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복합화기술을 응용한 목어석 복합체의 제조 및 이의 효능에 관한 연구

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The Preparation of Mockeoseuk (China Fossil) Composite by Hybridization Technique and Evaluation of Its Efficacy

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요 약: 목어석(중국화석)은 다양한 종류의 미네랄을 함유하고 있으며, 적외선을 방출하는 광물이다. 이러한 목어석을 화장료에 도입하기 위해서 복합화기술을 도입하였다. 이러한 기제로서 구형의 실리콘 분체를 응용하였다. 복합화된 목어석은 피부 사용감과 같은 물리적 성질이 개선되었으며, 외관상 색깔도 개선되었다. 또한 효능도 유지함을 알 수 있었다. 임상 시험 결과, 10 wt% 목어석 복합체를 함유한 화장료는 대조군 대비 유의적으로 얼굴 피부의 온도를 상승시킴을 확인하였으며, 이 결과는 목어석 복합체가 피부의 혈행 순환을 향상시킬 수 있음을 알 수 있었다.

Abstract: Mockeoseuk (China fossil) contains the various kinds of minerals and radiates far infrared light. In order to apply mockeoseuk to the cosmetic formulation, hybridization technique was adapted and modified by selecting a spherical silicone powder as substrate. The resultant composite improved the physical properties such as skin feeling and apparent color and still sustained the efficacy of mockeoseuk. In a clinical test, the cosmetic formulation with 10 wt% mockeoseuk composite raised the temperature of facial skin through enhancement of skin blood flow.

Keywords: *mockeoseuk, far infrared light, hybridization technique, spherical silicone powder, blood circulation*

1. Introduction

Mockeoseuk is a kind of fossil mined from specific region in China. It has been known that mockeoseuk contains the various mineral sand radiates far infrared light. And it was recorded in the ancient documents, which was described as effective material for enhancing the human health. So, we focused on developing cosmetic material by using mockeoseuk. But this material has the some problems such as intrinsic color and touch feeling on the skin in the case of cosmetic applications. So, we studied the organic-inorganic hybridization technique with various substrates to improve the physical properties of mockeoseuk such as color, spread and adhesion to the skin[1]. And it will be checked that the composite maintains its efficacy simultaneously. To evaluate the efficacy of the composite on the human skin, the variation of blood circulation and change of skin temperature were measured by LDI (laser Doppler image analyser) and IR (infrared rays) camera. Our study is intended to verify whether the mockeoseuk and its composite enhance the blood circulation. This efficacy effects the temperature of facial skin surface.

Finally the mockeoseuk composite will be confirmed to be safe or not on the skin by the various toxi-

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cological tests.

2. Materials and Methods

2.1. Elemental Analysis of Mockeoseuk

For analysis the element of mockeoseuk by using scanning electronic microscope (SEM, Hitachi Co. Ltd., Japan) samples were mounted on adhesive carbon discs. Minerals of mockeoseuk were detected by an energy dispersive X-ray (EDX, Rigaku Co. Ltd., Japan) analysis system.

2.2. Hybridization

Mockeoseuk is crushed by atomizer to reduce particle size. Pulverized particles are dispersed in ethanol solvent. The concentration of particles in ethanol solvent is 10 wt%. And bishydroxyethoxypropyl dimethicone was added to dispersed solution by 0.3 wt% as binder. The binder has the amphiphilic property like as surfactant, and easily adsorbed to the surface of silica, talc, mockeoseuk[2]. Finally, the substrate for hybridization of mockeoseuk was added to the solution. The two types of substrates would be used for hybridization. One is sphere particle such as spherical silica. And the other is flaky particle such as talc. The solution was mixed sufficiently by mechanical stirrer. Then, solution was filtered and dried at 70 ~ 90 ℃ to remove ethanol. The dried particle complex was hybridized by shear force[3,4]. The shear force of hybridization process was accomplished by mechanical tools such as hensel mixer with high speed (Jooshin Industrial Machine, Co. JS-H05, Korea).

2.3. Efficacy Evaluation

To evaluate the efficacy of mockeoseuk composite in the human skin, the quantity of blood circulation and change of skin surface temperature were measured by laser Doppler image analyzer (periscan system, Perimed Co. Ltd., Sweden) and IR camera (Therma CAM researcher 200, USA).

2.4. Safety Evaluation

To estimate the safety of the mockeoseuk and its composite, various safety testes were carried out. These tests included the primary skin test, eye irritation test, human patch test, repeated insult human patch test. All tests were done for subjects according to CTFA (Cosmetic, Toiletry, and Fragrance Association) guidelines.

