

Proliposome-coated Mica With Bioactives for Benefits in Facial Powder



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KEYWORDS: *proliposome, liposome, mica, skin care, actives, facial powder*

ABSTRACT: *The article discusses the preparation of proliposome-coated mica harboring bioactives from plant origin that have skin-whitening/-lightening effects. The treated mica is incorporated into facial powder for skin-smoothing, -moisturizing and -lightening properties, which are evaluated here.*

Mica is a naturally occurring silicate mineral. It is a fine, off-white and pearly powder and in cosmetics, it is used to impart a shiny and transparency appearance to products. This is due to the fact that mica adheres well to skin and reflects light, making skin appear more luminous. Therefore, it is the main ingredient applied in facial powders, especially those that are transparent.

Found in many women's makeup bags, facial powder is one of the most essential cosmetic products. It is generally available in two forms, loose and pressed, and may provide full or transparent coverage. Full-coverage

powders predominantly contain talc (hydrated magnesium silicate) with other covering substances including titanium dioxide, kaolin, magnesium carbonate, magnesium stearate, zinc stearate and cornstarch to increase opaqueness. Some of these ingredients may also provide additional functions. For example, magnesium carbonate can improve oil blotting, keep the powder fluffy and absorb any added perfume; kaolin (hydrated aluminium silicate) may help absorb oil and perspiration. Today's transparent powders are based on the same formula as full-coverage powders but include less talc and covering pigments. Instead, their light,

shiny appearance is produced by nacreous pigments such as mica, bismuth oxychloride, titanium dioxide-coated mica or crystalline calcium carbonate.¹

The main purpose of facial powder is to improve the appearance of skin, and the application of powder tends to be an easy and fast way to cover complexion imperfections, absorb grease and oils, and give a smooth, matte finish to the face. Due to this convenience, facial powder was chosen as the product form into which proliposome-coated mica without bioactives (PCM) and with bioactives (PCMB) were incorporated, to provide skin care benefits beyond camouflage effects.

Proliposomes

Proliposomes are prepared by drying liposome dispersions. Generally, liposomes are composed of one or more phospholipid bilayers and have an aqueous cavity that can entrap, protect and deliver both water- and oil-soluble substances.^{2, 3} In the present study, hop cone and myrobalan fruit extracts, which are evidenced to have antioxidant activity and claimed to have skin-whitening/-lightening

Table 1. Studied botanical extracts

Ingredient	Description	Clinical claim
Hop cone extract	A regulator of pigment disorders by inhibiting the cytokine GM-CSF (activator of melanogenesis)	Improves skin luminosity and decreases skin hyperpigmentation when a cream containing hop cone extract is applied twice daily
Myrobalan extract	A UV absorber (220–280 nm) and antioxidant and tyrosinase inhibitor; can also inhibit MMP-1	Improves melasma lesions and increases skin-lightening when a cream containing myrobalan extract is applied twice daily

potential (see **Table 1**),⁴⁻⁷ were loaded into liposomes.

While a number of studies have shown that liposomes can provide moisturizing benefits when applied to skin,^{8,9} one problem they face in liquid form is instability, i.e., the degradation of phospholipids by oxidation and hydrolysis.¹⁰ To increase stability, liposome dispersions are thus extensively dried by freeze- or spray-drying to transform liposomes to dry powder proliposomes. Since spray-drying is a less time-consuming process that is practical for large-scale production,^{11,12} this method was chosen for the present study to transform dispersions of liposome-coated mica without bioactives and with bioactives to PCM and PCMB, respectively, which were then employed in facial powders. The formulated facial powders were subsequently tested for skin-smoothing, moisturizing and lightening effects, described here.

Materials and Methods

Liposome dispersions: Liposome vesicles were first prepared. A lecithin mixture^a was combined with hop cone extract^b and dispersed, hydrated with water containing myrobalan fruit extract^c, and homogenized (10,000 rpm, 10 min) to produce forced lecithin hydration and subsequent liposome formation. These concentrated liposomes were diluted with water to give the final concentrations of hop cone extract and myrobalan fruit extract in liposome dispersions of 10% and 2.5%, respectively. Liposomes without bioactive loading served as the control. The mean particle size and size distribution

^a Pro-Lipo Duo (INCI: Lecithin (and) Ethyl Alcohol (and) Glycerol) is a product of Lucas Meyer, France.

