바륨설페이트/티타늄디옥사이드/디메치콘의 복합화 및 메이크업에서의 응용

최 경 $\overline{\mathtt{p}}^{\dagger} \cdot \mathtt{D}$ 승 용 \cdot 강 학 희 \cdot 이 옥 섭

태평양 기술연구원 화장품연구소

A Study on the Surface-Modification of Barium Sulfate/ TiO_2 /Dimethicone Composite Powder and its Application in Color Cosmetics

Kyung-Ho Choi[†], Seung-Yong Ko, Hak-Hee Kang, and Ok-Sub Lee

Amore Pacific R&D Center, 314-1, Bora-ri, Giheng-eup, Yongin-si, Gyeonggi-do 449-729, Korea

요 약: 감촉 및 광학적 요소는 파우다 파운데이션에 있어서 매우 중요한 요소이다. 이러한 감촉과 광학적인 요소를 동시에 만족하는 분체에 관심을 가지고 다양한 소재 및 제조방법을 연구 중에 바륨설페이트를 기본 입자로 하는 복합화 된 분체를 개발하게 되었다. 바륨설페이트는 입자의 모양이 매우 넓고 표면의 굴곡이 없는 판상으로 감촉면에서 부드러운 사용성과 균일한 부착특성을 나타내며 굴절율도 피부보다 약간 높은 수준으로 인위적이지 않고 자연스러운 광학적 특성을 나타내고 있다. 그러나 파운데이션에서 요구되는 광학적 특성인 자외선 차단능력이나 커버력, 블루밍 효과에 있어서는 기대수준에 못 미치는 것을 알 수 있었다. 이러한 특징은 파우다 파운데이션에서 사용되는 대부분의 분체에 있어서 공통적으로 나타나는 특성으로 감촉적인 측면 (소프트, 밀착력 등)과 광학적 측면(커버력, 자외선 차단력 등)을 동시에 만족하는 분체가 극히 드문 것을 알 수 있다. 본 연구에서는 이러한 점을 극복하고자 감촉적인 측면에서 우수한 바륨설페이트를 모(街)입자로 하고 자외선 차단능력과 블루밍에서 우수하나 자체의 응집력이 강하고 사용감이 거친 특징을 나타내고 있는 티타늄디옥사이드를 자(子)입자로 하여 복합화하려 하였다. 이때 중요 요소로 복합화 되는 티타늄디옥사이드의 함량에 따라 복합안료의 감촉적인 측면과 분산력 등이 민감하게 달라지는 것을 알 수 있었다. 이에 패널을 이용하여 복합안료의 사용성에 영향을 미치지 않는 수준의 함량을 결정하여 복합화 처리했으며, 사용자가 피부에 도포시 발생되는 피지나 땀에 대한 변색과 사용감을 고려하여 피지나 땀에 강한 발수, 발유 능력을 나타내며 부드러운 감촉의 디메치콘으로 복합안료를 코팅 처리하여 복합분체를 만들게 되었다. 이렇게 만든 복합분체(barium sulfate/TiO/dimethicone)를 패널을 이용한 테스트와 기기를 이용한 테스트를 실시하여 효과를 검증하게 되었다.

Abstract: Sensor and optical properties have become critical features in powder foundation. The flaky barium sulfate powder shows good smooth texture, adhesion and natural looking characteristics. However, it has limitations abilities in UV shielding, hiding and blooming effect. Thus we adopt TiO₂ that has excellent hiding power and blooming effect as well as UV shielding ability, but TiO₂ has still intrinsic problems in dispersion and texture. To overcome this disadvantages, the barium sulfate/TiO₂/dimethicone composite powder was prepared. The flaky barium sulfate powder was coated with TiO₂ in nanoscale and followed by coated with dimethicone. When this surface-modified powder was applied for make-up cosmetics, especially in powder foundation, the powder gave powder foundation more good characteristics than the original flaky barium sulfate, TiO₂ powder, dimethicone in abovementioned optical and sensory properties. To characterize the distinctive features of this surface-modified powder, we measured its characteristics with UV in vitro tester, hiding powder test method, goniophotometer, consumer panel test and so on.

