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Structural Analysis Confirms the Stabilization Mechanism Maintaining Hair Treatment Quality in Cold Environments

Milbon Co., Ltd. (head office: Chuo-ku, Tokyo, President and CEO: Hidenori Sakashita), a manufacturer of salon-exclusive haircare products and cosmetics, in collaboration with Professor Kenichi Sakai of Tokyo University of Science, analyzed the structural features of hair treatments. The study confirmed the stabilization mechanism that suppresses property changes caused by freeze-thaw cycles in cold environments. The results of this research were presented at the following conference.

[Presentation]

Academic conference: 63rd Annual Meeting of the Japan Oil Chemists' Society

Title of presentation: Suppression and Mechanism of Freeze-Thaw-Induced Changes in the Appearance and Texture of Creams

Date of presentation: September 5, 2025

[Research Background]

Milbon products are used in hair salons around the world, from hot and humid regions to cold regions. We are committed to research and development that enhances product stability, ensuring consistently high quality across these diverse environments.

Gradual changes in appearance or texture of hair treatments may occur due to long-term storage or extreme temperature fluctuations. In cold regions, hair treatments may freeze and lose their smoothness after thawing and become rough in texture (Figure 1). Such changes not only cause concern among consumers but may also hinder expected hair treatment effects.

Milbon has empirically developed highly stable hair treatments that resist changes in appearance and texture caused by freeze-thaw cycles (Figure 2). However, the mechanisms behind this stability had not been fully elucidated.

Therefore, in this research we aimed to validate our previous empirical findings and to elucidate the stabilization mechanism that suppresses changes in appearance and texture after freeze-thaw cycles. This work seeks to achieve higher functionality and stability of hair treatments across diverse environments.

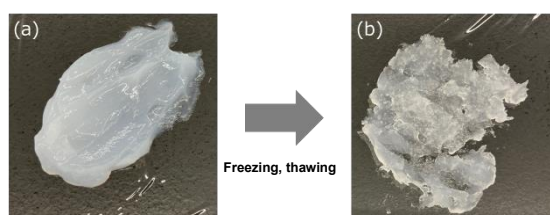


Figure 1. Changes in hair treatment appearances caused by freeze-thaw cycles
(a) Smooth appearance (b) Rough appearance after freeze-thaw cycles

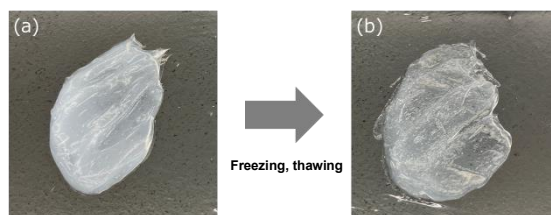


Figure 2. Empirically developed hair treatments with minimal changes caused by freeze-thaw cycles
(a) Smooth appearance (b) Smooth appearance even after freeze-thaw cycles

[Study Findings]

Confirmed the structural characteristics of hair treatments with high stability against freeze-thaw cycles.

Hair treatments contain various ingredients, and the stability of the formulation varies depending on the state in which these ingredients exist. Therefore, we examined the state of ingredients within each treatment to confirm why some hair treatments become rough after freezing and thawing while others remain smooth.

To investigate at the microscopic level, we conducted observations using a polarizing microscope^{*1} and small angle/wide angle X-ray scattering measurement^{*2}. As a result, it was revealed that both rough hair treatments and those that remain smooth contain an “ α -form hydrated crystal^{*3}”, a microstructure essential for hair treatments, and that this structure was preserved even after property changes induced by freeze-thaw cycles. As no major differences were observed in microstructure, fluorescence observation using confocal laser microscopy^{*4} was conducted to assess the state of the formulations from a macroscopic perspective. As a result, it was revealed that in the hair treatment that became rough after the freeze-thaw cycles, the α -form hydrated crystal is dispersed within the aqueous phase (the region containing water and water-soluble components) (Figure 3a). In contrast, in the hair treatment that later remained smooth, the aqueous phase is dispersed throughout the entire area of the α -form hydrated crystal (Figure 3b). Both treatments were observed before the freeze-thaw cycles.

