Construction of pH-Responsive Pseudo-Gemini Surfactants: Exploring the Microscopic Mechanisms for Enhancing the Performance of Hair Conditioner Systems

Lina Wang[1],*, Xiaoxue Cai[1], Xiaoqin Tang[1], Guodong Zhang[1], Huilin Li[1]

[1] Shanghai Chando Group Co., Ltd,Shanghai,China.

* Wang Lina, Shanghai, wanglina3@chandogroup.com

Abstract:

This study systematically explores the molecular design advantages of succinic acid (SA) in hair conditioner systems and its pH-responsive characteristics, aiming to overcome the bottlenecks of traditional conditioner formulations, such as high lipid dependence and loose structure, by proposing innovative strategies. Experimental results show that succinic acid (SA), through its dicarboxylic acid groups interacting with stearamidopropyl dimethylamine (SPA), forms a pseudo-gemini surfactant structure, thereby constructing a long-range ordered dense layered network. Under the condition of only 5% fatty alcohol, the static thickening efficiency of this system (η_0=18,810 mPa·s) is significantly improved by 4.5 times compared to the lactic acid (LA) system. Additionally, the introduction of succinic acid (SA) synergistically enhances the combability of the conditioner (wet combing improvement rate of 67.51%) and its antistatic properties (relative static voltage of 4.23%). Further studies reveal that when the pH of the system increases from 4.5 to 6.5, the charge neutralization effect is enhanced, optimizing the density of the layered network, thus significantly improving the static viscosity (η_0=35,940 mPa·s) and antistatic performance (relative static voltage of 1.48%). However, the shear dilution slope of the system remains consistent, ensuring that the dy-namic spreadability is not affected. This study reveals the equilibrium mechanism of "static densification-dynamic response" in the conditioner system, and proposes a combination of molecular design and environmental parameter regulation to simultaneously achieve formulation streamlining and performance optimization. This achievement provides a solid theoretical basis and innovative technical paradigm for the development of highly effective hair conditioner products.

1. Introduction

In the field of hair care, the functionality of conditioners is mainly based on the synergistic effect of cationic surfactants and fatty alcohols [1]. These two are effective in improving the combability of the hair by lubricating the hair scales and neutralizing static electricity [2-3]. Conventional formulations are typically designed with a pH of approximately 4.5, as this pH

environment encourages scales to close and reduces water loss from the hair, thereby enhancing shine [4-5].

However, when using tertiary amine cationic surfactants (such as SPA), this system faces severe challenges. Under pH 4.5 conditions, due to the pKa of SPA being approximately 9.5, SPA is nearly completely protonated, and the strong electrostatic repulsion between the cationic head groups ($-NH^+(CH_3)_2$) causes the surfactant-fatty alcohol composite structure to become loose. Traditional monocarboxylic acids, such as lactic acid, due to their molecular structure characteristics, cannot effectively counteract this repulsion through intermolecular forces. In order to maintain the thickening effect of the system, high concentrations of fatty alcohols in the range of 8% - 10% can only be added. However, this not only makes the formulation cumbersome, but also gives the user a sticky skin feeling, which seriously affects the experience of using the product.

To overcome these bottlenecks, this study innovatively proposes a pH-responsive pseudo-gemini surfactant strategy based on dynamic assembly of dicarboxylic acids. In recent years, it has been shown that SA and long-chain tertiary amine surfactants can construct a biantennary structure with the help of two-site electrostatic interactions, and exhibit excellent viscosity and pH responsiveness under weak acidic conditions [6]. Based on this, this study introduces SA into the SPA fatty alcohol system. Based on this, this study introduces SA into the SPA fatty alcohol system. Under conditions close to the physiological pH of the scalp (5.5-6.5), the dicarboxylic acid groups of SA completely dissociate into bivalent anions ($-COO^-$), bridging two protonated SPA molecules through two-site electrostatic interactions, forming a stable pseudo-gemini assembly [7-8].

