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Investigation of the Influencing Factors and Formation Mechanism of the Soaping Effect in Natural Source Cosmetics

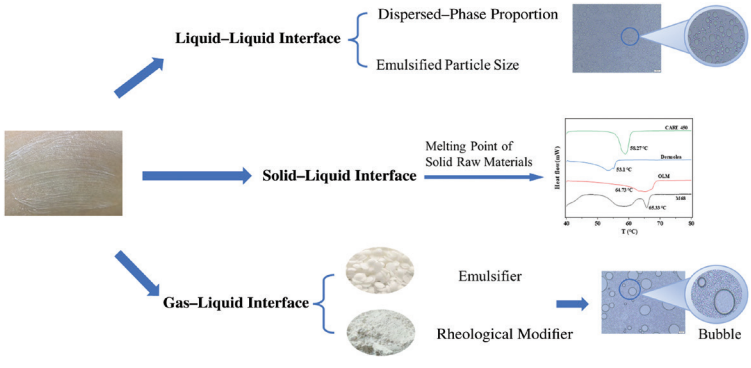
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Synopsis

Based on the previously established and standardized method for evaluating the soaping effect, this study systematically examined the mechanisms underlying this phenomenon, including the influence of the composition of naturally sourced cosmetic products. The experimental results indicated that emulsifiers strongly influence the soaping effect; in particular, formulations containing cetearyl alcohol typically lead to a more pronounced soaping effect. The incorporation of fatty alcohols intensify the phenomenon, with longer carbon chains resulting in a stronger soaping effect. Oils affected the soaping effect through their impact on emulsification quality, such that inferior emulsification generally led to a weaker effect. Rheological modifiers influenced the soaping effect by modulating system viscosity, absorption kinetics, and bubble stability. Further analysis revealed that from a visual perspective, the intensification of the soaping effect was frequently accompanied by the formation of new phase states. An examination of phase transitions within emulsions before and during application demonstrated that the soaping effect was amplified by the following: (1) increased liquid–liquid interfaces, due to an increased number of emulsified particles or reduced particle sizes; (2) increased solid–liquid interfaces caused by high-melting-point solid components; and (3) increased gas–liquid/solid interfaces induced by rheology modifiers.



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INTRODUCTION

Advancements in society, science, technology, and living standards have led to increases in the pursuit of beauty, naturalness, health and safety. Cosmetics are no longer a luxury item for a few; instead, they have become an indispensable part of people's daily lives.[1] With the developments of carbon neutrality goals, the concepts of sustainable development and clean beauty have gained increasing popularity, and natural cosmetics are increasingly favored by people.[2-4] According to ISO 16128-1/2, natural cosmetic products refer to products with ingredients that are mainly derived from natural resources (eg, plants, animals, minerals), extracted through physical methods, and lack harmful chemical substances. Therefore, when developing naturally sourced cosmetics, there are many restrictions regarding the selection of raw materials. Therefore, the stability, performance, and skin feel of the products will vary.

During the development of cosmetics, the skin feel of a product is an important aspect to evaluate. Sensory evaluations usually include assessments of spreadability, absorbability, thickness, greasiness, and the soaping effect. As consumers' requirements for products continue to increase, the importance of the soaping effect in sensory evaluation has also been increasing. The soaping effect refers to white traces that are visibly apparent on the skin during product application. This phenomenon tends to be more pronounced when products include naturally sourced oils, emulsifiers and polymers, especially emulsified products with high viscosity. At present, there is no systematic research on the mechanisms underlying the soaping effect; however, there has been speculation regarding the main causes. Firstly, solid fats have a melting temperature higher than human skin temperature; thus, during application, white streak appear when the fats have not melted.[5] Secondly, excessive surfactants generate bubbles during application. The rheological regulators or solid fats in the system may help to stabilize the bubbles, resulting in white streaks on the sample during application. As the sample absorbs, the bubbles burst, and the streaks disappear. Silicones—particularly cyclic siloxanes (D4, D5, D6)—effectively reduce the soaping effect in formulations and provide a light skin feel due to their unique properties such as low surface tension, low viscosity, and volatility.[6] However, silicone oil is not a natural ingredient and has environmental issues, such as difficulty in degradation. Therefore, it cannot be used in natural cosmetic products, thus limiting the availability of high-quality natural care products.

