}H_{28}$, $R^3=CH_3$, $a-h=12$)	1.5 (%)	5
	2)	Stearic acid	10.0	
10	3)	Stearic acid monoglyceride	1.5	10
	4)	Polyoxyethylene monostearate	1.5	
	5)	Triethanolamine	0.3	
15	6)	Methylparaben	0.1	15
	7)	Butylparaben	0.1	
20	8)	Perfume	0.2	20
	9)	Purified water	balance (%) by weight)	

25 (Process) 25

1) to 4) were mixed at 70°C, and to this was added with stirring a mixture of 5) to 7) and 9) which had been heated at 70°C. The resulting mixture was emulsified. After completion of the emulsification, the mixture was allowed to cool to about 40°C, to which was then added 8) to produce a hand cream.

30 30

Example 5

**Cold Cream
(Materials)**

35	1)	POSL ($R^1=CH_3$, $R^2=C_{11}H_{22}$, $R^3=C_{12}H_{25}$, $a-h=10$)	5.0 (%)	35
	2)	Squalane	5.0	
	3)	Liquid paraffin	10.0	
	4)	Bees wax	3.0	
40	5)	Lanolin	1.5	40
	6)	Polyoxyethylene sorbitaon monooleate	3.0	
	7)	Sorbitan monooleate	2.0	
	8)	Methylparaben	0.1	
	9)	Butylparaben	0.1	
45	10)	Perfume	0.2	45
	11)	Purified water	balance (%) by weight)	

(Process)

50 1) to 7) and 9) were mixed at 70°C, and to this was added with stirring a mixture of 8) and 11) which had been heated at 70°C. The resulting mixture was emulsified. After completion of the emulsification, the mixture was allowed to cool to about 40°C, to which was then added 10) to produce a cold cream. 50

18

Example 6

Cleansing Milk
(Milky Lotion)

5 (Materials) 5

1)	POSL ($R^1=CH_3$, $R^2=C_{11}H_{22}$, $R^3=CH_3$, $a-h=30$)	1.5 (%)	
2)	Liquid paraffin	1.5	
10 3)	Spermaceti	0.5	10
4)	Cetanol	0.5	
5)	Sorbitan monostearate	1.0	
6)	Polyoxyethylene monooleate	0.5	
7)	Methylparaben	0.1	
15 8)	Butylparaben	0.1	15
9)	Perfume	0.1	
10)	Purified water	balance	
		(% by weight)	

20 (Process) 20

1) to 6) and 8) were mixed at 70°C and to this was added with stirring a mixture of 7) and 10) which had been heated at 70°C. The resulting mixture was emulsified. After completion of the emulsification, the mixture was allowed to cool to about 40°C, to which was then added 9) to produce a cleansing milk.

25 Example 7 25

Hair Cream Oil

(Materials)

30 1)	POSL ($R^1=CH_3$, $R^2=C_{15}H_{28}$, $R^3=CH_3$, $a-h=10$)	15.0	30
2)	Liquid paraffin	3.0	
3)	Olive oil	5.0	
4)	Paraffin wax	1.0	
35 5)	Castor oil	0.5	35
6)	Polyoxyethylene sorbitol tetraoleate	3.5	
7)	Methylparaben	0.1	
8)	Butylparaben	0.1	
9)	Perfume	0.3	
40 10)	Purified water	balance	40
		(% by weight)	

(Process)

1) to 6) and 8) were mixed at 70°C, and to this added with stirring a mixture of 7) and 10) which had been heated at 70°C. The resulting mixture was emulsified. After completion of the emulsification, the mixture was allowed to cool to about 40°C, to which was then added 9) to produce a hair cream oil.

Example 8

50 Skin Lotion 50

(Materials)

1)	POSL $R^1=H$, $R^2=C_{16}H_{30}$, $R^3=C_{12}H_{25}$, $a-h=12$)	1.5 (%)	
55 2)	Ethanol	15.0	55
3)	Polyoxyethylene cetyl ether	1.5	
4)	Methylparaben	0.1	
5)	Perfume	0.2	
6)	Purified Water	balance	
60		(% by weight)	60

(Process)

1) to 6) were stirred at an ordinary temperature to make a homogeneous mixture.