This dynamic assembly achieves performance enhancement through three synergistic effects: firstly, the pseudo-gemini structure effectively reduces the electrostatic repulsion between SPA head groups, strongly promoting the densification of the surfactant-fatty alcohol composite structure; secondly, the formation of a hydrogen bond network significantly enhances interfacial stability, thereby reducing the system's dependence on high concentrations of fatty alcohol; thirdly, the pH-responsive characteristics endow the system with good adaptability, allowing it to cleverly balance high viscosity and strong adsorption under mild conditions [9-11]. This study is the first to apply the dynamic pseudo-gemini mechanism to hair conditioner systems, laying a solid theoretical foundation for the development of new conditioners that are both gentle and effective, and is expected to promote technological innovation and product upgrades in the hair care field.

2. Materials and Methods

2.1 Materials and Methods

A total of 5 groups of hair conditioner samples were prepared in this experiment. Three groups of samples with a pH of 4.5 were adjusted with SA, hydrochloric acid (HCl), and LA respectively. The other two groups were samples adjusted to pH 5.5 and 6.5 with SA. The basic components of all samples were SPA (3.0%), stearyl alcohol (3.5%), cetyl alcohol (1.5%), and the rest was made up to 100% with deionized water (Table 1).

The specific preparation process is as follows:

First, accurately weigh a certain amount of SPA, stearyl alcohol, and cetyl alcohol and place them in a clean beaker. Then, add deionized water to the beaker to make the total mass of the solution reach 100%. After that, put the beaker into an 80°C constant temperature water bath device, and use an IKA® EUROSTAR 40 mixer to stir at a speed of 300 rpm until all components are evenly dispersed.

Subsequently, at 80°C, SA, HCl, and LA (all of analytical grade) were added dropwise directly into the solution. The neutralization of the acid with SPA promoted the gradual dissolution of SPA. During this process, the pH value of the solution was monitored in real-time using a pH meter and adjusted to the target pH value, with an allowable deviation range of ±0.1. After the system cooled to 25°C, it was left to stand for 24 hours for aging treatment. Regarding the pH test conditions: To simulate the actual use scenario, the hair conditioner sample needs to be diluted to a 10% aqueous solution before measuring the pH value.

Table 1. Information of Test Hair Conditioner Samples

Sample Number	pH	Regulator	SPA Content	Stearyl Alcohol Content	Cetyl Alcohol Content	Deionized Water Content
Sample A	4.5	SA	3.0%	3.5%	1.5%	Make up to 100%
Sample B	4.5	HCl	3.0%	3.5%	1.5%	Make up to 100%
Sample C	4.5	LA	3.0%	3.5%	1.5%	Make up to 100%
Sample D	5.5	SA	3.0%	3.5%	1.5%	Make up to 100%
Sample E	6.5	SA	3.0%	3.5%	1.5%	Make up to 100%

2.1 Test Methods

The rheological properties were tested by dynamic oscillation strain sweep experiments using a rotational rheometer. The fixed frequency was 1 Hz, the strain amplitude was scanned from 0.001% to 100% in logarithmic mode, and the test temperature was 20°C. Through this experiment, the change curves of the storage modulus (G') and loss modulus (G'') with strain were recorded to evaluate the structural stability of the system. Meanwhile, the curve of the dynamic viscosity versus the strain was also recorded.

The microstructure was characterized by a Gemini 300 cryo-field emission scanning electron microscope. The sample was quickly immersed in liquid nitrogen (-196°C) for freezing and fixing, then transferred to the preparation chamber (-140°C, vacuum degree ≤ $5×10^{-6}$ mbar) for brittle fracture. After exposing the internal structure, it was sublimated at -75°C for 10 minutes to remove surface condensed water, and a platinum film was sputtered with a current of 7 mA for 10 seconds. The treated sample was transferred to the observation chamber (-140°C), and high-resolution images were collected under an acceleration voltage of 5 kV and a working distance of 10 mm using a secondary electron detector.