Therefore, this paper systematically examined the influence of single-factor changes in formula compositions on the soaping effect during the application process of emulsion samples. Various emulsifiers were examined, and the effects of fatty alcohols, solid greases, and liquid oils were studied. Furthermore, this study examined how the formation of liquid–liquid, solid–liquid and gas–liquid interfaces impacted the occurrence of the soaping effect, thus providing a theoretical basis for the preparation of high-quality natural creams.

EXPERIMENTAL SECTION

MATERIALS

The raw materials used in this study are shown in Table I.

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Table I
Material Information

Category	INCI Name	Abbreviation	Company
Emulsifiers	Polyglyceryl-6 Distearate (and) Euphorbia Cerifera (Candelilla) Wax/Jojoba Esters/Oryza Sativa (Rice) Bran Polyglyceryl-3 Esters	DM	Gattefossé
	Cetyl Alcohol (and) Stearyl Alcohol (and) Glyceryl Stearate (and) Jojoba Esters (and) Helianthus Annuus (Sunflower) Seed Wax (and) Sodium Stearoyl Glutamate (and) Water (and) Polyglyceryl-3	DC	
	Polyglyceryl-6 Distearate	SQ	
	Polyglyceryl-6 Distearate (and) Jojoba Esters (and) Polyglyceryl-3 Beeswax (and) Cetyl Alcohol	MF	
	Cetyl Alcohol (and) Glyceryl Stearate (and) PEG-75 Stearate (and) Ceteareth-20 (and) Steareth-20	DT	
	Cetearyl Alcohol (and) Cetearyl Glucoside	M68	SEPPIC
	Cetearyl Olivate (and) Sorbitan Olivate	OLM	Host
	Sodium Hydrogen N-(1-oxooctadecyl)-L-glutamate	SG	BASF
	Polyglyceryl-3 Methylglucose Distearate	Care 450	Evonik
Fatty Alcohols	Cetearyl Alcohol	CA	Emuly
	Behenyl Alcohol	BA	BASF
	Stearyl Alcohol	SA	
Oils and Fats	Jojoba Esters (and) Helianthus Annuus (Sunflower) Seed Wax (and) Acacia Decurrens Flower Wax (and) Polyglyceryl-3 (and) Tocopherol	AC	Gattefossé
	Octyldodecyl Myristate	MOD	
	C10-18 Triglycerides	LA	
	Diethylhexyl Ether	DE	
	Butyrospermum Parkii (shea) Butter	SB45	BASF
	Undecane (and) Tridecane	ULM	
	Hydrogenated Coco-Glycerides	SFT	IOI
	Limnanthes Alba (Meadowfoam) Seed Oil	WPF	Heyi Commercial Group Shanghai Branch
	Beeswax	BSW	Vantage
	Simmondsia Chinensis (Jojoba) Seed Wax	JJBW	
	Squalane	SQU	
	Simmondsia Chinensis (Jojoba) Seed Oil	JJB	
	Caprylic/Capric Triglyceride	GTCC	BASF
Rheology Modifiers	Xanthan Gum	CGT	Azelis
	Carrageenan	CRG	CP Kelco
	Sclerotium Gum	STG	Cargill
	Carbomer	U20	Lubrizol
pH Modifiers	Citric Acid	CTA	Titan
Preservatives	Phenoxyethanol	PhE	Sunpu BioChem
	Ethylhexylglycerin	PE9010	

INCI: International Nomenclature Cosmetic Ingredient

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PREPARATION OF EMULSIONS

General emulsification method. The general emulsification method was as follows. (1) The oil phase and water phase were weighed separately and heated to 75°C. (2) Phase A was slowly added into phase B while stirring at 560 rpm for 3 minutes in a water bath using a digital overhead stirrer (EUROSTAR 20, IKA, China). (3) Subsequently, the water bath was homogenized at 12,000 rpm for 3 min at 75°C using a homogenizer (T18, IKA, China). (4) After stirring at 400 rpm and cooling to less than 50°C, phase C was added. (5) Finally, the product was stirred and cooled to room temperature.

High-energy emulsification method. The high-energy emulsification method was as follows. (1) The oil phase and water phase were weighed separately and heated to 75°C. (2) Phase A was slowly added into phase B while stirring at 560 rpm for 3 minutes in a water bath using a digital overhead stirrer (EUROSTAR 20, IKA, China). (3) Subsequently, ultrasonic treatment was carried out under a water bath at 75°C using an ultrasonic disperser (SCIENTZ-CP750, Scientz, China) with an ultrasonic power of 600 W and a treatment time of 15 min (5 s of ultrasonic operation followed by a 5-s interval). (4) The product was stirred at 400 rpm and cooled to below 50°C. Then, phase C was added. (5) Finally, the product was stirred and cooled to room temperature.