Keywords: composite, barium sulfate, TiO2

1. Introduction

In the design of the powder foundation, we have to consider not only touch property, the softness which the consumers feel, but also optical property that means natural cover effect, soft focus, ability of UV screening. But the common powder particles are not satisfied with both touch property and optical property. Moreover, some are contrary to each other. We estimate that the reason is the optical property of powder itself, the

[†] 주 저자 (e-mail: khchoi@amorepacific.com)

morphology and surface properties. Because the powder foundation is applied on all over face which has a keen sense, we must consider both softness and optical properties of the powder particle to make the product that the consumers want. In example, TiO₂ which has excellent optical property, especially good UV screening and cover effect, has bad touch property. The other hand, Mica which has excellent touch property but has bad optical properties (coverage and UV screening).

In this research, we intended to develop the powder particle which has not only good skin fitting and touch feeling but also appropriate levels of cover effect and transparency in the skin. So we plan to make a composite powder with two different powders. As explained in Table 1, we chose the mother particle which is the plate shape because of the considering of similarity of skin reflective index, discoloration property and skin fitting morphology and the son particle which has excellent cover effect because of high reflective index. We used BaSO₄ as the mother particle of plate because of smooth feeling, hardness of turning darkish when it is wetted with sebum or oil. The skin of humans has a reflective index of 1.60. Barium sulfate has the same property, safety inert powder like used in a projective reagent for roentgen. And also we adopt TiO₂ as the son particle because of good coverage, UV shield effects. In the complexation of TiO₂, we developed the particle which exhibit excellent touch property and natural cover effect according to the coarse touchness of TiO2 itself. And we coated the particle with dimethicone for the modification of dispersibility, resistance of discoloration by sebum, prohibition of photocatalyst effect, softness[1]. Through the process, we can incidentally certify that UV screening effect of the particle was improved because of modification of dispersibility according to the complexation of TiO₂

2. Materials and Methods

2.1. Select the Mother Particle and the Son Particle

For the natural cover effect necessary with the powder foundation, the particle which is fitted to the skin through the appropriate area is favorable. So, we chose the some candidate materials, the mother particles, which have a similar reflective index with skin $(RI = 1.5 \sim 1.6)$ considering the morphology of particle. It was corresponded to MICA, BaSO₄, TALC. But TALC was omitted in the candidate materials because of coarse feeling (Talc had more relative coarse feeling). In the next step, we could confirm that the discoloration of BaSO4 was relatively slight and it was maintained as original color. It is the essential function of the powder foundation.

As explained in the Figure 1, BaSO₄ is below 1.5 of △E before and after wetting. But MICA or SYN-THETIC MICA is above 2 of △E. Therefore, BaSO₄ was used as the mother particle considering reflective index, touch property, morphology, and discoloration.

2.2. Making Composite Powder and Control of TiO₂ Dosage

After making of mica slurry by inputting the BaSO₄ to distilled water, the hydrolysis and condensation re-

Synthetic Max

Table 1. Property of Pigment

-	Chemical formula	KAl ₂ (S ₃ Al)O ₁₀ (OH) ₂	KAl ₂ Si ₃ O ₁₀ (OH) ₂	BaSO ₄	Al ₂ O ₃	TiO ₂
-	Property	White fine powder	White fine powder	White fine powder	White fine powder	White fine powder
-	Reflective index	1.552~1.588	1.539~1.589	1.64	1.760~1.768	2.52

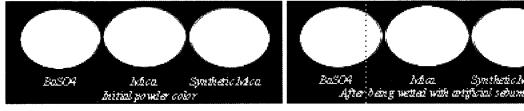


Figure 1. Comparison of discoloration.

action were carried out in $60\sim90^{\circ}\text{C}$ for 10 hr by adding TiOSO₄ solution with shaking the BaSO₄ slurry and simultaneously adding sulphuric acid for the adjusting of pH. After filtration, washing, the calcinations process was carried out in $700\sim800^{\circ}\text{C}[4]$. Finally, after mixing of powder particle and ethanol, projection of dimethicone[2,3], filtration, drying, pulverizing is applied (above process had been done in the cooperation with Chemland (Korea), N.K.K (Japan)).