^b Wonderlight (INCI: Humulus Lupulus (Hops) Strobile) is a product of Sederma, France.

^c SJ Chubulia (INCI: Terminalia Chebula Fruit Extract) is a product of SJI, Thailand.

of loaded and unloaded bioactive liposome dispersions were then measured by laser diffraction^d; the mean particle size was expressed as volume diameter of 50% [d (0.5)], whereas the size distribution was described by SPAN value—a statistical parameter used for evaluating particle size distribution. Images of the dispersions were further observed by a light microscope^e equipped with a digital camera.

Proliposome coated mica: As noted, liposome dispersions with and without bioactives were used for mica coating to prepare proliposome-coated mica. After mica^f was thoroughly suspended in the dispersions described, the suspensions were separately spray-dried using a mini spray-dryer^g with the final outlet temperature of 85°C. The resulting powders were measured for mean particle size, again by laser diffraction. Non-coated mica was also measured via the same technique, for comparison. These substances were further observed for their surface morphology using a scanning electron microscope (SEM)^h.

Moisturization studies: In a moisturization study, PCM was compared with non-coated mica to determine whether the hydration potential of PCM was based on its liposome content rather than its actives. Twenty volunteers applied 2 mg/cm² of the two test substances on each on their forearms, and both treated and untreated areas were measured by corneometerⁱ for moisture levels at 0, 1, 2, 4, 6 and 8 hr.

^d The Mastersizer 2000 is manufactured by Malvern Instruments, UK.

^e The Olympus CX31 is manufactured by Olympus, Japan.

^f Sericite GMS-4C is a product of Kinsei Matec, Japan.

^g The Büchi-B290 mini spray dryer is manufactured by Büchi, Switzerland.

^h The TM-3000 SEM is manufactured by Hitachi, Japan.

ⁱ The Corneometer CM 820 is manufactured by CK Electronic, Germany.

Formula 1. PCM, PCMB and non-coated mica facial powder

Methicone-coated Talc PCM, PCMB or Non-coated Mica	52.48% w/w 20.00
Titanium Dioxide	7.00
Aluminium Starch Octenylsuccinate	3.50
Nylon-12	2.50
Polymethyl Methacrylate	2.00
Magnesium Stearate	1.50
Iron Oxides (CI 77491, CI 77492, CI 77499)	1.65
Mica (and) Titanium Dioxide (and) Barium Sulfate	1.00
Boron Nitride	0.50
Ethylhexyl Methoxycinnamate	3.00
Caprylic/Capric Triglyceride	3.00
Octyldodecyl Stearoyl Stearate	0.50
Squalane	0.50
Caprylyl Glycol	0.80
Fragrance (<i>parfum</i>)	0.05
Tocopherol	0.02
	100.00

Statistical evaluations were made by one-way analysis of variance at a 95% confidence level.

Penetration studies: The capability of PCM and PCMB to penetrate the stratum corneum in comparison with non-coated mica was also assessed. The studies were carried out by tape-stripping. As a preliminary study, five separate volunteers were evaluated. Two hours after volunteers applied the test substances, adhesive tapes were used to sequentially remove layers of the stratum corneum from their forearms. These samples were observed under SEM, and the fifth strip was chosen for photographing.

Facial powder: PCM, PCMB and non-coated mica were separately

formulated into facial powders (see **Formula 1**). After homogeneous mixing, the bulks were separately pressed and compacted in preparation for application to the face.

Sensory studies: Sensory evaluations were performed in 20 volunteers, ages 20–50, using only non-coated mica-containing and PCMB-containing facial powders. After applying each of the two powders to one-half of their faces, subjects scored the powders for spreadability, brightness, coverage, duration, smoothness and uniformity. The mean score of these self-evaluations was expressed in a radar chart. Additionally, the smoothness of PCMB and non-coated mica facial powders was

tested on artificial black skin^k made from double layers of urethane elastomer, and also on human forearm skin. The character of the powder film after application was visually observed and photographed.

