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"Functional Composite Powders for Cosmetic Ingredients Using Novel Manufacturing Technology"

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1. Introduction

Metallic soaps are white powders composed of fatty acids covalently or ionically bonded to metals like calcium (Ca), magnesium (Mg), or zinc (Zn). Their properties, including shape, melting point, and solubility, vary with the type of metal, fatty acid chain length, production method, and purity. Industrially, metallic soaps using long-chain saturated fatty acids (C12–C22) are widely used as functional additives in fields such as release agents for rubber and plastics, dispersants for pigments, and pharmaceutical lubricants. In cosmetics, they enhance pigment dispersion and water resistance, making them suitable as base materials for products like foundations, with a long history of use [1-3].

Cosmetic metallic soaps are typically produced through the double decomposition process, which involves creating a soap solution from fatty acids and alkali metal compounds, followed by a metal exchange reaction with non-alkali metal salts in water to form a metallic soap slurry. This process, called the wet method, yields powders with uniform particle sizes (several micrometers to over ten micrometers) and allows control of particle size and shape [4].

However, excessive metallic soap in formulations can cause caking and worsen texture due to excessive packing from their fatty acid-bonded structure. Addressing these issues through raw material or processing changes has been challenging. To overcome this, we hypothesized that combining metallic soaps with low-packing powders could suppress aggregation and improve usability.

Plate-shaped glass powder was selected for its low packing tendency, ability to improve texture, and plate-like shape that facilitates metallic soap growth. Generating metallic soaps on the surface of dispersed inorganic powders was proposed as an effective method to achieve greater homogeneity compared to premixed powders.

To validate this, we developed and evaluated composite powders combining metallic soaps and inorganic powders, aiming to address the limitations of conventional metallic soaps. Additionally, the composite powders were compared with metallic soaps alone to confirm their advantages. A comparative evaluation with mica, a widely used mineral-based powder with functional benefits but some safety and social concerns, was also conducted [5]. If the composite powder exhibits comparable properties to mica, it could serve as a viable substitute.

2. Materials and Methods

2-1. Manufacturing of Composite Powders

2-1-1. Materials Used

Metallic Soap: Magnesium myristate, which has a proven track record as a cosmetic raw material, was selected. To achieve in-situ composite formation during the production of the metallic soap, sodium myristate and a water-soluble magnesium salt, the raw materials for the metallic soap, were used.

Inorganic Powder: From plate-shaped powders with low packing tendencies with metallic soaps and a history of use as cosmetic raw materials, plate-shaped glass was selected.

2-1-2. Manufacturing Method

A soap solution of sodium myristate, a component of the metallic soap, was prepared. While stirring the sodium myristate solution, the selected inorganic powder was added and mixed to achieve uniform dispersion without sedimentation. Maintaining the stirring state, an aqueous solution of metal salt, another component of the metallic soap, was added dropwise at a constant rate. After completing the addition, thorough stirring was performed to ensure the complete formation of the metallic soap. The resulting slurry was filtered and washed to remove any unreacted metallic salts or soap residues. The filtered cake was dried, and the aggregated particles were broken down to achieve a uniform particle size distribution.

2.2 Observation of the Composite Process

Plate-shaped glass was introduced into the soap solution and stirred thoroughly to create a uniformly dispersed solution. The solution was then filtered, washed thoroughly with water, and dried. The resulting powder was examined using a scanning electron microscope equipped with energy dispersive X-ray spectroscopy (SEM-EDX) (JEOL JSM-IT300HR). A small amount of the sample was placed on carbon tape, coated with platinum to impart conductivity, and prepared for observation. Under high vacuum and a 15kV acceleration voltage, the presence ratios of silicon (a major element of plate-shaped glass) and carbon (from sodium myristate) were measured, and the average of five measurements was calculated. For comparison, untreated plate-shaped glass and powders produced by dry mixing of plate-shaped glass with soap, followed by filtration, washing, and drying, were also evaluated similarly.