MICROSCOPIC OBSERVATION

A biological microscope (BX53, Olympus, Japan) was used to observe the microstructure of the samples before application and at the time when the white streaks were most pronounced. The samples were collected using a spoon and coverslip. The coverslip was gently covered and immediately observed under the microscope.

DIFFERENTIAL SCANNING CALORIMETRY

A differential scanning calorimeter (DSC3, METTLER TOLEDO, China) was used to test the heat flow curve corresponding to the temperature increase from 0°C to 80°C to derive the phase transition temperature of the feedstock. The testing process was carried out under nitrogen protection with an airflow rate of 50 mL/min. The temperature increase rates were all 5°C/min.

EMULSION DROPLET SIZE DETERMINATION

The Zetasizer Nano ZS (90PLUS PALS, Nano Brook, Brookhaven, USA) was used in dynamic light scattering (DLS) mode to determine size of the emulsion particles. Three sets of parallel experiments were set up for each sample to ensure the accuracy of the data.

RHEOMETRY

A rheometer (DHR 20, TA Instrument Company, USA) was used to examine the rheological behavior of carbomer aqueous solutions and emulsions with different concentration gradients under a constant temperature of 25°C. By comparing the viscosity curves of different samples, the rheological characteristics of the samples were analyzed. The shear rate range was 0.01–100 s^{-1}.

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RESULTS AND DISCUSSION

ESTABLISHMENT OF A STANDARD METHOD FOR EVALUATING THE SOAPING EFFECT

The soaping effect is an important indicator in sensory evaluation. Establishing a standardized method for sensory evaluation is a prerequisite for objectively and accurately quantifying the soaping effect. However, to date, there is no established method for direct application. To ensure the objectivity and accuracy of soaping effect evaluations in subsequent studies, this study first systematically investigated and compared multiple methods for triggering the soaping effect. Subsequently, a standardized method that conformed to daily application scenarios and can reliably produce the soaping effect. The inner side of the forearm was selected as the test site, and a 4-cm^2 area was marked. This area was first cleansed with a standard facial cleanser and then dried using a tissue. After being placed in a constant temperature and humidity environment (25°C ± 1°C, 50% ± 5% RH) for 30 minutes, 0.08 mL of the sample was applied to the test area by moving the pad of the index finger in a circular motion at a constant rate (5 circles per second). The development of the soaping effect was observed and recorded. This study also established a scoring system for the soaping effect, as shown in Figure 1. When the soaping effect was obvious, it was recorded by taking photos with a mobile phone. Subsequently, the degree of the soaping effect was scored and graded according to the following criteria: a score of 0 indicated no white traces; a score of $0 < x < 2$ indicated minimal white traces; a score of $2 \leq x < 4$ indicated a small amount of white traces; a score of $4 \leq x < 6$ indicated a moderate amount of white traces; a score of $6 \leq x < 8$ indicated a large amount of white traces; a score of $8 \leq x < 10$ indicated a very large amount of white traces. All subsequent related studies strictly adopted this sample application method and scoring system to ensure standardization.

EFFECT OF PRODUCT COMPOSITION ON THE SOAPING EFFECT

Emulsifiers. During the preparation of emulsions, the type of emulsifier directly affects multiple properties of the system, such as the emulsification effect, viscosity, and skin feeling. Therefore, 9 commonly used natural emulsifiers were selected, and the effects of emulsifier structure on the soaping effect were investigated. The amount of emulsifier added in the emulsion formulation was fixed at 4 wt.%, and the rest of the product composition is shown in Table II. The micrographs of the prepared emulsions (which were taken 24 h

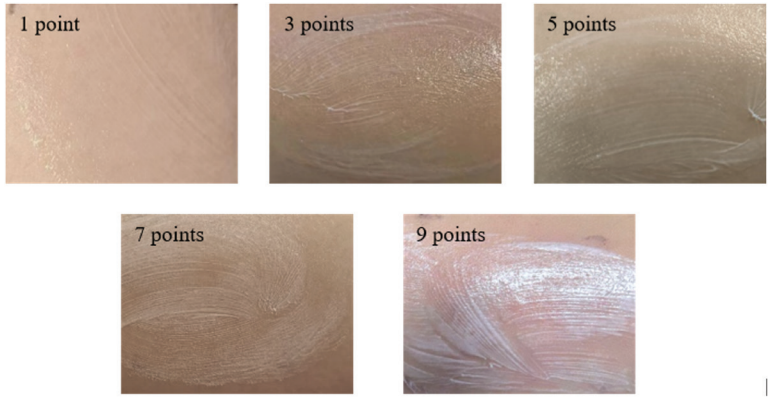


Figure 1. Standard scoring chart for the soaping effect.