2.5. Method of Spreadibility and Adhesion to Skin of Mockeoseuk

We selected the force of spread and adhesion among the various sense of the touch. These factors have great relation of frictional force. Therefore, the force of spread is defined as frictional force between some materials, and the force of adhesion is also defined as sticking between skin and pigments. We made standard operating procedures (SOP) about friction tester (KATO TECH Co., Ltd, KES-SE, Japan), rheometer (Sun Scientific Co., Ltd., CR-500DX, USA) in order to measure the spread and adhesion.

3. Results and Discussion

3.1. Characterization of Hybridized Complex

As a result of elemental analysis by SEM-EDX, mockeoseuk contains SiO_2 31.9 %, CaO 28.0 %, MgO 20.5 %, Al_2O_3 11.2 %, Fe_2O_3 4.5 %, K_2O 2.9 % and the small amount of other minerals (AgO, NiO, CuO, N_2O) was detected. In the study of emissivity of far infrared light, emissivity of mockeoseuk was measured by 0.920 (40 ℃), which is the same level of emissivity of jade. Emission power is 3.74×102 w/m^2 (Figure 1).

This means that mockeoseuk radiates a great amount of far infrared rays. But, this material has some problems to introduce it in the cosmetic formulation because of its coarse surface and optical color.

To improve these physical properties, we introduced the hybridization technique of solid particles.

This technique was developed in order to combine more than two particles having different properties one another by applying high shear energy by means of mechanical instrument to the objective particles. In this process, binder can be introduced as the case may be so as to increase the physical adhesion force of inter particles.

Figure 2 shows the mechanism of hybridization between spherical silicone powder and mockeoseuk particle. When applying high shear force to mixture of two particles, the agglomerates of spherical silicone powder which seem to be grape-shaped is separated

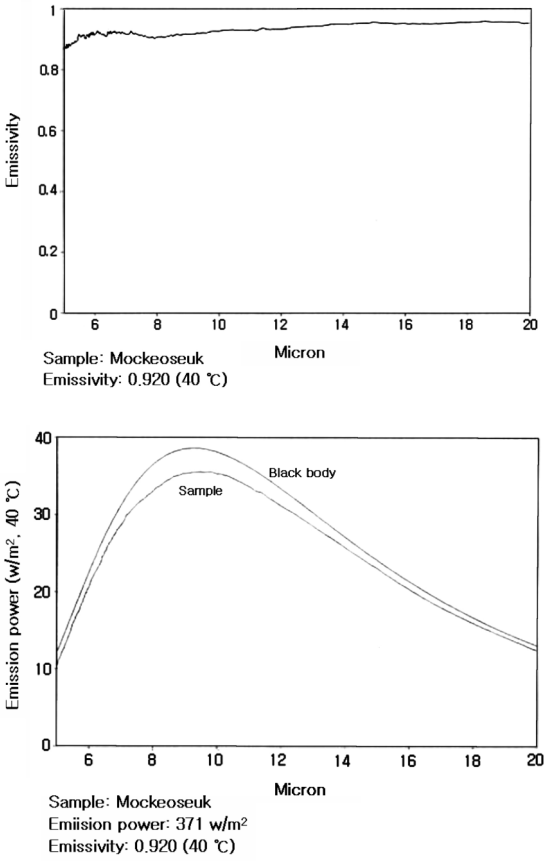


Figure 1. Characterization of far infrared ray of mockeoseuk (Emissivity : 0.920 (40 ℃), Emission power : 374 w/m^2).

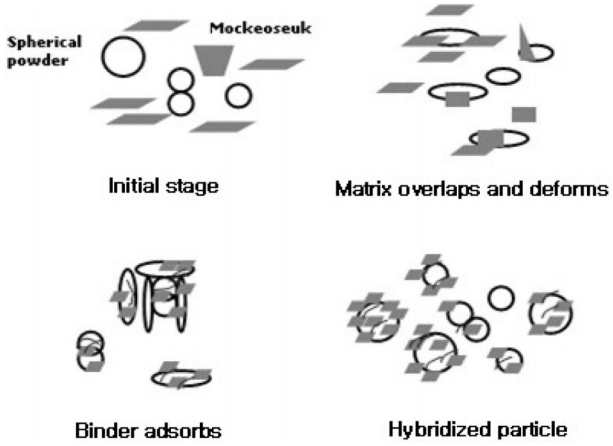


Figure 2. Hybridization mechanism of spherical silicone powder/mockeoseuk composite.