2.3 Observation of Composite Powders

The composite state of the resulting powders was confirmed through the following evaluations: Particle observation was conducted using a scanning electron microscope (SEM) (JEOL JSM-IT300HR). A small amount of the sample was placed on carbon tape, coated with platinum to impart conductivity, and prepared for observation. Observations were conducted under high vacuum and a 1.5kV acceleration voltage. Additionally, to evaluate changes in particle size due to composite formation, particle size measurements were performed using a laser diffraction scattering particle size distribution analyzer (MICROTRAC MT3300II).

2.4 Evaluation of Aggregation

To assess the tendency of powders to harden due to composite formation, the aggregability of the powders was measured using a powder characteristics testing device (Hosokawa Micron PT-X). The powder was placed on the top sieve of a three-tier sieve system and subjected to a specified vibration intensity for a predetermined time. The amount of powder remaining on each sieve was used to calculate the degree of aggregation using the following formula:

Aggregation Degree (%) = X + Y + Z

X = (mass of powder retained on the top sieve) / 2 × 100
Y = (mass of powder retained on the middle sieve) / 2 × 100 × 3/5
Z = (mass of powder retained on the bottom sieve) / 2 × 100 × 1/5

2.5 Viscoelastic Evaluation of Oil Mixture Samples

To evaluate the flowability and ease of removal when the composite powder is used in cosmetic formulations containing oils, shear tests were conducted using a rheometer (Anton Paar MCR302). A putty-like mixture with a 6:4 weight ratio of powder to liquid paraffin was molded into a 25mm diameter sample for measurement. Parallel plates were used for analysis, and shear stress and loss tangent (tan δ) were evaluated. The loss tangent was calculated using the following formula:

$\tan \delta = G'' / G'$

G”: Loss modulus
G’: Storage modulus

2.6 Texture Evaluation

To confirm changes in texture due to composite formation, the feel of the powders when applied to the skin was evaluated. Five researchers applied the test powders to the back of their hands and rated them on four criteria: spreadability, smoothness, glossiness, and whiteness, using a five-point scale. Metallic soap alone and mica were similarly evaluated for comparison.

3. Results

3-1. Observation of the Composite Process

To confirm the surface state of plate-shaped glass dispersed in the soap solution, the plate-shaped glass was extracted from the solution and subjected to elemental analysis. It was hypothesized that dispersing the glass in the soap solution would lead to soap adsorption on the glass surface, as shown in Figure 1, enabling efficient composite formation. Elemental analysis of the plate-shaped glass extracted from the sodium myristate solution revealed that the carbon content (originating from sodium myristate) relative to silicon (the primary element of the glass) was 22.9% (Figure 2). For comparison, untreated plate-shaped glass showed a carbon content of 3.2%, while plate-shaped glass subjected to dry mixing with soap showed 4.2%. These results indicate that dispersing the glass in the soap solution significantly increases the presence of soap on the glass surface. Based on these findings, it was suggested that generating metallic soap while dispersing plate-shaped glass in sodium myristate solution could efficiently produce metallic soap on the surface of the glass.