By the process mentioned above, we manufactured particle coated with 10% (Wt%) of TiO₂, 1% dimethicone. But we got a result of smooth property in touchness test. As a result of test of the particle coated with 6.5% of TiO₂, 2.5% dimethicone, we could get a result of touchness test similar with common TALC.

3. Measurements

3.1. Panel Test

The evaluation of complex particle and compared sample is carried by 5 special panel for two weeks for the touchness testing using in-company panel.

As we can see the result of each samples, touchness properties (coverage, spreadability, smoothness) are depended on the amount of Titanium Dioxide and Dimethicone. So we can decide the component ratio of the composite powder. Table 2 explain that the final complexation particle has the excellent result of cover effect, spreadability, smoothness[2].

3.2. Coverage Test

Compared the discoloration on the face with a common snipe egg, 0.1 g of foundation included with 10% each powder sample was spread and eye measurement

Table 2. Panel Test Result of Each Sample

Sample	Coverage	Spreadability	Smoothness
BaSO ₄	X	0	0
BaSO ₄ /TiO ₂ (90:10)	0	Δ	Δ
BaSO ₄ /TiO ₂ /Dimethicone (89:10:1)	0	0	0
BaSO ₄ /TiO ₂ /Dimethicone (91:6.5:2.5)	0	О	0
TiO ₂	0	X	X

 $X: bad, \triangle: not bad, \bigcirc: good, \bigcirc: very good$

was carried out after sufficient time. The sample used with only TiO_2 had unnaturally hazy coverage and the sample used with only $BaSO_4$ had poor coverage, creation of surface spot.

3.3. Soft Focus Effect Test

We estimated reflection patterns to get a reflection pattern of skin and complexation particle using the Goniophotometer (Goniophotometer GP-200).

Figure 3 explains that complexation particle mentioned above has a similar reflection pattern with skin and that it is not an unnatural coverage but a natural coverage like naked skin. And we could certify the better soft focus index than the other plate-shaped particles.

3.4. In Vitro SPF Test

For the measurement of UV screening effect of the complexed powder particle mentioned above, each sample (TiO₂ 1.3%) was inserted to the powder foundation of same formula and the formulation is carried. After that each samples were measured by using the *in vitro* SPF instrument (the optometric corporation SPF290).

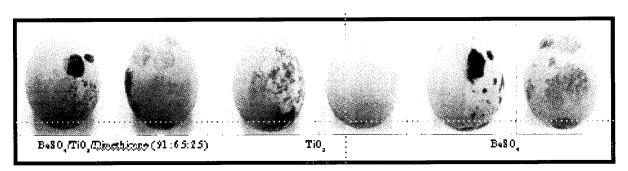
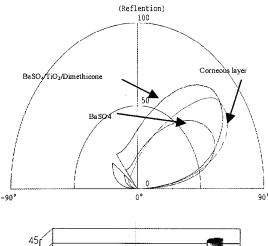
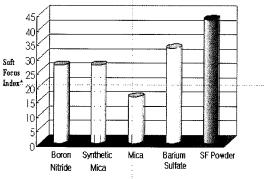


Figure 2. Comparison of coverage.





(* Soft Focus Index = O'reflection / 45'reflection)

Figure 3. Goniometer measurement & soft Focus index of pigment.

Table 3. SPF Comparison in Powder Foundation

Sample name	Wt% of key component	Index of SPF
A	BaSO ₄ (18.2%), TiO ₂ (1.3%)	2.60
В	BaSO ₄ /TiO ₂ /Dimethicone (20%) : Net BaSO ₄ (18.2%), TiO ₂ (1.3%)	3.15

We see that it is different an UV screening effect of complexed powder particle from an UV screening effect of mixed powder particle though the quantity of the ingredients is same. We could presume that complexed powder has more dispersibilty than the original TiO_2 powder.

4. Conclusion

As mentioned above, the plate-shaped $BaSO_4$ which has skin similar reflective index was complexed with TiO_2 and coated with dimethicone could maximize emotional touchness of consumer by controlling the ingredients of TiO_2 . Moreover, the designer could realize the coverage and soft focus effect of desired levels in foundation mixed with the powder particle mentioned above and could certify 20% development of UV screening effect compared with the sample which is added with same quantity of TiO_2 .

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