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Table II
Fundamental Formulation

Phase	Raw Material Name	Mass Fraction(%)
A	Emulsifier	4.0
	CA	1.5
	MOD	8.0
	DE	3.0
	GTCC	5.0
B	Water	To 100
	Gly	5.0
	CGT	0.3
C	10% CTA	Adjust to pH=5.5
	PE9010	0.8

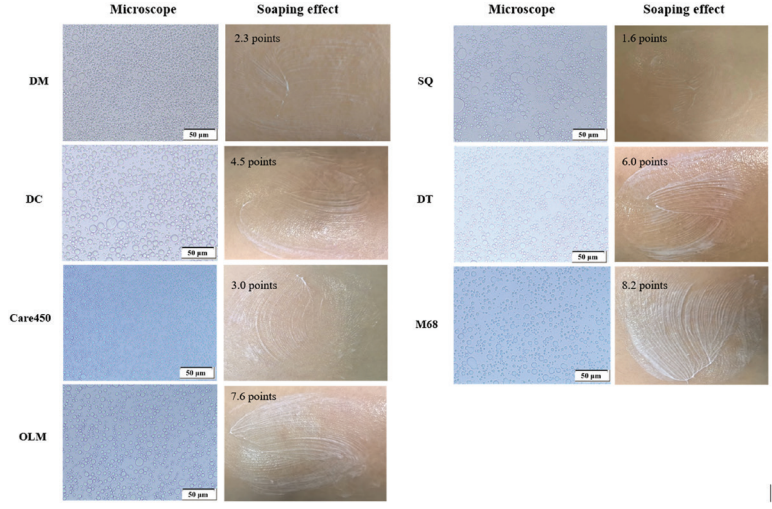


Figure 2. Microscopic images and photos of the soaping effect prepared with different emulsifiers.

after preparation) and the photos of the soaping effect (which were taken immediately after application) are shown in Figure 2. The summary of the soaping effect is shown in Table III.

As shown in Figure 2 and Table III, all samples exhibited favorable emulsification performance, with the emulsified particles being uniformly dispersed and morphologically regular. Polyglyceryl-6 distearate (SQ), DM, and Care450 exhibited a relatively mild soaping effect, whereas DC, DT, cetearyl olivate and sorbitan olivate (OLM), and cetearyl alcohol and cetearyl glucoside (M68) demonstrated a stronger soaping effect. A comparative analysis of the compositional and structural characteristics revealed that DM and SQ are primarily composed of SQ, while cetearyl alcohol (CA) was present in DC, M68, and OLM. Based on these observations, it was hypothesized that CA may play a direct role in triggering the soaping effect. Therefore, DM, which exhibited a weaker tendency to produce this effect, was selected as the primary emulsifier in subsequent investigations to evaluate the individual impact of other components on the soaping effect.

Fatty alcohols. Fatty alcohols represent a class of oily, raw materials that are commonly utilized in cosmetics. Their physicochemical properties, as well as their influence on emulsification performance, are closely related to the number of carbon chains. In this

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Table III
Summary of the Soaping Effect of Samples Prepared With Different Emulsifiers

Emulsifier	Grading of the Soaping Effect	Soaping Effect score
DM	Small	2.3
SQ	Slight	1.6
DC	Medium	4.5
DT	Large	6.0
Care450	Small	3.0
M68	Extra Large	8.2
OLM	Large	7.6

study, DM was selected as the fixed emulsifier at a concentration of 4 wt.%, while various fatty alcohols were incorporated at a constant dosage of 1.5 wt.%. The impact of fatty alcohols with different carbon chain lengths (CA, stearyl alcohol [SA], behenyl alcohol [BA]) on the soaping effect was investigated separately. The micrographs of the samples prepared with different fatty alcohols (which were taken 24 h after preparation) and the photographs of the soaping effect (which were taken immediately after application) are shown in Figure 3. The summary of the soaping effect is shown in Table IV.