Lightening efficacy: Twenty volunteers, ages 18–50, with medium and dark skin were evaluated for the lightening efficacy of the PCMB facial powder. Before any test powder was applied, volunteers' skin was measured by chromameter^m for skin lightness (L^*). The test PCMB facial powder was

^k Bio Skin Plate is manufactured by Beaulax, Japan.

^m The Chromameter CR 400 is manufactured by Konica Minolta, Japan.

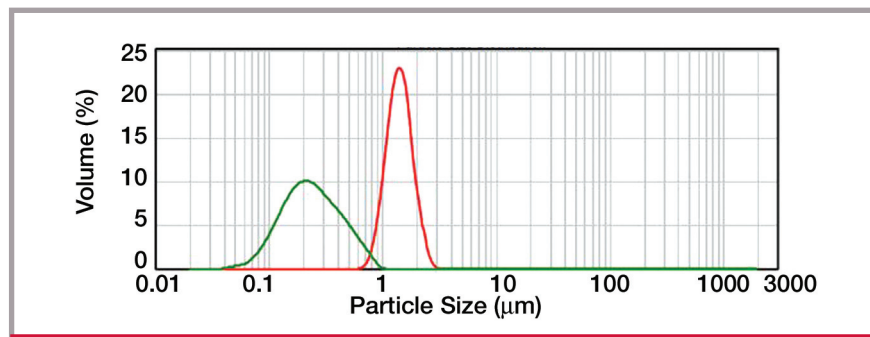


Figure 1. Particle size distribution of liposome dispersions, green = without bioactives and red = with bioactives

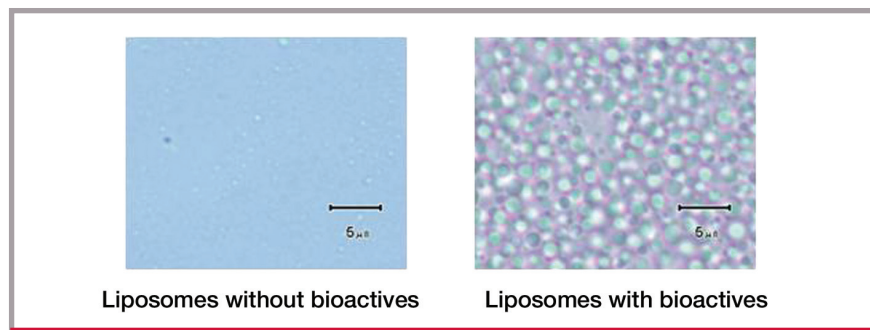


Figure 2. Particle images of liposomes without and with bioactives (1000X)

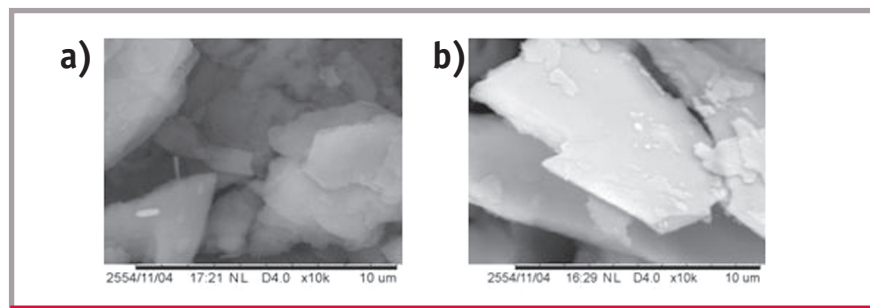


Figure 3. SEM images of a) non-coated mica and b) PCMB (10,000X); proliposome-coated mica with bioactives = PCMB

then applied to the whole face twice daily for up to four weeks. Subjects' facial skin was again measured by chromameter for L^* at weeks two and four; statistical significance was conducted by a student's paired *t*-test at 95% confidence level.