Example 9

Facial Pack

5 (Materials)

5

1)	POSL ($R^1=CH_3$, $R^2=C_{13}H_{26}$, $R^3=-(A)_hH$, $a-h=5$)	1.5 (%)	
2)	Polyvinyl Alcohol	15.0	
3)	Titanium Oxide	2.0	
10 4)	Ethylene Glycol	4.0	10
5)	Methylparaben	0.1	
6)	Perfume	0.2	
7)	Purified Water	balance	
		(% by weight)	

15

(Process)

15

7) was heated to 90°C, to this was added with stirring 2) in limited amounts to make a homogeneous mixture and then added 1), 3), 4) and 5), and the mixture was stirred to become homogeneous. The mixture was allowed to cool to about 40°C, to which was then added 6). The mixture was made homogeneous and

20 cooled to obtain a facial pack.

20

Example 10

Foundation Cream

25

(Materials)

25

1)	POSL ($R^1=CH_3$, $R^2=C_{15}H_{28}$, $R^3=CH_3$, $a-h=10$)	3.0 (%)	
2)	Isopropyl myristate	3.0	
30 3)	Stearic acid	5.0	30
4)	Talc	12.0	
5)	Titanium oxide	5.0	
6)	Red iron oxide	0.5	
7)	Polyoxyethylene stearate	2.5	
35 8)	Stearic acid monoglyceride	1.5	35
9)	Methylparaben	0.1	
10)	Propylparaben	0.1	
11)	Perfume	0.2	
12)	Purified water	balance	
40		(% by weight)	40

(Process)

1) to 3), 7), 8) and 10) were mixed at 70°C, to this was added with stirring a mixture of 9) and 12) which had been heated at 70°C. To the resulting mixture were added with stirring 4) to 6) while keeping the temperature at 70°C. The mixture was allowed to cool to a temperature less than about 40°C, to which was then added 11) to produce a foundation cream.

45

Example 11

50 After test samples A to D having the compositions and produced by the preparation methods listed in Table 5 and commercial goods E and F were preserved at 50°C for 12 hours, any cracks caused by drying were observed with the naked eye. The results obtained are shown in Table 6.

50

From these results, it can be seen that the samples C and D produced by the present method have nothing defective and are stable.

TABLE 5

Samples	Compositions (%)	Preparation methods
A Usual goods 1	Talc	60 Dry method
	Stericite	20 Compressive pressure:
	Kaolin	10 50 kg/cm ²
	Aluminium stearate	2
	Lanolin	2
	Squalane	3
	Titanium oxide	2
B Usual goods 2	Iron oxide	1
	Talc	70 Wet method
	Stericite	25 Compressive pressure:
	Aluminium stearate	2 40 kg/cm ²
	Titanium oxide	2
	Iron oxide	1
	0.1% Carboxymethyl cellulose	20
C Present goods 1	Talc	75 Dry method
	Stericite	16 Compressive pressure:
	Aluminium stearate	2 40 kg/cm ²
	POSL (I)	4
	Titanium oxide	2
	Iron oxide	1
D Present goods 2	Talc	80 Wet method
	Kaolin	8 Compressive pressure:
	Aluminium Stearate	3 40 kg/cm ²
	POLS (II)	6
	Titanium oxide	2
	Iron oxide	1
E Commercial solid face powder		
F Commercial cheek rouge		

POSL (I): R¹=CH₃, R²=C₁₅H₂₈, R³=C₁₂H₂₅, a-h=7

POSL (II): R¹=CH₃, R²=C₁₅H₂₈, R³=-(A)_nH, a-h=15

Table 6

Samples	Cracked conditions	
5 A	Cracked and exfoliated partly	5
B	Cracked	
C	Not cracked nor exfoliated	
10 D	Not cracked nor exfoliated	10
E	Cracked	
15 F	Cracked and exfoliated partly	15

Example 12

20 The same samples A to F as used in Example 11 were each placed in a compact, and the compact was dropped naturally from a height of 1.5 m ten times. Any cracks caused by such falling were observed with the naked eye. The results obtained are shown in Table 7. 20

From these results, it can be seen that the samples C and D produced the present method have nothing defective and are stable and excellent in feeling when applied.

25 25

Table 7

Samples	Cracked conditions	
30 A	Cracked and exfoliated almost wholly from the compact	30
B	Cracked and exfoliated partly from the compact	
35 C	Not cracked nor exfoliated	35
D	Not cracked nor exfoliated	
40 E	Cracked and exfoliated partly from the compact	40
F	Cracked and exfoliated almost wholly from the compact	
45		45

Example 13

Four species A to D extracted from the same samples as used in Examples 11 and 12 and two species G 50 and H freshly prepared according to the following compositions and methods (Table 8) were cut off in a range of 3 cm × 3 cm. Each of these samples was placed horizontally and given a stress (5 kg/cm²) from a horizontal direction at a rate of 5 mm/minute so that any cracks occurred. The distance (mm × 10) by which the dynamic point had migrated was measured. The results obtained are shown in Table 9. 50