As illustrated in Figure 3, CA demonstrated a positive effect on emulsification performance. However, an increase in carbon chain length resulted in adverse effects on emulsification, leading to progressively sparse and non-uniform emulsified particles. By integrating Figure 3 and Table IV, it can be observed that as the carbon chain length of fatty alcohols increased, the soaping effect gradually intensified. Specifically, the CA system only showed a trace amount soaping (corresponding to the score range $0 < x < 2$), while the BA system generated a large amount of soaping (score range $6 \leq x < 8$). This phenomenon may be related to the change in the melting point of fatty alcohols. An increase in the carbon chain length raised the melting point of fatty alcohols. Compared with CA (a mixture of C16

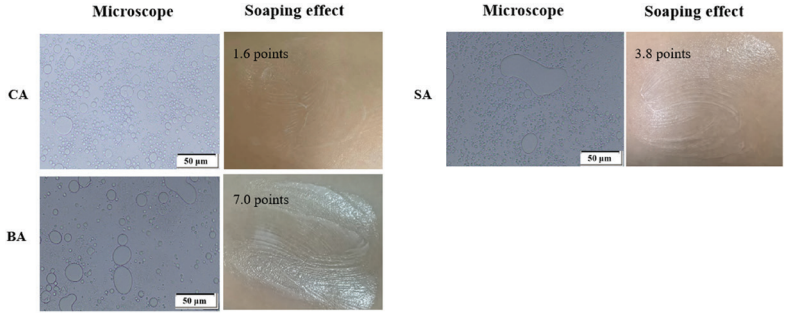


Figure 3. Micrographs and photographs of the soaping effect among samples prepared with fatty alcohols.

Table IV
Summary of the Soaping Effect Among Samples Prepared with Different Fatty Alcohols

Fatty Alcohol	Grading of the Soaping Effect	Soaping Effect Score
CA	Slight	1.6
SA	Small	3.8
BA	Large	7.0

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and C18), BA (C22) has a longer carbon chain, resulting in a higher melting point.[7] The increase in melting point not only slowed down the melting rate of the oil but also delayed the absorption rate of the system on the skin, thereby intensifying the soaping effect.

Solid greases. Solid grease constitutes a category of frequently incorporated solid raw materials in cosmetics. Their distinct structural characteristics result in variations in melting points and differential impacts on emulsification performance.

In this study, DM was employed as the fixed emulsifier at a concentration of 4 wt.%, CA was employed as the fixed fatty alcohol at a concentration of 1.5 wt.%, and solid grease was added consistently at 3 wt.%. The rest of the formulation composition is shown in Table II. The influence of different solid grease on the soaping effect was systematically examined. The micrographs of the samples prepared with different solid grease (which were taken 24 h after preparation) and the photographs the soaping effect (which were taken immediately after application) are shown in Figure 4. The summary of the soaping effect is shown in Table V.

As illustrated in Figure 4, different solid grease exhibited distinct effects on the emulsification performance of the system. The addition of simmondsia chinensis (jojoba) seed wax (JJBW) and beeswax (BSW) significantly impaired the emulsifying effect, resulting in irregular and disorganized particle structures, whereas butyrospermum parkii (shea) butter (SB45), AC, and C10-18 triglycerides (LA) had relatively minor impacts on the emulsion microstructure. By integrating the microstructural observations from Figure 4 with the soaping effect scores summarized in Table V, it became evident that all solid grease except for LA, exacerbated the soaping effect to varying degrees. This trend may be attributed to their higher melting

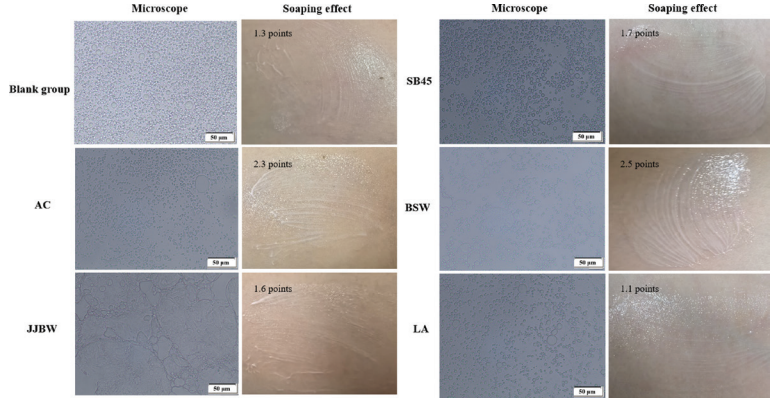


Figure 4. Micrographs and the soaping effect images of samples prepared with different solid grease.