The dynamic combing performance was evaluated using a SK - 7A dynamic combing instrument. The hair bundle samples were washed 3 times with an SLES solution (w = 5%) to remove surface oil and impurities. The hair bundle was placed on the dynamic combing instrument to test the combability of the blank dry hair and the static voltage on the hair bundle surface, and then the combing force of the blank wet hair was tested. The hair bundles in the experimental group were evenly applied with 2 mL of hair conditioner, left to stand for 1 minute, and then the processes of wet combing, combing while rinsing, and final combing were repeated. The combability of the dry hair and the static voltage on the hair bundle surface were tested. Each group was tested with 3 parallel samples. The calculation formula for the combing force improvement rate is: Combing improvement rate = (Blank combing force - Combing force after

product use) / Blank combing force × 100%; Relative electrostatic voltage = Static voltage after product use / Blank static voltage × 100%.

3. Results and Discussion

3.1 Effects of Different Acids on the Performance of Hair Conditioner Systems

3.1.1 Microstructure Comparison

The cryo-electron microscopy images (Figure 1A to 1D) systematically presented the differences in the microstructures of the SA and LA systems at pH 4.5. When observing the LA system at a low magnification (Figure 1A), it showed a wide lamellar spacing (3 - 4μm), with a relatively clear interlayer interface, but there were a large number of vesicle structures (mainly with a diameter < 1μm). When switching to a high magnification (Figure 1B), it could be further seen that the wide lamellar spacing area still maintained a continuous lamellar arrangement. However, due to the interference of electrostatic repulsion on SPA and fatty alcohol molecules, they failed to closely fill the interlayer voids, resulting in a lack of rigidity in the lamellar network. In contrast, in the SA system. When observed at the same low magnification (Figure 1C), it showed a uniform and dense lamellar arrangement (lamellar spacing < 1μm), with only a few vesicles. The high - magnification image (Figure 1D) further confirmed that the interlayer interface of this system was very sharp, and the fatty alcohol molecules and the hydrophobic chains of the surfactant formed a tight stacking structure. The long-range order of its lamellar phase was significantly better than that of the LA system.
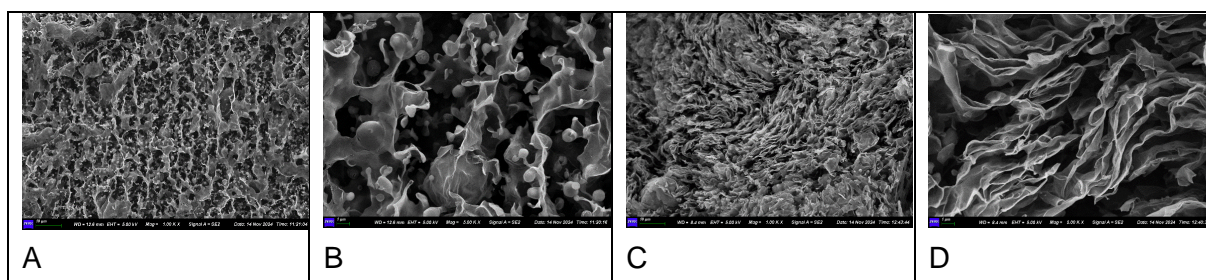

Figure 1. Cryo - scanning electron microscopy images of hair conditioner systems with different acids at pH = 4.5. (A) Low - magnification image of the lactic acid system (1000×). (B) High - magnification image of the lactic acid system (5000×). (C) Low - magnification image of the succinic acid system (1000×). (D) High - magnification image of the succinic acid system (5000×).

In-depth analysis shows that due to the insufficient charge neutralization efficiency of the monocarboxylic acid group of LA, there is still residual electrostatic repulsion between SPA head groups. During the shear emulsification process, the combined action of interfacial tension and shear rate forces some regions to form spherical lamellar structures [12-14]. Although such closed - curvature structures are composed of SPA and fatty alcohols, their geometric characteristics cause an ineffective diversion of active ingredients: the components involved in the spherical lamellar structure cannot be simultaneously embedded in the planar lamellar network, which directly reduces the proportion of the planar lamellar phase in the overall structure. In addition, although the cationic head groups of SPA are exposed on the outside of the spherical structure due to the curvature effect, their arrangement density is lower than that in the planar lamellar network, resulting in a reduction in the effective charge neutralization area.