Data and Discussion

Liposome dispersions: The appearance of liposome dispersions with and without bioactives was opaque (milky) and translucent yellowish, respectively. The particle size of the former was shown to be larger than the latter. **Figure 1** reveals that the particle sizes and size distributions of liposomes with and without bioactives were 1.411 μm and 0.231 μm , respectively, with the SPAN value of 0.65 and 1.744, respectively. This means the size of the liposomes is likely to be influenced by the loaded bioactives.

Under the light microscope, liposome dispersions with and without bioactives were both detected to be spherical, as shown in **Figure 2**. In this figure, the larger size of the former is also shown.

Proliposome-coated mica: From liposome dispersions, PCM and PCMB were prepared. The appearance of both powders after spray-drying was off-white. Non-coated mica was used as the control throughout the study. **Table 2** on Page 184 shows that the particle sizes and size distributions of non-coated mica, PCM and PCMB were 11.232 μm , 12.763 μm and 20.130 μm , respectively, with the SPAN value of 1.841, 2.252 and 2.282, respectively. Again, the mean particle size of PCMB was shown to be larger than that of PCM.

Under SEM, the difference between PCM and non-coated mica was difficult to detect. PCMB was differentiated visually from non-coated mica, as shown in **Figure 3**. The smoother edges and surface were observed in PCMB, which would be due to the thin film formation of proliposomes after coverage on the mica surface. This appearance was primarily expected to make PCMB be able to provide smoother application on skin.

Moisturization studies: In the hydration study comparing PCM with

non-coated mica, **Figure 4** reveals that the moisture content of skin to which PCM was applied increased significantly, compared with the application of non-coated mica and the untreated area that served as the control ($p < 0.05$). This improvement in skin hydration was remarkably detected within the first two hours of product application. The contribution could be attributed to proliposomes in the PCM that were hydrated by skin moisture to make them act as liposomes in forming film on skin to protect and provide moisturizing benefits^{8,9} These results seem to support the moisture-providing benefit

of PCMB in facial powder.

Penetration studies: In studying the penetration capability of PCMB, PCM and non-coated mica into the stratum corneum, results showed that non-coated mica was not observed on the fifth strip of adhesive tape. This is likely because mica does not show penetrating ability when applied to skin. On the contrary, PCMB was observed on the fifth tape strip. This would be due to the proliposomes being hydrated by skin moisture to make them behave as liposomes and penetrate into the stratum corneum. PCM was unfortunately undetected due to its small size and the

limited magnifying power of the SEM. With the SEM model used, the highest magnification was 30,000 times and the particle size of the PCMB was 1.411 μm while that of the PCM was 0.231 μm . **Figure 5** therefore reveals SEM images of the fifth tape strips taken from PCMB and non-coated mica applied areas only.

It was therefore reasoned that the penetration of liposomes with the bioactives, obtained from hydrated PCMB, could provide facial powders with the ability to deliver bioactives into the skin. To confirm the penetration capability of PCMB in terms of liposomes with bioactives, new studies will be conducted to remove deeper skin layers by adhesive tape strips, as will new in vitro tests be conducted to evaluate the permeation of the extracts in PCM into the skin via the Franz diffusion cell technique.

Sensory studies: The sensory evaluations of PCMB and non-coated mica facial powders were plotted in a radar chart (see **Figure 6** on Page 186), which shows that volunteers preferred the PCMB powder for its smoothness first, followed by its uniformity and spreadability. This ability of PCMB to impart a smooth film in facial powder was confirmed by testing it on artificial black skin and human forearm skin using non-mica coated facial powder as the compared product. **Figure 7** on Page 186 reveals that PCMB imparted a smoother film on both substrates.

Lightening efficacy: On Page 186, **Figure 8** shows the skin-lightening efficacy of the PCMB facial powder before and after application. The lightening effect imparted by the product was found to be significant at week four ($p < 0.05$). This result is likely to support the hypothesis that PCMB facial powder could help improve skin lightening. This outcome would be expected to result from the whitening/lightening botanical extracts entrapped in proliposomes and delivered into skin; however, more studies must be performed to confirm this benefit.