Table V
Summary of the Soaping Effect Among Samples Prepared with Different Solid Greases

Solid Grease	Grading of the Soaping Effect	Soaping Effect Score
Blank	Slight	1.3
SB45	Slight	1.7
AC	Small	2.3
BSW	Small	2.5
JJBW	Slight	1.6
LA	Slight	1.1

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points, increased system viscosity, and delayed absorption kinetics.[8] Notably, in the case of JJBW, its strong destabilizing influence on emulsification overshadowed any potential benefit derived from its elevated melting point, thereby leading to a comparatively lower overall soaping effect score. In summary, the mechanism by which solid grease influenced the soaping effect was complex and multifactorial. It was preliminarily hypothesized that, under conditions of similar emulsification performance and oil-water interfacial film area, higher melting points are associated with a greater tendency to intensify the soaping effect.

Liquid oils. Liquid oils constitute a primary component of the oil phase in cosmetic cream products. Variations in their molecular structures lead to differences in absorption behavior, spreading properties, and impacts on emulsification performance. To systematically investigate the influence of liquid oils on the soaping effect, this study employed DM as the fixed emulsifier at a concentration of 4 wt.%, CA was employed as the fixed fatty alcohol at a concentration of 1.5 wt.%, no solid oils were added, with a consistent liquid oil addition level of 8 wt.%, and the rest of the formulation composition was shown in Table II. The effects of 8 representative liquid oils, spanning 4 major chemical categories, on the soaping effect were evaluated. The micrographs of the samples prepared with different liquid oil (which were taken 24 h after preparation) and the photographs of the soaping effect (which were taken immediately after application) are shown in Figure 5. The summary of the soaping effect is shown in Table VI.

As shown in Figure 5, the studied emulsion system demonstrated good compatibility with various types of liquid oils. Except for the squalane (SQU) sample, which showed slightly uneven emulsion particles, the emulsification effect of the other systems did not change significantly. By integrating the information from Figure 5 and Table VI, it was observed that compared with the reference sample octyldodecyl myristate (MOD), the soaping effect produced

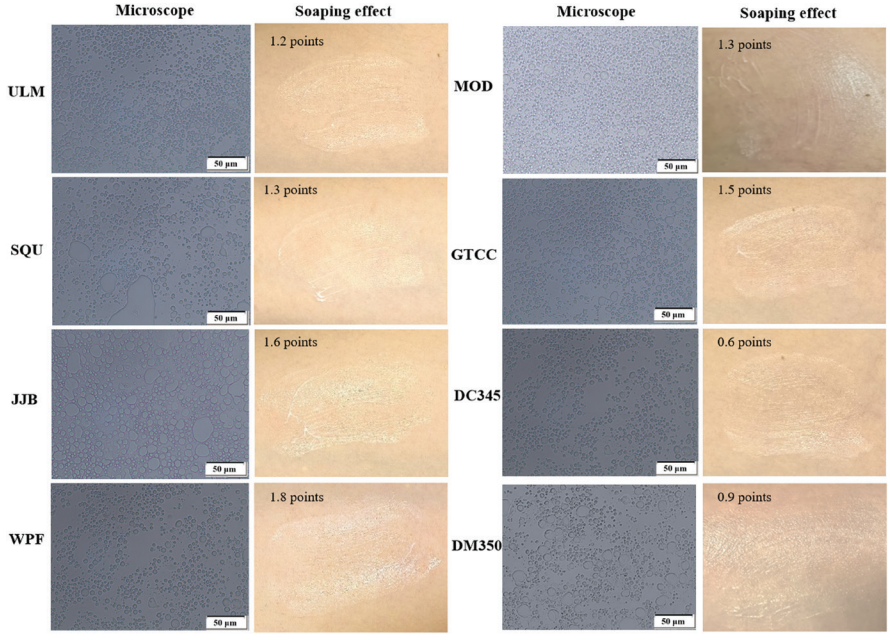


Figure 5. Micrographs and photographs of the soaping effect among samples prepared with different liquid oils.