On the contrary, the dicarboxylic acid groups of SA can neutralize the charges of SPA head groups through two - site binding, effectively eliminating the interference of electrostatic repulsion on molecular arrangement. As can be seen from Figure 1D, its planar lamellar structure forms a long - range ordered molecular cooperative network through the periodic and close arrangement of SPA and fatty alcohols. The uniformity of this structure benefits from the highly optimized charge distribution by the dicarboxylic acid groups, effectively suppressing the tendency to form curvature and laying a microscopic foundation for subsequent performance advantages.

3.1.2 Rheological Behavior Analysis

By comparing the rheological properties of the SA, LA, and HCl systems through dynamic strain scanning at pH 4.5, significant differences in network stability were found (Figure 2). Taking the strain $\gamma = 0.142$ as an example, the storage modulus $G' = 340.9$ Pa and loss modulus $G'' = 285.3$ Pa of the SA system were 5.6 times the G' of the LA system (61.08 Pa) and 3.5 times the G'' of the LA system (81.09 Pa), respectively, and 5.4 times the G' of the HCl system (63.38 Pa) and 3.5 times the G'' of the HCl system (82.45 Pa). When the strain increased to $\gamma = 0.252$, the G' of the SA system still remained at 171.3 Pa, and G'' (206.5 Pa) did not significantly exceed G'. The critical strain value $\gamma\_c \approx 0.25$, indicating that its network could quickly recover after shear. However, in the LA and HCl systems, $G'' > G'$ had already occurred at $\gamma = 0.142$, and $\gamma\_c < 0.14$, and the structural collapse occurred significantly earlier. The analysis of dynamic viscosity (η') further verified the above conclusions (Figure 3). The initial viscosity of the SA system in the low - strain region ($\gamma < 0.01$) was as high as 18,810 mPa·s, which was 4.5 times that of the LA system (4,183 mPa·s) and 4.9 times that of the HCl system (3,858 mPa·s). It is worth noting that although the initial viscosity of SA was significantly higher, as the strain increased, the η' of the three groups of systems all showed a shear - thinning trend, and the change slopes were similar, indicating that their shear - response behaviors were similar, indicating that it had a spreadability comparable to that of traditional systems.
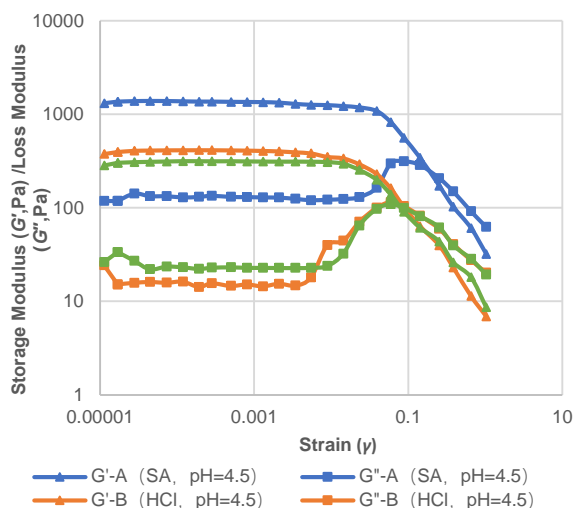