Table 1. The Change of Luster by the Hybridized Ratio of Mockeoseuk and Silicone Powder

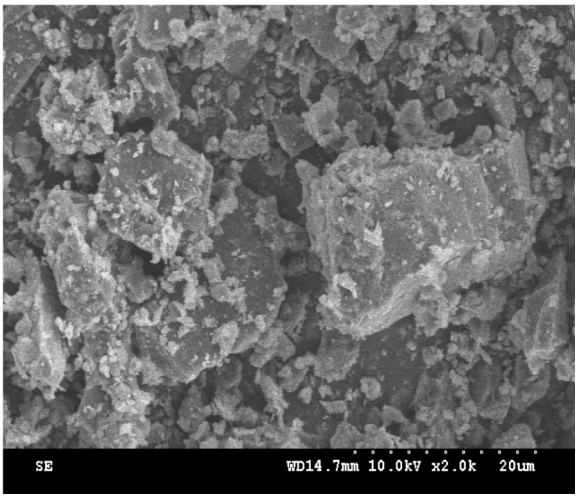
	D (Diffusion light)	S (Specular gloss)	L (S/D) Luster
Mockeoseuk	29.81	64.04	2.14
Mockeoseuk : Silicone powder (1 : 1)	24.9	62.61	2.51
Mockeoseuk : Silicone powder (1 : 2)	22.31	61.16	2.74
Mockeoseuk : Silicone powder (1 : 6)	20.9	54.11	2.59
Silicone powder	18.53	57.77	3.11

into independent spherical particle. The size of mockeoseuk particle is simultaneously reduced to about 0.5 ~ 2.0 μm.

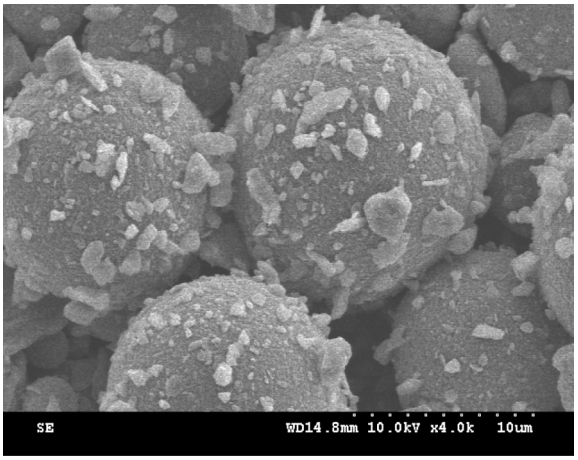
Finally, the matrix of two particles is formed and the binder is distributed homogeneously onto two particles. In the next step, high shear force is applied to the matrix once more. This step needs to be supplied high energy sufficiently to adhere small fragments of mockeoseuk particle to the surface of separated spherical silicone particles. As a result of these processes, hybridized particles were prepared. And the stable structure was maintained in the various conditions.

The morphology of hybridized particle was confirmed by SEM. Figure 3 shows that the great part of small mockeoseuk particle stably adheres to the surface of silicone powder. The binder reinforces the adhesion between two kinds of particles. The spread and adhesion to the skin of mockeoseuk composite were improved. The value of friction factor was 91 although the value of mockeoseuk was 125. And the adhesion value of composite decreased to 2.5 in comparison to mockeoseuk. These results convinced that the composite can be applicable in the cosmetic formulation.

We studied the change of glossiness of mockeoseuk composite as the weight ratio of silicone powder to mockeoseuk increases. The goniophotometer is able to measure the reflection light exactly at all angles according to the input light beam. The reflection light at 45 degrees has the proportional relation with glossiness. This reflection is called as specular gloss[5]. And luster value is defined as reflection light at 45 degrees divided by diffusion light. Table 1 shows the luster value increases to 2.59 when the hybridized ratio of silicone powder to mockeoseuk is 6. This result means that the color of mockeoseuk composite moves from



(a)



(b)

Figure 3. SEM image of mockeoseuk particle (a), mockeoseuk composite (b).

deep reddish to pale and glossy reddish because the silicone powder has high luster intrinsically in comparison to mockeoseuk.

3.2. Efficacy Evaluation

It was mentioned preliminarily that mockeoseuk has an ability of emission of far infrared light. And this material has known to have a various efficacy with relation to skin disease mainly by blood circulation.