Conclusions

The present study reveals that dry powders of PCM and PCMB can be successfully produced from spray-drying liposome-coated mica without and with bioactives, respectively, the

Table 2. Particle sizes of PCM and PCMB, in comparison with non-coated mica

Item	Particle size (d (0.5), μm)	SPAN value
Non-coated mica	11.232	1.841
PCM	12.763	2.252
PCMB	20.130	2.282

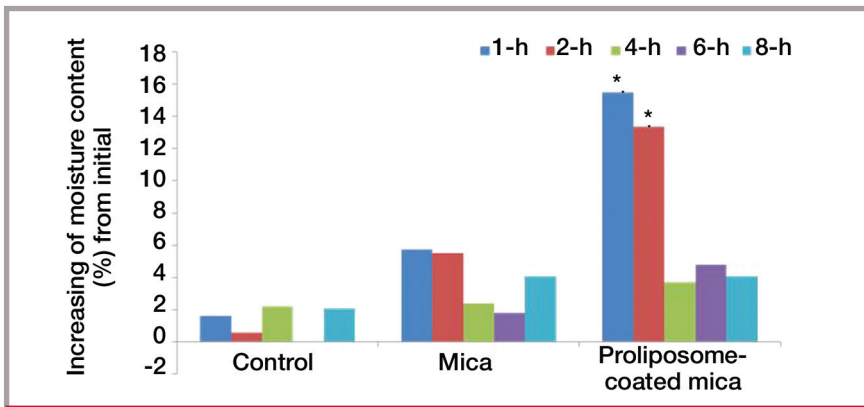


Figure 4. Skin moisturizing effect of PCM compared with non-coated mica and the control on subjects' forearms (* $p < 0.05$); proliposome-coated mica without bioactives = PCM

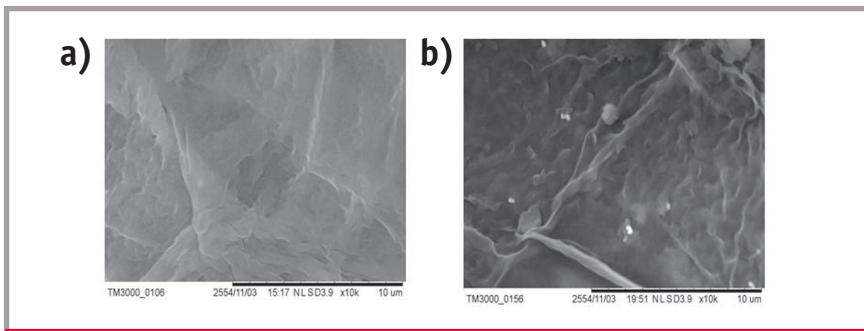


Figure 5. SEM images of the fifth strip taken from the forearm a) with non-coated mica and b) with PCMB (10,000X); proliposome-coated mica with bioactives = PCMB. The PCMB-applied forearm shows the liposomal particles in stratum corneum.

latter of which was shown to have larger particle sizes. When applied to skin, PCMB containing hop cone and myrobalan extracts as skin-whitening/lightening ingredients was shown to have penetration capability through

the stratum corneum and exhibited moisturizing potential.

When incorporated into a facial powder formulation, the product was rated to be the most preferred by volunteers for its smoothness, uniformity

and spreadability, compared with facial powder containing non-coated mica. Interestingly, PCMB facial powder was also found to lighten the skin after four weeks of application.

Results of the described study therefore show that facial powder containing effective, permeable and suitable skin care ingredients can provide skin care as well as camouflage benefits. However, more studies are required to confirm the usefulness of PCMB in facial powder as simultaneous makeup and skin care aspects.