Figure 2. Dynamic strain scanning curves of hair conditioner systems with different acids at pH = 4.
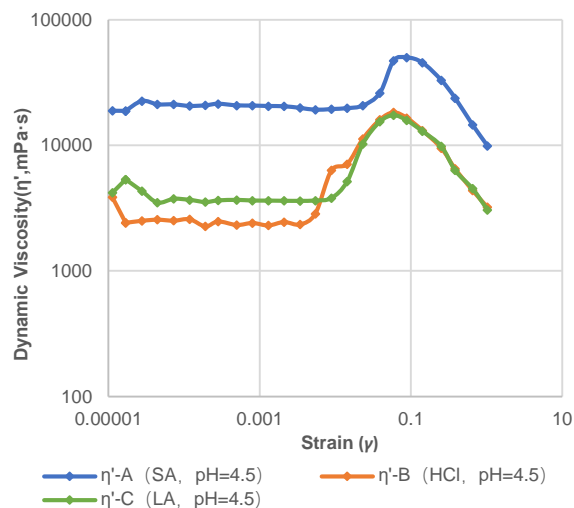
Figure 3. Change curves of dynamic viscosity with strain of hair conditioner systems with different acids at pH = 4.5

SA forms a pseudo-gemini surfactant through the two-site binding of its dicarboxylic acid groups with SPA, which can better neutralize the charges of head groups, eliminate electrostatic repulsion and the tendency to form curvature, and force all SPA and fatty alcohols to be

oriented and arranged into a tight planar lamellar network. This structure has dual rheological advantages: high static viscosity ensures storage stability, and the shear - thinning property maintains the use viscosity (1,000-2,000 mPa·s), achieving a "high static stability - low use viscosity" synergistic effect.

In contrast, due to insufficient charge neutralization, the traditional mono - acid system forms vesicle structures, reducing the proportion of the planar lamellar phase available for thickening. Its thickening efficiency is only 22% of that of the SA system. It needs to rely on high - concentration fatty alcohols to fill the interlayer voids, resulting in formula redundancy. The rheological behavior of the HCl system is similar to that of the LA system, which further indicates that the nature of the acid (organic/inorganic) is not the main cause of performance differences. It confirms that the performance differences stem from the dicarboxylic acid molecular design of SA rather than the acid property. The structural optimization of SA achieves both formula simplification and efficacy improvement, providing an innovative solution for the development of lightweight and long - acting hair care products.

3.1.2 Combing Property and Antistatic Property

Experimental data shows that the SA system significantly outperforms the LA and HCl control systems in the key performance indicators of hair conditioners. The tests of the improvement rates of wet - combing and dry - combing (Figure 4) reveal that the wet - combing improvement rate of the SA system reaches 67.51%, which is 34% and 45% higher than those of the LA (50.24%) and HCl (46.33%) systems respectively; the dry - combing improvement rate is 65.64%, 53% and 65% higher than those of the LA (43.00%) and HCl (39.78%) systems respectively. The antistatic property test further validates its superiority (Figure 5): the relative electrostatic voltage of the SA system is only 4.23%, 70% and 82% lower than those of the LA (14.37%) and HCl (24.21%) systems respectively. Although the LA and HCl systems still have a certain ability to improve antistatic property and combability, their performance efficiencies are significantly lower than that of the SA system.
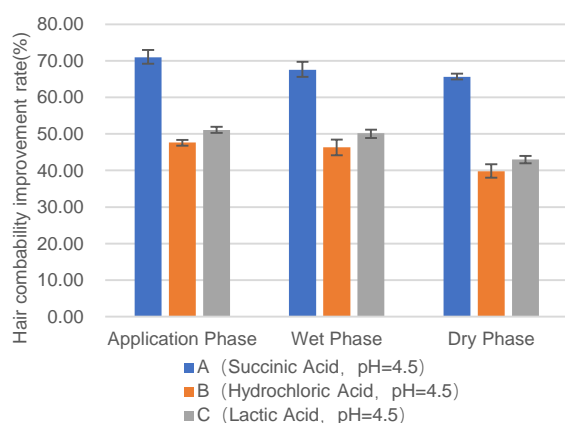