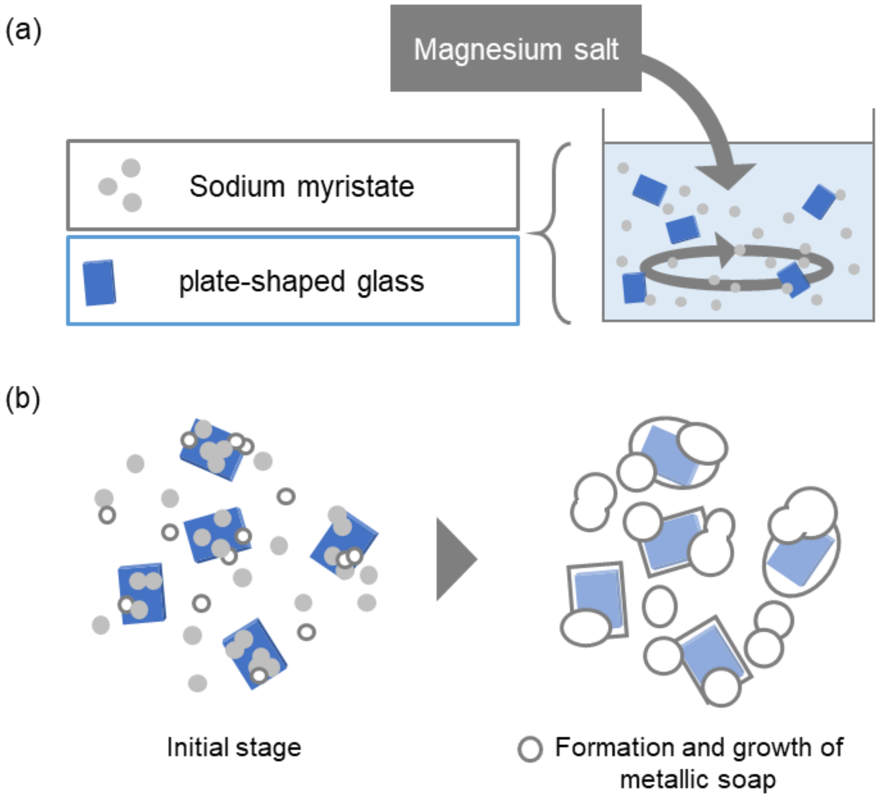

Figure 1. Schematic diagram of (a) manufacturing method of composite powder and (b) complexation process

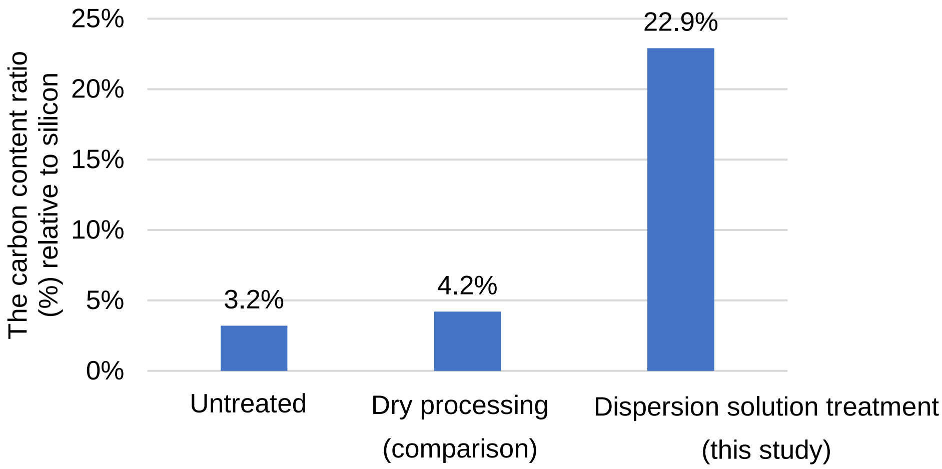

Figure 2. Comparison results of the amount of sodium myristate present on the plate-shaped glass surface

3.2 Observation of Composite Powders

The obtained composite powders were observed using an electron microscope to evaluate their composite state. The composite powders exhibited plate-shaped structures with uniform particle sizes (Figure 3). Furthermore, as shown in Figure 4, the average particle size of the composite powders was slightly smaller and more uniform compared to the metallic soap alone.

