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# Effect of sodium chloride on precipitation and adsorption behavior of polymer-surfactant complex particles in aqueous solution



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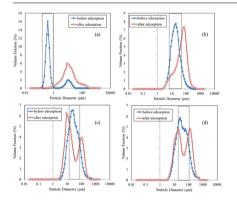
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#### GRAPHICAL ABSTRACT



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#### ABSTRACT

Polymer-surfactant complex particles formed by dilution of shampoo play an important role in the sensory properties of shampoo, but their microscopic mechanism has not been clarified yet. This study clarifies the change mechanism influencing hair surface smoothness induced by this complex. The degrees of precipitation and adsorption of the polymer-surfactant complex particles are varied by changing the sodium chloride (NaCl) concentration in the shampoo formulation; hence, the relationships between the frictional and sensory properties during rinsing, the particle formation numbers in water, and the particle adsorption rate on the hair surface are investigated. The frictional property measurement and microscopic observation of human hair treated with the various shampoo solutions confirm that two different particle types, floating and adsorptive, are formed in water during the shampoo dilution process, both of which contribute to improving the hair smoothness. Then, NaCl is added to the basic shampoo composition to control the formation of these two particle types. The particle size distribution and chemical identification show formation of submicron-sized particles mainly consisting of cationized polymer in water with less than 0.20-wt % NaCl, which do not adsorb on the hair surface. For 0.75–3.00-wt.% NaCl, polymer-surfactant complex particles are formed, which easily adsorb on hair. Finally, we propose an optimal NaCl concentration of 0.75 wt.% to obtain excellent frictional properties. The result will contribute to the formulation of cosmetic products based on microscopic mechanism.

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#### 1. Introduction

Knowledge of the precipitation and adsorption properties of polymer-surfactant complex particles in a shampoo solution during dilution is important in order to clarify the mechanism influencing the change in hair surface smoothness during washing. Polymer-surfactant complex particles are generated and become cloudy by dilution of the shampoo during rinsing after whipping, and their formation conditions and the hair morphology changes in response to the washing conditions have been widely studied [1–4]. The adsorption behavior of the complex on the hair surface has also been investigated [5,6]. However, the change mechanism influencing the sensory and frictional properties of the hair and induced by formation and adsorption of the complex has rarely been discussed.

Previously, we performed a quantitative evaluation of hair surface smoothness based on sensory evaluation and considering the reactive force during brushing from the mechanical perspective [7–9]; hence, we determined the microscopic mechanism of the shampoo product that improves hair smoothness during rinsing [10]. Our previous study revealed two different particle types in the presence of human hair, i.e., particles remaining in the water (floating particles) and those adsorbed on the hair (adsorptive particles) [7]. We also clarified that the adsorptive particles adhere to the hair surface mainly through electrostatic interaction, and that the content remaining on the hair surface even after rinsing mainly consists of cationic polymer [8]. This adsorbed polymer has a large effect on the hair surface smoothness; however, the role of the floating particles is unclear.

This study aims to clarify the roles of floating and adsorptive particles and the contribution of each towards hair smoothness, and then to clarify the best microscopic conditions to achieve optimal hair smoothness. The uniqueness of our study is that we focus on the microscopic behavior of two different types of particles around the hair surface, and establish a correlation between this behavior and the physical properties of the hair surface. To investigate this relationship, the volumes of the two kinds of particle were systematically changed by addition of sodium chloride (NaCl) to the shampoo, which minimized the effect of the electrostatic interaction between the molecules.

#### 2. Experimental methods

#### 2.1. Experiment overview and flow

There were three stages to the experiment. First, the reactive force during brushing was measured, to examine the contributions of the floating and adsorptive particles to the hair surface smoothness (described in section 2.2). Then, the formation numbers of the two types of particle were varied systematically by changing the NaCl concentration, with the formation numbers being determined based on the solution turbidity (Section 2.3). Finally, the contributions of these particles to the hair smoothness was investigated (Section 2.4). Hence, we determined the change mechanism according to the ratio of the two particle types.