Figure 4. Comparison of the Combing Improvement Rates of Hair Conditioner Systems with Different Acids at pH = 4.5
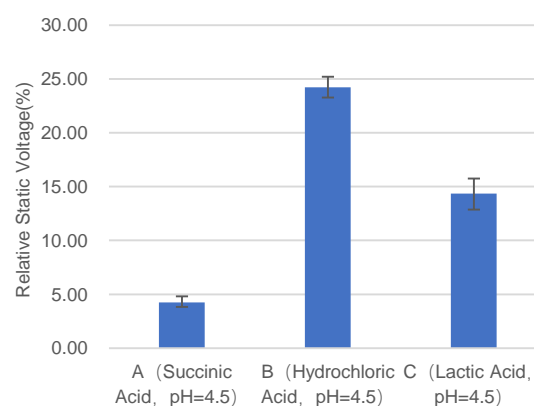
Figure 5. Test Results of the Antistatic Properties of Hair Conditioner Systems with Different Acids at pH = 4.5

The long - range ordered and dense lamellar network of the SA system maximizes the exposed area to efficiently bind the negative charges on the hair strands. It reduces the differences in local frictional contact by uniformly coating the surface of the hair strands. In contrast, the vesicular structure of the LA system has defects, resulting in a reduction in the effective exposed area, insufficient lubrication efficiency, and a relatively increased combing resistance. Moreover, due to the uneven charge distribution in the planar lamellar regions with wide lamel-

lar spacing, the antistatic efficiency is further weakened. In addition, the shear - thinning property of the SA system ensures that the viscosity of the hair conditioner drops rapidly when applied, further promoting the sliding between hair strands and thus enhancing the combing experience during actual use. This "uniform lubrication - high-efficiency antistatic" synergistic mechanism verifies the decisive role of microstructural design in the utilization rate of active ingredients and provides a direct basis for regulating the efficacy of hair conditioners through microstructural design.

3.2 pH Responsiveness of the Succinic Acid Hair Conditioner System

3.2.1 Rheological Analysis

The dynamic strain scan data (Figure 6) shows that the storage modulus (G') of the succinic acid (SA) hair conditioner system is overall similar under pH conditions of 4.5, 5.5, and 6.5. In the low-strain region ($\gamma < 0.01$), the G' values are 1306 Pa (pH 4.5), 1342 Pa (pH 5.5), and 1291 Pa (pH 6.5), indicating that the lamellar networks under the three pH conditions all have good static structural support. However, the response characteristics of the loss modulus (G'') and dynamic viscosity (η') show significant pH dependence (Figure 7). When the strain increases to $\gamma = 0.0388$, the G'' value (267.4 Pa) of the pH 6.5 system rapidly exceeds the G' value (913.2 Pa), and the critical strain value $\gamma\_c \approx 0.03$, which is significantly earlier than that of pH 4.5 ($\gamma\_c \approx 0.25$) and pH 5.5 ($\gamma\_c \approx 0.05$), indicating that its lamellar network is more likely to undergo irreversible deformation under shear at high pH.

The dynamic viscosity (η') curve further reveals the pH response mechanism (Figure 7). The initial viscosity (35,940 mPa·s) of the pH 6.5 system in the low-strain region ($\gamma<0.01$) is 44% and 91% higher than that of pH 5.5 (24,990 mPa·s) and pH 4.5 (18,810 mPa·s), respectively, indicating that a high pH environment is more conducive to the static thickening of the lamellar network. However, as the strain increases ($\gamma>0.1$), the η' values of the three systems gradually approach, and finally reach a similar range at $\gamma = 1.026$ (pH 4.5: 9,900 mPa·s; pH 5.5: 10,650 mPa·s; pH 6.5: 10,580 mPa·s), with highly consistent shear thinning slopes. This characteristic of "high static viscosity - synchronous shear response" indicates that although the pH 6.5 system has a better thickening efficiency during static storage, its spreading performance during actual use is still comparable to that of conventional pH (4.5 - 5.5) systems.
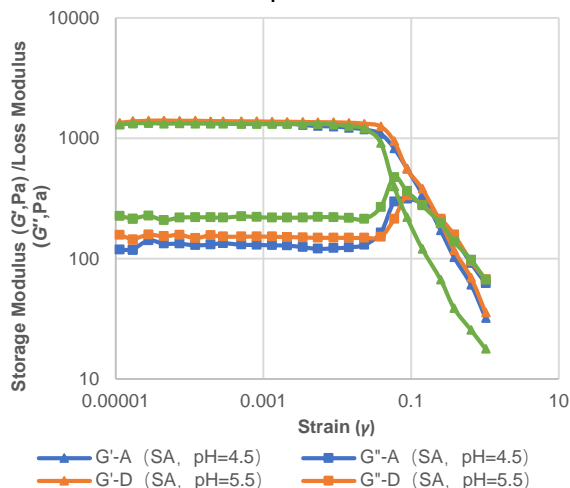