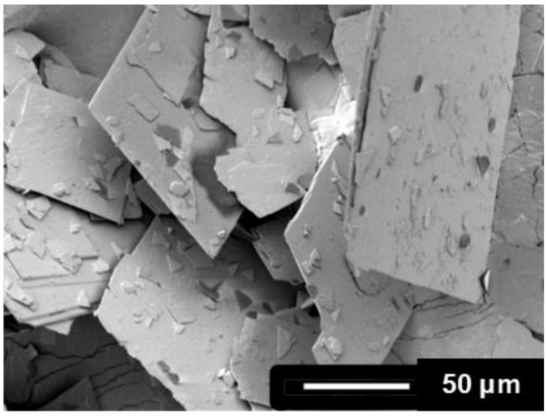

Figure 3. SEM image of Composite powder

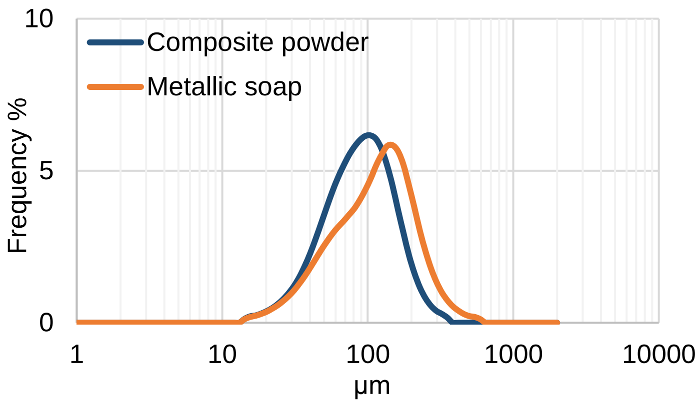

Figure 4. Average particle size

3.3 Evaluation of Aggregation

The tendency of the obtained powders to harden was evaluated by measuring their degree of aggregation. A higher aggregation degree indicates a greater tendency to aggregate, which is thought to affect hardness and susceptibility to caking. Analysis results showed that the metallic soap alone had an aggregation degree of 49.6%, whereas the composite powder exhibited a significantly reduced value of 15.4% (Figure 5). For comparison, mica, a commonly used base powder, had an aggregation degree of approximately 8%. Although the composite powder did not reach the same level as mica, a notable reduction in aggregation was observed.

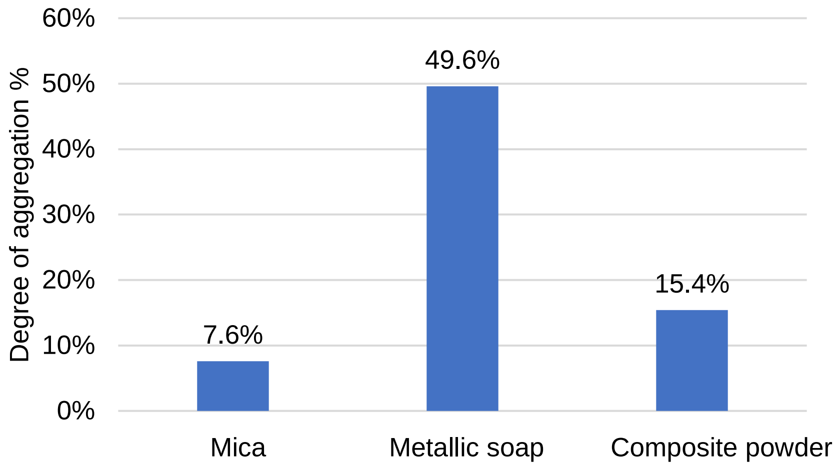

Figure 5. Degree of aggregation for each powder

3.4 Viscoelastic Evaluation of Oil Mixture Samples

To assess the removability and spreadability of the composite powder when mixed with oil, analyses of shear stress and loss tangent were conducted. Higher shear stress indicates greater resistance to deformation and better adherence after application to the skin. Additionally, a higher loss tangent value suggests greater viscosity and easier flowability. The evaluation results showed that the composite powder exhibited higher shear stress and loss tangent compared to the metallic soap alone (Figure 6). This indicates that the composite powder is less prone to deformation than the metallic soap alone while maintaining higher viscosity and better flowability.