### 2.2. Contributions of floating and adsorptive particles determined through reaction force measurement

First, the reactive force during brushing [6,11] was measured to evaluate the contributions of the floating and adsorptive particles to the hair smoothness. The model shampoo formulation detailed in Table 1 was used in this experiment, in which the formed number of polymer-surfactant complex particles was relatively high compared to typical shampoo formulations. Hair bundles (28 cm in length, ca. 10 g, mixed multiple Chinese female hair, Beaulax Co., Ltd., Japan) with no pre-treatment were used for physical measurement. The brushing reactive force was measured for three conditions: (a) a normal shampoo solution with both floating and adsorptive particles, (b) after removal of the

Table	1	
Model	shampoo	formulation.

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Material	Ratio (wt.%)
Lauroyl sarcosine triethanolamine	10.0
(Kawaken Fine Chemicals Co.,Ltd., Japan)	
Cocamidopropyl betaine	5.0
(NOF Corporation, Japan)	
Cationized cellulose	0.5
(LION Specialty Chmicals Co.,Ltd., Japan)	
Purified water	47.5

adsorptive particles, and (c) after removal of the floating particles.

Before experiment, a hair bundle was washed with tap water at 40 °C and passed through a metal brush sufficiently over 10 times (1.0mm projection pitch, 1.0-mm projection diameter). For the experiment featuring condition (a), involving a normal shampoo solution with both floating and adsorptive particles, a test bundle was simply immersed in 3.00 wt.% shampoo solution (1.0 L) at 40 °C for 1 min. For the condition (b) experiment, involving removal of the adsorptive particles, three other hair bundles were immersed for 5 min before a test bundle in the shampoo solution. The three hair bundles were used only for adsorptive particle removal, and not for the brush test, and then a new test bundle was immersed in the shampoo solution for 1 min. For the condition (c) experiment, with removal of the floating particles, a hair bundle was immersed in a shampoo solution for 1 min. Then, the floating particle removal was performed by shaking the hair bundle gently and repeatedly drawing and immersing the hair bundle in the shampoo (approximately 5 times). It was confirmed that repeated drawing and immersion does not affect the reaction force by performing a blank test with tap water, with and without shaking. The selective removal of floating or adsorptive particles by the above methods was checked through microscopic observation of the shampoo solution and hair surface after each treatment. In detail, to verify whether the floating and adsorptive particles were effectively removed by the above methods, the hair in the solution after the three treatments described above was observed using a polarizing microscope (BX51, Olympus Corporation, Japan).

After the three treatments detailed above, the reactive force during brushing was measured for each of the conditions using the method reported in our previous study [7]. That is, the required hair bundle was extracted from the shampoo solution and pre-brushed once. Immediately after pre-brushing, the root of the hair bundle was fixed to the load cell of a universal tester by a tensile jig, and seven projections of the metal comb were inserted at a height of 150 mm from the bundle tip to measure the reactive force. In addition, the hair was rinsed with tap water at approximately 40 °C for 30 s after soaking in the solution; then, the same experiment was conducted. The displacement length was 150 mm from the middle to the tip of the hair bundle, and the displacement velocity was 50 mm/min. The experimental system for measurement of the reactive force during brushing is shown in Ref. [6]. The reactive force was measured with a compact universal tester (EZ-Test, Shimadzu Corporation, Japan). The measurement was conducted three times with a different hair bundle each time, for each condition.

### 2.3. Measurement of shampoo solution turbidity before and after soaking of human hair in different NaCl concentrations

The additive amount of NaCl in the model formulation reported in Table 1 was changed to 0, 0.75, and 3.00-wt.%, and the corresponding particle numbers generated and adsorbed on the hair were investigated. Both the number of particles generated and adsorbed were evaluated based on the turbidity of the shampoo aqueous solution by using the light transmittance (Colorimetric Color Difference Meter Z 6000: Nippon Denshoku Industries Co., Ltd., Japan), because the solution was clouded by generation of the polymer-surfactant complex particles. A

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