Figure 6. Dynamic Strain Scan Curves of Succinic Acid Hair Conditioner Systems with Different pH Values
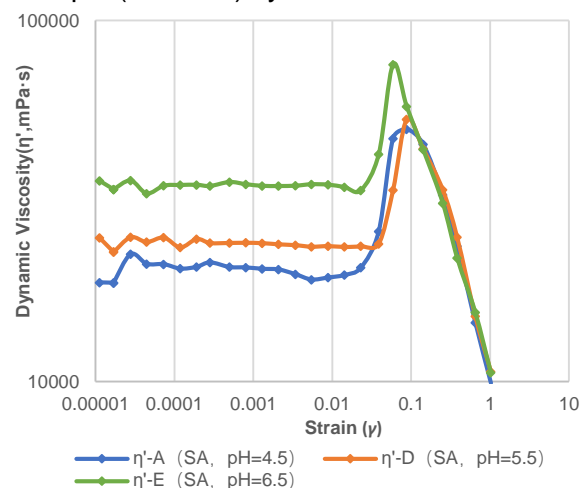
Figure 7. Curves of Dynamic Viscosity Changes with Strain in Succinic Acid Hair Conditioner Systems at Different pH Values

Increasing the pH enhances the dissociation degree of the carboxylic acid groups of SA, promoting more uniform charge distribution of the pseudo-gemini surfactants formed by two-site binding, thereby enhancing the static compactness of the lamellar network (the initial values of G' and η' increase). However, excessive negative charges simultaneously weaken the van der Waals forces between the hydrophobic layers, resulting in a decrease in the network's shear resistance (γ_c advances). This pH responsiveness endows the SA system with dual advantages: during the storage stage (static) of the hair conditioner, the pH 6.5 condition improves the stability of the paste through a high initial viscosity; during the use stage (dynamic), the shear thinning property ensures that different pH systems are all easy to spread. The experimental results confirm that the balance of "static thickening - dynamic lubrication" can be achieved by adjusting the pH of the system, providing theoretical support for the development of adaptable hair conditioner formulations.

3.2.2 Combability and Antistatic Properties

The experimental data show that the combability and antistatic properties of the SA hair conditioner system increase significantly as the pH increases from 4.5 to 6.5 (Figures 8 - 9). In the wet combing test, the improvement rate of the pH 6.5 system reaches 76.04%, which is 10.4% and 12.6% higher than that of pH 5.5 (68.83%) and pH 4.5 (67.51%), respectively; the dry combing improvement rate increases from 65.64% at pH 4.5 to 74.87% at pH 6.5, with an increase of 14.1%. The antistatic performance shows the same trend: the relative electrostatic voltage (1.42%) of the pH 6.5 system is 54% and 66% lower than that of pH 5.5 (3.08%) and pH 4.5 (4.23%), respectively. This result is consistent with the static thickening advantage (η' = 35,940 mPa·s) of the pH 6.5 system in the rheological data in 3.2.1, indicating that high pH conditions further improve the terminal performance by optimizing the microstructure.
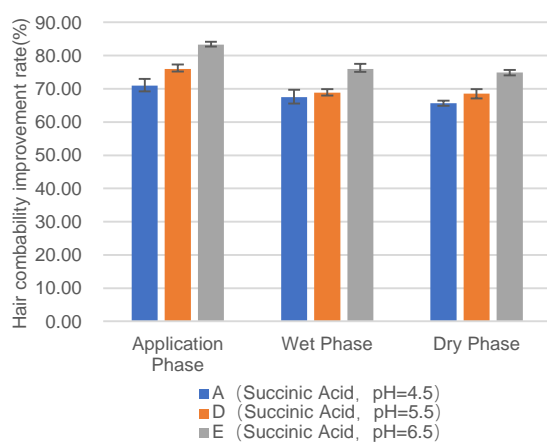