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Table VI
Summary of the Soaping Effect Among Samples Prepared with Different Liquid Oils

Type	Liquid Oil	Refractive Index	Grading of the Soaping Effect	Soaping Effect score
Alkanes	ULM	1.42-1.43	Slight	1.2
	SQU	1.45	Slight	1.3
Vegetable Oil	JJB	1.47	Slight	1.6
	WPF	/	Slight	1.8
Ester Oil	MOD	1.46	Slight	1.3
	GTCC	1.45	Slight	1.5
Silicone Oil	DC345	1.39∼1.46	Slight	0.6
	DM350	1.40	Slight	0.9

by alkanes (eg, undecane and tridecane [ULM], SQU) was minimal. The soaping effect of vegetable oils (eg, simmondsia chinensis (jojoba) seed oil [JJB], limnanthes alba (meadowfoam) seed oil [WPF]) was slightly enhanced, potentially due to the presence of long-chain fatty acids within their triglyceride structures. The long-chain fatty hydrocarbon structure might promote the crystallization and solidification of the system or reduce the solvation efficiency through intermolecular forces, thereby enhancing light scattering.[9] The influence of ester oils (eg, caprylic/capric triglyceride [GTCC]) on the soaping effect was more complex and did not follow a clear rule. Notably, silicone oils (eg, DC345, DM350) significantly reduced the soaping effect, which was consistent with traditional empirical results. It was speculated that this reduction might be related to their lower refractive index and higher volatility.

The refractive index (n) is defined as the ratio of the speed of light in a vacuum (c) to the speed of light in the medium (v) ($n = c/v$).[10] A lower refractive index of the oil phase reduced the refractive index difference (Δn) between the oil phase and the aqueous phase after application. According to Mie scattering theory or Rayleigh scattering theory, the intensity of light scattering is proportional to the square (or higher powers) of Δn.[11] Therefore, the reduction of Δn greatly weakens the scattering effect of the oil–water interface on incident light, which is the key mechanism for the emulsion to present a nontransparent white appearance.[12] A lower refractive index reduced the scattering of light by the film formed after the emulsion waxes applied, thereby visually reducing the appearance of the soaping effect. As shown in the refractive index data in Table VI, DM350 and ULM (short-chain alkanes) had lower refractive indices, and their corresponding soaping effects were also weaker. Additionally, the elimination effect of silicone oil on the soaping effect also involves its dynamic behavior characteristics. The main chain of silicone oil molecules was composed of silicon-oxygen bonds (Si-O-Si), and the side chains were usually methyl groups (polydimethylsiloxane [PDMS]) or other organic groups. Low-viscosity silicone oils (eg, DC345, cyclopentyl dimethylsiloxane and cyclohexyl dimethylsiloxane) often had higher saturated vapor pressures.[13] The spatial hindrance of the cyclic structure and the low rotational barrier of the silicon-oxygen bond make the intermolecular forces of silicone oils much weaker than those of long-chain alkane oils. This led to higher molecular mobility and stronger volatility.[14] When applied to the skin, low-molecular-weight siloxanes, due to their high volatility (rapid evaporation) and low surface tension (excellent spreading, easy penetration and migration), can significantly accelerate the distribution and absorption of the material on the skin surface.[15] This rapid physical state change reduced the formation and stable existence time of non-uniform, highly scattering microscopic structures in the initial application stage. Therefore, from a visual perspective, the soaping effect produced was not only weaker in intensity but also had a significantly shorter duration.

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THE MECHANISM UNDERLYING THE SOAPING EFFECT

Based on single-factor analysis, this study proposes a core mechanistic hypothesis: The soaping effect is an optical phenomenon that is driven by the formation of new phase states and the synergistic variation of the three-phase interfacial area. During the application of cosmetic emulsified products, several interfaces exist and undergo dynamic transformations. (1) The intrinsic liquid–liquid interface within the emulsion system and the solid–liquid interface are introduced by solid raw materials. (2) During the spreading process, under the influence of shear forces and absorption, water gradually evaporates while oils are absorbed, leading to an increase in the proportion of solid–phase components. This results in an expansion of the solid–liquid interface. (3) As the application continues, repeated contact between the fingertip ridges and the product introduces air, forming a gaseous phase and consequently generating new gas–liquid and gas–solid interfaces. With the formation or expansion of these interfaces, the soaping effect gradually becomes stronger. Therefore, this study investigated the underlying mechanism of the soaping effect from three interfacial perspectives: the liquid–liquid interface, the solid–liquid interface, and the gas–liquid / gas–solid interfaces.

Liquid–liquid interface. When the emulsifier introduced into the oil-water system, it adsorbed at the interface and formed an interfacial film, thereby effectively reducing the interfacial tension. Simultaneously, mechanical energy input through processes such as stirring and homogenization facilitated the formation of new liquid–liquid interfaces between the originally immiscible oil and water phases. During the application process, the presence of liquid–liquid interfaces generated different optical effects, thereby influencing the intensity of the soaping effect phenomenon.