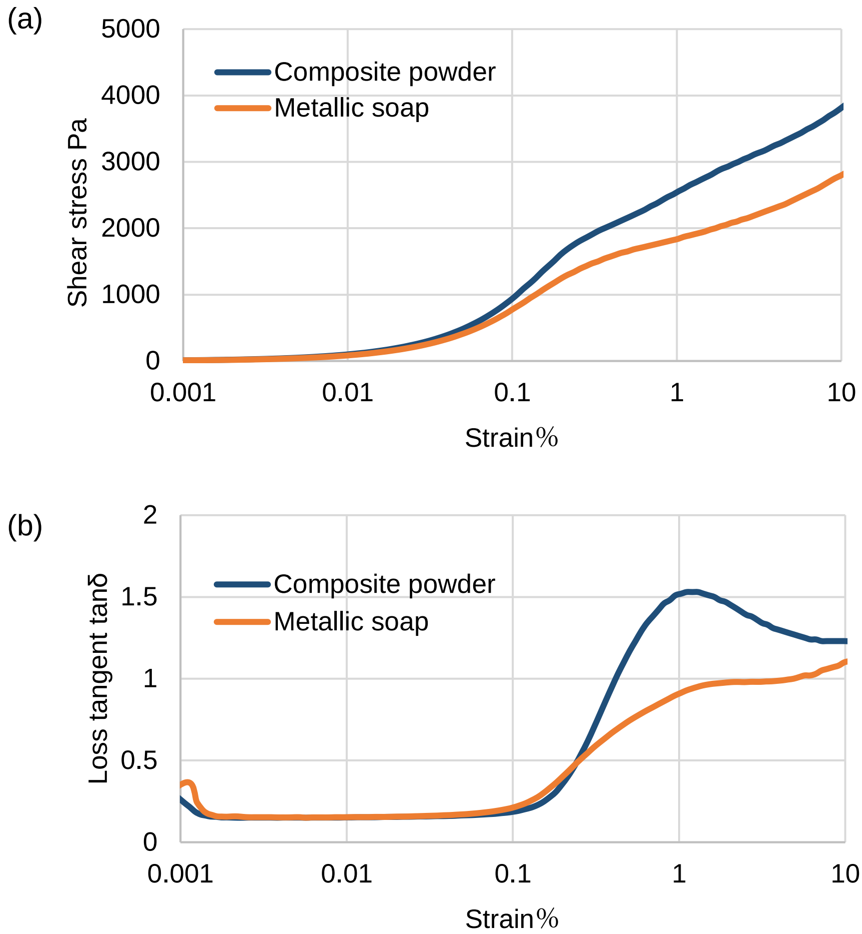

Figure 6. Sheer stress and loss tangent measurement results
(a) Sheer stress (b) loss tangent

3.5 Texture Evaluation

To confirm changes in texture due to the composite formation, the feel of the powders when applied to the skin was evaluated. For comparison, evaluations were also conducted for the metallic soap alone and mica, one of the most commonly used base powders. The results are shown in Figure 7.

In terms of spreadability, both the metallic soap and the composite powder scored higher than mica. Regarding smoothness, the metallic soap alone felt slightly heavy, with less of a smooth sensation. In contrast, the composite powder demonstrated greater lightness when spread on the skin, showing an improvement in smoothness compared to the metallic soap alone.

For glossiness, the metallic soap alone showed a more glaring shine, whereas the composite powder exhibited a more subdued gloss, with a luster that was more noticeable than mica. As for whiteness, the composite powder and mica were evaluated as being on par. An optimal balance of whiteness is important, as excessive whiteness can lead to an unnatural appearance due to white cast, while too little whiteness may result in reduced coverage.