From these results, it can be seen that the present samples each show a long migration distance which 55 indicates that the samples are resistant to deformation and are stable. 55

Table 8

Compositions	Samples		H	
	G			
5 Talc	56 parts	65 parts		5
Stericite	20	16		
10 Aluminium stearate	2	2		10
Titanium oxide	2	2		
Iron oxide	1	1		
15 Kaolin	10	8		15
Lanolin	2	1		
20 Squalane	3	2		20
POSL (III)	4	-		
POSL (IV)	-	3		
25 Preparation method	Dry	Dry		25
Compressive pressure	40 kg/cm ²	40 kg/cm ²		
30 POSL (III): $R^1=CH_3, R^2=C_{15}H_{28}, R^3=CH_3, a-h=30$ POSL (IV): $R^1=CH_3, R^2=C_{15}H_{28}, R^3=-(A)_nH, a-h=5$				30

Table 9

Samples	Runs						Average values	
		1st	2nd	3rd	4th	5th		
A		30	33	28	29	32	30.4	
40 B		28	27	29	32	31	29.4	40
C		45	42	44	43	42	43.2	
D		41	42	41	41	43	41.6	
45 G		39	41	42	39	40	40.2	45
H		40	41	39	42	42	40.8	
50								50

Example 14

Commercial samples A to G, samples H to Q of the present invention having the following compositions (Tables 10 to 15) produced by any usual method and usual samples R to T (total: 20 stick-shaped cosmetic samples) were preserved at 10°C for 4 hours and then at 40°C, 80% R. H. for 4 hours. Sweating was observed with the naked eye. Blooming was also observed with the naked eye after the samples A to T were preserved at 40°C for 8 hours and then at 5°C for 24 hours. The results obtained are shown in Table 16.

From these results, it can be seen that the present samples H to Q are stable with no sweating and blooming.

Table 10

(Stick-shaped lip rouge)

Compositions	H	I	J	K
Carnauba wax	10 parts	8 parts	8 parts	10 parts
Bees wax	15	14	15	15
Castor oil	40	42	42	43
Oleyl alcohol	12	10	8	6
Ceresine wax	10	13	15	15
Stearic acid	1	-	1	-
POSL (I)	-	-	3	6
POSL (II)	3	-	-	-
POSL (III)	-	4	-	-
Red iron oxide	3	3	3	2
Pigment (Red 202)	2	2	2	2
Titanium dioxide	4	4	3	1

Table 11

(Stick-shaped lip cream)

Compositions	L	M	N
Castor wax	30 parts	29 parts	31 parts
Oleyl alcohol	23	25	23
Microcrystalline wax	12	10	8
Bees wax	14	16	15
Glycerine monostearate	8	8	8
POSL (I)	-	4	-
POSL (III)	6	-	-
POSL (IV)	-	-	6
Carnauba wax	5	6	7
Lanolin	2	7	2

Table 12

(Stick-shaped eye-shadow)

Compositions	Samples	O	P	Q
Castor wax		20 parts	20 parts	20 parts
Oleyl alcohol		25	27	22
Squalane		10	10	10
Carnauba wax		10	5	20
Microcrystalline wax		13	10	7
Glycerine monostearate		8	8	8
POSL (I)		4	-	-
POSL (II)		-	3	6
Titanium oxide		7	4	4
Ultramarine		3	4	3

Table 13

(Stick-shaped lip rouge by usual prescription)

Compositions	Samples	R	S
Bees wax		15 parts	15 parts
Ozocerite		10	-
Carnauba wax		5	10
Ceresine wax		4	-
Lanolin		5	5
Lanolin absorption base		14	-
Isopropyl alcohol		10	-
Diethyl sebacate		10	-
Castor oil		15	65
Cetanol		-	5 parts
Eosine		2 parts	-
Bromic acid		-	3
Pigment		10	7
Antioxidant		Optional	-
Perfume		Optional	0.3

Table 14

(Stick-shaped eye-shadow by usual prescription)

	Sample	T
Compositions		
Ceresine		26 parts
Hydrogenated oil		5
Castor oil		43
Carnauba wax		4
Liquid paraffin		6
Titanium dioxide		8
Iron oxide (Red)		4
Iron oxide (Black)		4

Table 15

(Kinds of POSL used in samples)