Figure 8. Comparison of the Combing Performance of Succinic Acid Hair Conditioner Systems with Different pH Values
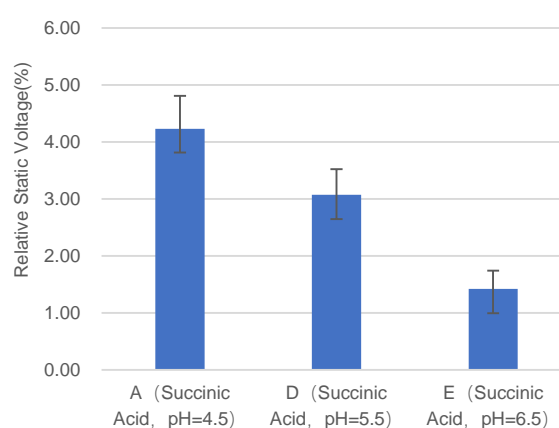

Figure 7. Comparison of the Antistatic Performance of Succinic Acid Hair Conditioner Systems with Different pH Values

High pH conditions enhance the static compactness of the lamellar network through enhanced electrostatic neutralization. This structural feature reduces the frictional resistance by uniformly coating the hair surface on the one hand, and further improves the electrostatic binding efficiency between the SPA cationic head groups and the negative charges on the hair by maximizing the exposure area of the SPA cationic head groups on the other hand. The pH responsiveness of the SA system proves that the intermolecular forces (electrostatic neutralization and hydrophobic synergy) can be precisely regulated by adjusting the environmental pH, thereby optimizing the performance of the hair conditioner. This "static compact - dynamic

response" synergy mechanism provides a theoretical basis for the development of customized products that meet different storage conditions and usage requirements.

4. Conclusions

This study systematically analyzed the microstructure, rheological properties, and performance in use of the SA hair conditioner system, revealing the core value of the molecular design of dicarboxylic acid groups for the utilization efficiency of active ingredients and the performance of the formulation. Compared with traditional monocarboxylic acid systems, SA forms pseudo-gemini surfactants through two-site binding, constructing a long-range ordered and dense lamellar network. This structure maximizes the exposure area of SPA cationic head groups and, through synergistic embedding with fatty alcohol molecules, achieves a synergistic breakthrough in low-fat and high-efficiency thickening, uniform lubrication, and high-efficiency antistatic properties, providing a molecular design example for replacing traditional formulae with extensive ingredient stacking.

Further research shows that the SA system has unique pH-responsive characteristics. When the pH increases from 4.5 to 6.5, the charge distribution is optimized by enhancing the dissociation degree of carboxylic acid groups, significantly improving the static thickening efficiency and antistatic performance. At the same time, the consistency of the shear thinning slope ensures that the hair conditioner maintains good spreadability during actual use. This "static compact - dynamic response" balance mechanism indicates that by regulating environmental parameters, it is possible to take into account both the storage stability of the product and the user experience, providing a new strategy for the development of adaptable hair conditioner products.

This study offers the following inspirations for the development of hair conditioners from the perspectives of molecular force regulation and structure design. Firstly, in terms of ingredient synergy, pseudo-gemini surfactants break through the bottlenecks of traditional mono-acid systems through charge-hydrophobic synergy. Secondly, in terms of innovative formulation strategies, pH responsiveness paves a new way for the development of "multi-functional in one" customized formulations. For instance, by adjusting the pH, functions like "antistatic - rapid repair" can be switched, thus optimizing product performance and expanding application scenarios.

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