New Water Soluble Lauroyl Lysine Derivative and Its Unique Physical Properties in Applications in Cosmetics

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Short summary
New bis (Nε-lauroyl lysine) derivatives, in which two Nε-lauroyl lysine are linked by alkylene chain of various lengths were synthesized, and their properties in aqueous media were examined. We found that a compound with alkyene chain length C10 (GE-10-LL) forms hydrogel at a pH range of 5.3 - 7.0, and existence of a three-dimensional network of nanofibers in the hydrogel. FT-IR measurement revealed that GE-10-LL works as a low molecular gelator resulting in the formation of self-assembled nanofibers via hydrogen bonding. Besides, GE-10-LL turned out to be an effective foam booster for various foaming surfactants because the nanofibers stabilize bubble.

Introduction
Nε-lauroyl lysine (LL) is an organic powder, barely soluble in any kinds of cosmetic media. It is well known as a beneficial ingredient in various cosmetic applications; its excellent lubricity, wear-ability and non-greasy emolliency make it an ideal ingredient especially for makeups. These properties are also welcomed in skincare or hair care applications such as body lotions, daily use sunscreens and hair conditioners, and moreover, Nε-lauroyl lysine exhibits a fine pearlized effect and a foam boosting effect in surfactant-based cleansers. However, its use in these formulations has been very restricted because its inherent hydrophobicity makes it difficult to be dispersed in the aqueous media. We developed derivatives of Nε-lauroyl lysine to find a compound soluble in water, and studied its characteristics as a cosmetic ingredient.

Material and Methods
Synthesis of bis(Nε-lauroyl lysine) derivatives: Nε-lauroyl lysine was reacted with dicarboxylic acid with different alkyene chain length (C4 - C10) to give bis (Nε-lauroyl lysine) derivatives. (Scheme.1)

![Chemical structure](image)

Scheme.1 Synthetic procedure

Scanning Electron Micrograph (SEM): The hydrogel was rapidly frozen in liquid nitrogen, and then freeze-dried using a vacuum pump. This sample was sputtered with Pt-Pd before the observation.
Transmission Electron Micrograph (TEM): The hydrogel was dropped on a collodion and grid mesh for TEM. Next, the grid mesh was rapidly frozen in liquid nitrogen, and then freeze-dried using a vacuum pump. The dried sample was negatively stained by osmic acid, and then dried under reduced pressure before the observation.

FT-IR Spectrum: FT-IR spectrum of the hydrogel was measured by ATR method at r. t. The gel was heated to 60°C to give a solution and FT-IR spectrum of the solution was measured in the same way.

Foam Volume Measurement: A surfactant solution (pH6, active 10 wt %) 2g was diluted with water (35°C) to obtain 100g of a test solution and the test solution was stirred at around 3,400rpm for 10 sec. by a hand mixer for cooking use. Foam volume immediately after the stirring was measured.

Results
We found that a compound with alkylene chain length C10, named GE-10-LL, has good water solubility at pH7 or over and that GE-10-LL forms hydrogel in a pH range of 5.3 - 7.0. In order to obtain visual insights into the aggregation mode of GE-10-LL in hydrogel, we conducted SEM and TEM observation. We found out GE-10-LL forming nanofibers with a diameter of several tens of nanometers. Hydrogelation occurs because the nanospaces in the three-dimensional network structure entrap water. (Fig.1, Fig.2)

It is well known that many amino acid derivatives work as low molecular gelators and that hydrogen bonding and van der Waals interactions are main driving forces for the self-assembly of the gelators into nanofibers. IR spectroscopy is a powerful tool for studying these interactions. Fig. 3 shows FT-IR spectrum of GE-10-LL in DMSO. In spectrum of hydrogel, a C=O stretching vibration peak of intermolecular hydrogen bonding mode was observed around 1640cm⁻¹, on the other hand, in spectrum of the solution a free C=O stretching vibration peak was observed around 1660cm⁻¹.
These results suggest GE-10-LL form nanofibers by intermolecular hydrogen bonding as driving force.

**Foam Boosting Effect**

Addition of GE-10-LL hydrogel increases foam volume of various surfactants. Fig 4 shows results of foam volume measurement of various surfactant solutions\(^1\), to a 10wt% surfactant solution. GE-10-LL was added as a form of hydrogel, the final concentration in the solution was 0.05%. On the other hand, we could not observe foam boosting effect when GE-10-LL solution, not hydrogel, was added to the surfactant solutions, suggesting GE-10-LL nanofibers stabilize bubble.

Sensory Evaluation of Facial Wash Products

Fig 5 shows results of sensory evaluation of facial wash products, in which a product with 0.05% GE-10-LL was rated in comparison to a control product without GE-10-LL. GE-10-LL improves foaming speed, foam volume, and imparts distinctive smoothness to dry skin.

Conclusions
We developed derivatives of Nε-Lauroyl Lysine to find a compound soluble in water to some extent, and observed its unique property as a low molecular gelator for an aqueous solution, while maintaining sensorial and beneficial characteristics of original Nε-Lauroyl Lysine. We believe that GE-10-LL is a useful cosmetic ingredient.

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Reference