Kinds	R ¹	R ²	R ³	Sum of a-h
POSL (I)	CH ₃	C ₁₅ H ₂₈	CH ₃	7
POSL (II)	CH ₃	C ₁₅ H ₂₈	C ₁₂ H ₂₅	30
POSL (III)	CH ₃	C ₁₅ H ₂₈	$\begin{array}{c} \text{CH}_3 \\ \\ (\text{CH}_2\text{-CH-O})_n\text{H} \end{array}$	5
POSL (IV)	CH ₃	C ₁₅ H ₂₈	$\begin{array}{c} \text{CH}_3 \\ \\ (\text{CH}_2\text{-CH-O})_n\text{H} \end{array}$	15

Table 16

Samples		Sweating	Blooming	
5	A	++	+	5
Commercial lip rouge	B	++	—	
10	C	+	+	10
Commerical lip cream	D	+++	++	
15	E	+++	+	15
Commercial stick-shaped eye-shadow	F	++	+	
	G	+	—	
20	H	—	—	20
	I	—	—	
25 Present invention	J	—	—	25
Lip rouge	K	—	—	
30	L	—	—	30
	M	—	—	
Lip cream	N	—	—	
35	O	—	—	35
Present invention	P	—	—	
40	Q	—	—	40
	R	++	+	
45 Lip rouge by usual prescription	S	++	++	45
	T	+	+	
Eye-shadow by usual prescription				
50				50
Evaluation standards; — : Nothing				
+ : Slightly				
++ : Fairly				
+++ : Severe				
55				55

Example 15

60 Thirteen species extracted from the same samples A to T as used in Example 14 were cut crosswise, each having a thickness of 5 mm, and preserved at 5°C and 30°C for 12 hours. The stress was measured that was required to cause destruction when the species was compressed with a rheometer. The results obtained are shown in Table 17.

From these results it can be seen that the usual samples now on the market and the samples by the usual 65 prescription are large in their compressive strength ratios between 5°C and 30°C, while the present samples 65

are small in the same ratios. That is, the present samples each show only a slight difference of the application feeling under cold and warm conditions.

Table 17

5	Samples	Compressive strengths ($\times 10^6$ dyn/cm ²)		Ratios of compressive strengths at 5°C and 30°C	5
		5°C	30°C		
10	A	9.0	2.1	4.3	10
	B	8.8	2.3	3.8	
15	D	9.3	1.9	4.9	15
	E	7.5	1.6	4.7	
	H	5.2	4.1	1.7	
20	I	8.5	4.7	1.8	20
	K	6.5	4.1	1.6	
25	L	5.1	2.8	1.8	25
	N	4.6	3.3	1.4	
30	O	4.1	2.2	1.9	30
	R	9.2	2.2	4.2	
	S	9.1	2.3	4.0	
35	T	7.4	1.8	4.1	35

Example 16

40 Five species of stick-shaped lip rouge extracted from the samples A to H used in Example 14 were preserved at 5°C and 40°C for 48 hours. Each rouge was applied to the lips of five subjects who had remained standing at 5°C and 40°C for 30 minutes, respectively, in a thermostated chamber. The results obtained are shown in Table 18.

From these results, it can be seen that the feeling on application of each of the present samples is stable 45 and excellent at 5°C and 40°C.

Table 18

5°C							40°C						
5	1	2	3	4	5	Average	1	2	3	4	5	Average	5
B	+1	+1	+2	+2	+1	1.4	-2	-2	-2	-1	-2	-1.8	
C	+2	+1	+2	+1	-1	1.4	-2	-1	-2	-1	-2	-1.6	
10 I	+1	0	0	+1	0	0.4	-1	0	-1	0	-1	-0.6	10
K	0	+1	0	+1	+1	0.6	0	-1	0	-1	0	-0.4	
15 R	+2	+2	+1	+2	+1	1.6	-2	-2	-2	-2	-2	-2.0	15

Evaluation standards:

-2 : Soft and sticky

-1 : Slightly soft

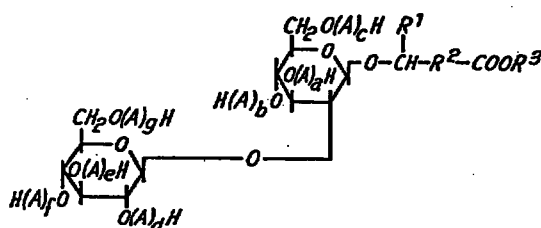
0 : Smooth and good spreading

+1 : Slightly hard and bad spreading

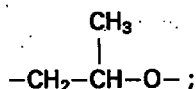
+2 : Hard and difficult to color

25 CLAIMS

1. A skin cosmetic material in the form of a composition including a hydroxypropyl-etherified glycolipid ester represented by the formula,



wherein R^1 represents a methyl group or a hydrogen atom; R^2 represents a saturated or unsaturated acyclic hydrocarbon group having 11 to 15 carbon atoms when R^1 is a methyl group, or a saturated or unsaturated acyclic hydrocarbon group having 12 to 16 carbon atoms when R^1 is a hydrogen atom; A represents the group



R^3 represents a saturated acyclic hydrocarbon group having 1 to 20 carbon atoms, or an unsaturated acyclic hydrocarbon group having 2 to 20 carbon atoms, or $-(A)_h\text{H}$; and a, b, c, d, e, f, g and h are integers, whose sum is in the range of from 1 to 60.