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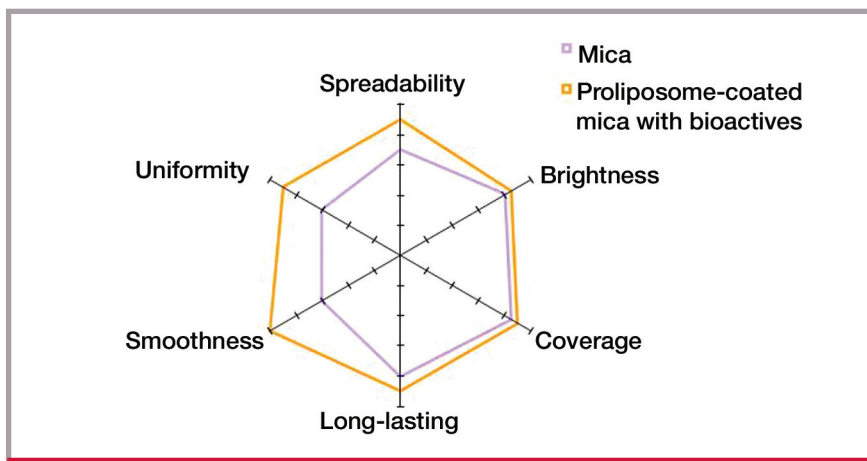


Figure 6. Sensory profile of non-coated mica and PCMB facial powders for each usability evaluation parameter; proliposome-coated mica with bioactives = PCMB

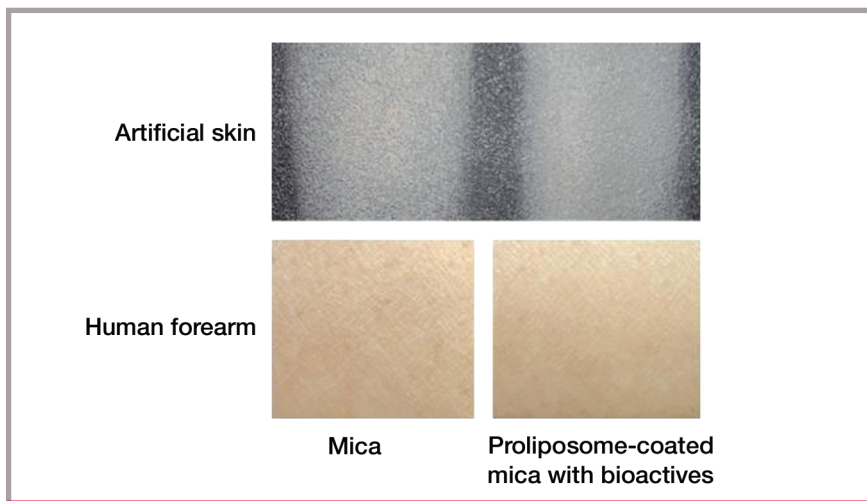


Figure 7. Film smoothness from applications of non-coated mica and PCMB on artificial black skin and human forearm; proliposome-coated mica with bioactives = PCMB

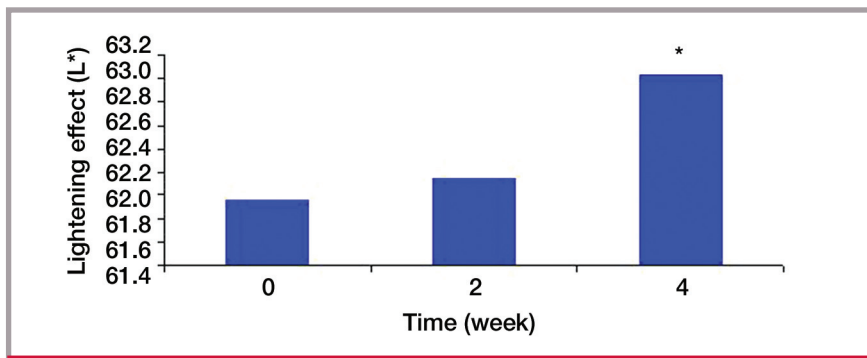


Figure 8. Lightening efficacy of PCMB facial powder after application on the face twice daily, compared with before application (week 0) (* $p < 0.05$)