These results suggest that it is important that the aqueous phase be dispersed throughout the α -form hydrated crystal to suppress changes in hair treatment properties after freeze-thaw cycles.

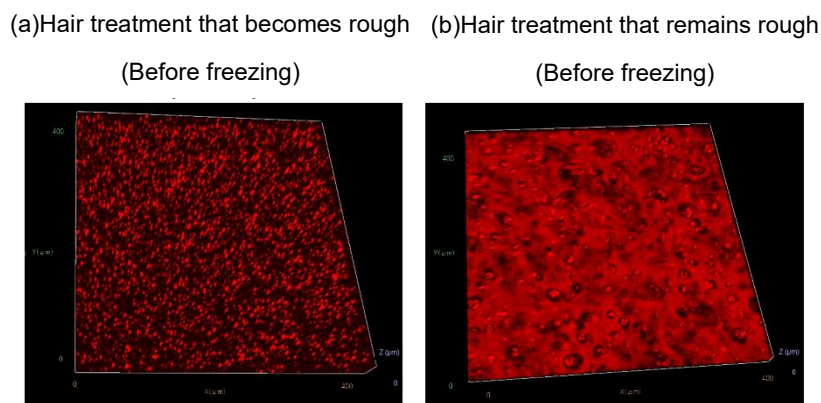


Figure 3. Observation results of the aqueous and oil phases in the treatments using confocal laser microscopy

- (a) α -form hydrated crystal dispersed in the aqueous phase (b) The aqueous phase is dispersed in the α -form hydrate crystals

Nile Red, an oil-soluble fluorescent dye was incorporated into each hair treatment to visualize the oil phase. Nile Red in the hair treatments is considered to localize in the lipophilic region of the α -form hydrated crystal. Therefore, red-colored areas indicate the presence of the α -form hydrated crystal, while black areas correspond to the aqueous phase.

[Future Vision]

Elucidating the stabilization mechanism of hair treatments during freeze-thaw cycles will enable formulation designs that accommodate diverse ingredients and formulations. We will continue advancing formulation technology research to develop products that can reliably deliver functionality across diverse environments.

《Supplementary Information》 Analysis Results of Microstructure by Small Angle/Wide Angle X-ray Scattering Measurement

Results from the small angle/wide angle X-ray scattering measurement showed that for both treatments, peaks were observed around $q = 15 \text{ nm}^{-1}$ in both the before freezing and after freezing and thawing states (Figure 4). The peaks indicate the presence of a hexagonal packing formed by the alkyl chains that constitute the α -form hydrated crystal. (Figure 5) No major differences were observed in this microstructure between the two treatments measured in this research and before and after the freeze-thaw process.



Figure 4 Results of small angle/wide angle X-ray scattering measurement

Figure 5 Image of hexagonal packing arrangements

《Terminology》

*1 Polarizing microscope

An optical microscope that utilizes the vibration direction (polarization) of light waves to visualize the internal structure of materials and the orientation of molecules. It is particularly effective for observing materials with ordered structures, such as liquid crystals and crystals.

*2 Small angle/wide angle X-ray scattering measurement

A technique that analyzes internal structures by irradiating materials with extremely short wavelengths of light in the form of X-rays. It can be used to obtain non-destructive structural information at the nano to atomic scale. Small-angle measurements allow examination of lamellar structures and molecular

arrangements, whereas wide-angle measurements enable investigation of crystallinity and fine microstructures.

***3 α -form hydrated crystal**

A lamellar structure formed by surfactants and long-chain alcohols, including water molecules, and exhibiting a regularly arranged crystalline structure. In this structure, the alkyl chains adopt a hexagonal packing, and the dense packing of molecules achieves high viscosity and high water retention.

***4 Confocal laser microscopy**

An optical microscope that achieves high-resolution imaging by using laser light and a pinhole to eliminate extraneous scattered light entering. By scanning the samples in layers, it enables three-dimensional observation of fine microstructures.

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