2. A skin cosmetic material as claimed in Claim 1, of the emulsion-solution, compressed powder or stick-shaped type.

3. An emulsion-solution skin cosmetic material as claimed in Claim 2, wherein said hydroxypropyl-etherified glycolipid ester forms from 0.1 to 100% by weight of the oily substance present in the cosmetic material.

4. A compressed powder cosmetic material as claimed in Claim 2, wherein said hydroxypropyl-etherified glycolipid ester is present in an amount of 0.1 to 10% by weight of the cosmetic material.

5. A stick-shaped cosmetic material as claimed in Claim 2, wherein said hydroxypropyl-etherified glycolipid ester is present in an amount of 0.1 to 10% by weight of the cosmetic material.

6. A skin cosmetic material as claimed in Claim 1 substantially as hereinbefore described with reference to any of the Examples.

Table 18

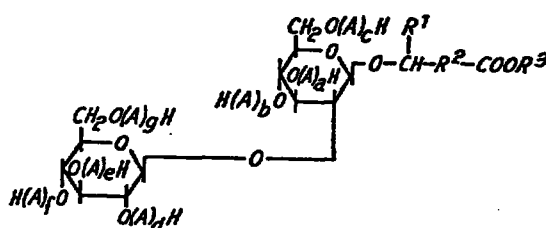
5°C							40°C						
5	1	2	3	4	5	Average	1	2	3	4	5	Average	5
B	+1	+1	+2	+2	+1	1.4	-2	-2	-2	-1	-2	-1.8	
C	+2	+1	+2	+1	-1	1.4	-2	-1	-2	-1	-2	-1.6	
10 I	+1	0	0	+1	0	0.4	-1	0	-1	0	-1	-0.6	10
K	0	+1	0	+1	+1	0.6	0	-1	0	-1	0	-0.4	
15 R	+2	+2	+1	+2	+1	1.6	-2	-2	-2	-2	-2	-2.0	15

Evaluation standards:

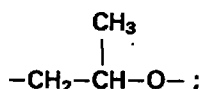
- ation standards:
- 2 : Soft and sticky
 - 1 : Slightly soft
 - 0 : Smooth and good spreading
 - +1 : Slightly hard and bad spreading
 - +2 : Hard and difficult to color

25 CLAIMS

1. A skin cosmetic material in the form of a composition including a hydroxypropyl-etherified glycolipid ester represented by the formula,



wherein R¹ represents a methyl group or a hydrogen atom; R² represents a saturated or unsaturated acyclic hydrocarbon group having 11 to 15 carbon atoms when R¹ is a methyl group, or a saturated or unsaturated acyclic hydrocarbon group having 12 to 16 carbon atoms when R¹ is a hydrogen atom; A represents the group



R³ represents a saturated acyclic hydrocarbon group having 1 to 20 carbon atoms, or an unsaturated acyclic hydrocarbon group having 2 to 20 carbon atoms, or $-(A)_nH$; and **a**, **b**, **c**, **d**, **e**, **f**, **g** and **h** are integers, whose
50 sum is in the range of from 1 to 60.

2. A skin cosmetic material as claimed in Claim 1, of the emulsion-solution, compressed powder or stick-shaped type.

3. An emulsion-solution skin cosmetic material as claimed in Claim 2, wherein said hydroxypropyl-etherified glycolipid ester forms from 0.1 to 100% by weight of the oily substance present in the cosmetic material.

4. A compressed powder cosmetic material as claimed in Claim 2, wherein said hydroxypropyl-etherified glycolipid ester is present in an amount of 0.1 to 10% by weight of the cosmetic material.

5. A stick-shaped cosmetic material as claimed in Claim 2, wherein said hydroxypropyl-etherified glycolipid ester is present in an amount of 0.1 to 10% by weight of the cosmetic material.

6. A skin cosmetic material as claimed in Claim 1 substantially as hereinbefore described with reference to any of the Examples.