Before the evaluation of efficacy, we checked the safety. As a result of the primary skin test, eye irritation test, maximum mockeoseuk concentration of non-irritation was 5 wt% in the vehicle. In the human patch test, repeated insult human patch test, mockeoseuk showed the safe results.

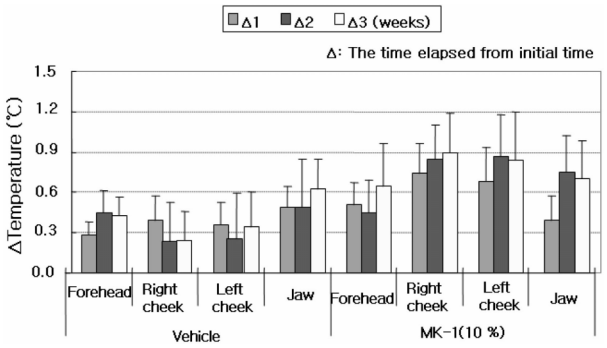


Figure 4. Temperature change of the skin surface after vehicle (control) sample and sample with mockeoseuk composite was applied respectively for 3 weeks.

In this study, the efficacy of mockeoseuk composite in the cosmetic formulation was evaluated by measuring the temperature of facial skin. The mockeoseuk composite (the weight ratio of silicone powder to mockeoseuk is 6. this sample is named MK-1.) was added to 10 wt% in foundation formulation. The concentration of mockeoseuk in the formulation is within permissible range which was decided in preliminary safety tests. Female was selected for clinical test of the temperature change on the facial skin. The number of female for clinical testing group is 20.

Evaluation procedure of efficacy is as follows. Clean the face entirely with cleansing cream. Stand by for 20 min in the room in the condition of constant temperature, constant humidity (temperature : 24 ± 2 ℃, relative humidity : 40 ± 2 %). Measure the change of skin temperature with IR camera (Thermo CAM researcher 200) every week for 3 weeks[6]. The group of 10 persons which foundation formulation containing 10 wt% of mockeoseuk composite was applied to show that the temperature of facial skin rise up for 3 weeks uniformly in comparison to the other group of 10 persons which was applied to control formulation which does not contain mockeoseuk ($p < 0.05$, paired t-test). Especially, the large change of temperature was observed in the cheek as compared with other area of face (Figure 4). This result of clinical test proves that the physical properties of mockeoseuk effect bloodstream or blood circulation of facial skin.

The enhancing of bloodstream is expected to increase

the temperature of skin surface and the experimental results confirmed this assumption. And it is very important that hybridized particle of mockeoseuk which is prepared to apply to cosmetic formulation easily sustains its efficacy in the condition of cosmetic surroundings. Finally, mockeoseuk composite was proven not to be irritant by means of various toxicological tests.

4. Conclusions

1) Preparation of mockeoseuk composite with spherical silicone powder by hybridization technique was confirmed by SEM.

2) The skin feeling of mockeoseuk composite was enhanced as a result of increased spreadibility, adherence to skin.

3) Intrinsic color of mockeoseuk is changed to pale reddish color and the glossiness increased as the ratio of silicone powder increase

4) Application to cosmetic formulation of mockeoseuk composite is possible to be used easily.

5) When the formulation containing 10 wt% of mockeoseuk composite was applied to the skin surface, the skin temperature rose up.

6) Mockeoseuk composite was proven not to be irritant in various toxicological tests. These results show that this composite material can be safely introduced to the skin care formulation.

References

1. A. Mujumdar and D. Wei, Improvement of humidity resistance of magnesium powder using dry particle coating, *Powder Tech.*, **140**, 86 (2004).
2. G. Parashar, D. Srivastava, and P. Kumar, Ethyl silicate binders for high performance coatings, *Progress in Organic Coatings*, **42**, 1 (2001).
3. T. Iwasaki, M. Satoh, and S. Ichio, Preparation of a UV-attenuating agent using a mechanochemical reaction, *J. Materials Processing Tech.*, **142**, 131 (2003).
4. C. Suryanarayana, E. Ivanov, and V. V. Boldyrev, The science and technology of mechanical alloying, *Material Science and Engineering*, **A304**, 151 (2001).
5. M. Gunde, M. Kunaver, and M. Cekada, Surface analysis of matt powder coatings, *Dyes and Pigments*, **74**, 202 (2007).
6. F. Charlot, F. Bernard, and E. Gaffet, In situ time-resolved diffraction coupled with a thermal i.r. camera to study mechanically activated SHS reaction: case of Fe - Al binary system, *Acta. Mater.*, **47**, 619 (1999).