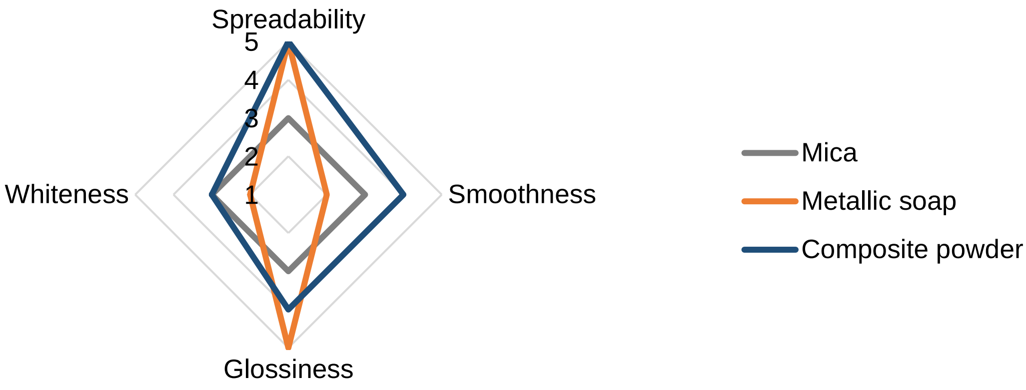

Figure 7. Results of tactile evaluation

4. Discussion

In Section 3.1, it was suggested that soap efficiently adsorbs onto the surface of plate-shaped glass within the soap solution. The carbon content on the glass surface processed in the soap solution increased compared to untreated plate-shaped glass, and significant enhancement of adsorption was observed compared to dry mixing. These results suggest that the use of a soap solution facilitates efficient soap adsorption on the surface of plate-shaped glass, forming a foundation suitable for the creation of composite powders.

In Section 3.2, the morphology of the obtained composite powders was observed using an electron microscope. As shown in Figure 3, the particles in the composite powder exhibited uniform shapes, and the average particle size was smaller than that of the metallic soap alone. These results indicate that the composite process forms a homogeneous composite state, with the plate-shaped glass effectively controlling the particle growth of the metallic soap. This may also contribute to the improvements in texture and reduced aggregation discussed later.

In Section 3.3, it was confirmed that the composite powder exhibited lower aggregation compared to the metallic soap alone. This is likely due to the uniform adsorption of soap onto the surface of the plate-shaped glass, which suppressed strong bonding between powder particles. Although the composite powder did not reach the level of mica, a commonly used base powder, a clear improvement in aggregation was observed. These findings provide important insights into the enhanced handling and processability of powders achieved through composite formation.

In Section 3.4, the composite powder mixed with oil exhibited seemingly contradictory properties, showing both high shear stress and high loss tangent compared to the metallic soap alone. This can be interpreted as requiring high stress to deform the material initially, but allowing for easy flow after it breaks down. In other words, the composite powder possesses excellent moldability while providing a smooth, spreadable, and lightweight texture. These results suggest that the composite powder addresses the challenges of poor removability and heavy, stiff textures that were issues with the metallic soap when mixed with oils.

In Section 3.5, texture evaluation demonstrated superior characteristics of the composite powder compared to the metallic soap alone and mica. In particular, the composite powder showed a marked improvement in "smoothness" when applied to the skin, reducing the heaviness associated with the metallic soap alone. Additionally, in terms of glossiness, the composite powder successfully suppressed the glaring shine of the metallic soap while achieving a natural luster superior to mica. For whiteness, the composite powder and mica were rated similarly, achieving a balanced appearance. Excessive whiteness can result in an unnatural finish due to white cast, while insufficient whiteness reduces coverage; the composite powder achieved an optimal balance. These findings suggest that composite formation not only improves texture properties but also enhances the quality of the finish in practical applications.

The series of results from this study indicates that the generation of composite powders through the treatment of plate-shaped glass in soap solutions provides benefits in terms of surface adsorption, reduced aggregation, and improved texture properties. In particular, the soap solution-based processing method was found to enhance the efficiency of composite formation while also improving the tactile and physical properties of the resulting powders. These insights provide effective guidelines for the development of future applied products with superior performance and user experience.

5. Conclusion

In this study, a novel composite powder was designed by combining magnesium myristate with plate-shaped glass powder. This developed powder successfully addressed issues associated with metallic soaps, such as aggregation and resulting texture deterioration, achieving sensory properties comparable to or better than mica, a widely used base powder. Furthermore, the developed powder utilizes plant-based, environmentally friendly materials, contributing to environmental conservation and resource efficiency through sustainable development, and potentially supporting the achievement of the SDGs.

However, aspects such as the control of the composite state and the behavior of the powder when incorporated into formulations remain partially unexplored. Clarifying these aspects in detail is expected to lead to further performance improvements and optimization of its properties. Additionally, advancing research to elucidate the detailed mechanisms based on the findings of this study and expanding the range of applications could bring significant progress in the use of metallic soaps as cosmetic raw materials.

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