Consequently, this study investigated the influence of the liquid–liquid interface on the soaping effect by systematically regulating both the volume fraction of the dispersed phase and the size distribution of emulsified particles.

Dispersed–phase ratio. With a comparable particle size distribution of the emulsified droplets, an increased volume fraction of the dispersed phase corresponded to a greater total liquid–liquid interfacial area within the system. To elucidate the mechanism by which the expansion of the interfacial area influenced the soaping effect, which induced by variations in the dispersed–phase proportion, this study selected SQ, a structurally uniform compound, as the primary emulsifier. Under controlled conditions of raw material composition and preparation methodology, the impact of the dispersed–phase proportion on the soaping effect was systematically investigated. This study utilized commercially available gel products and the basic formulation detailed in Table VII. The formulation table is shown below, and the preparation process was the same as the ordinary emulsification method described above.

As shown in Figure 6 and Table VIII, with the increase of the proportion of the dispersed phase, the soaping effect gradually increased from no soaping to a trace amount of soaping, and the score shows an overall increasing trend. This finding suggests that the higher the content of the dispersed phase, the stronger the soaping effect will be. Therefore, there was a positive correlation between the strength of the soaping effect and the liquid–liquid interface area. However, since the oil in this formula system was a liquid oil with good affinity to the skin and no other solid-phase substances were introduced except for the emulsifier, the increase in the soaping effect was relatively small.

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Table VII
Sample Formulations with Different Dispersed–Phase Proportions

Dispersed–Phase Ratio		10%	20%	30%
Phase	Raw Material Name		Mass Fraction (wt%)	
A	SQ		5.00	
	GTCC	5.00	15.00	25.00
B	Gly		5.00	
	CGT		0.30	
	Water		TO 100.00	
C	PE9010		0.80	

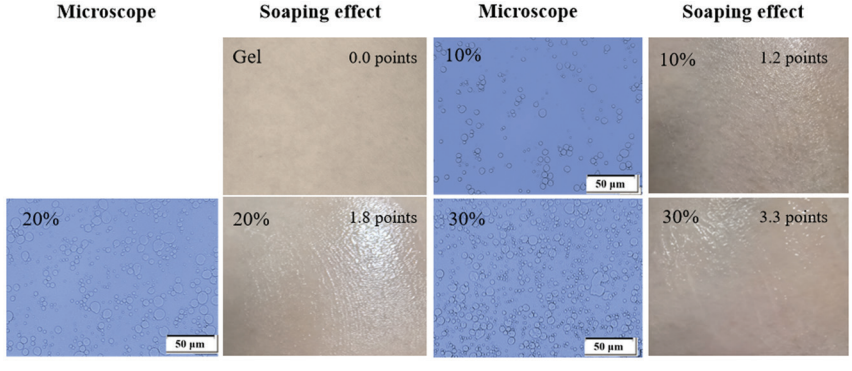


Figure 6. Micrographs and photographs of the soaping effect among samples prepared with different dispersed–phase proportions.

Table VIII
Summary of the Soaping Effect Among Samples Prepared with Different Dispersed–Phase Proportions

Dispersed Phase Content (wt%)	Grading of the Soaping Effect	Score of the Soaping Effect
0.00 (Gel)	None	0.0
10.00	Slight	1.2
20.00	Slight	1.8
30.00	Slight	3.3

Emulsified Particle Size. An emulsion is a highly dispersed system in which the dispersion of the dispersed phase requires external energy input. During this process, the liquid–liquid interface gradually stabilizes, resulting in the formation of emulsion particles. Variations in particle size can lead to significant differences in the total liquid–liquid interfacial area.[16] To elucidate the mechanism by which the expansion of interfacial area influences the soaping effect induced by the reduction in emulsion particle size, this study employed the base formulation detailed in Table IX and prepared the samples using both conventional emulsification and high-energy emulsification methods.

As shown in Figure 7 and Table X, samples with particle sizes of 2500.00 nm and 201.78 nm were prepared using two different emulsification methods, resulting in significantly different manifestations of the soaping effect. As the emulsification particle size decreased, the intensity of the soaping effect increased from trace to slight. The principle underlying ultrasonic action lies in its ability to generate a cavitation effect when propagating through

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Table IX
Sample Formulations with Different Preparation Processes

Phase	Raw Material Name	Mass Fraction (wt%)
A	SQ	5.00
	GTCC	10.00
B	Glycerin	5.00
	CGT	0.30
	Water	TO 100.00
C	PhE	0.10

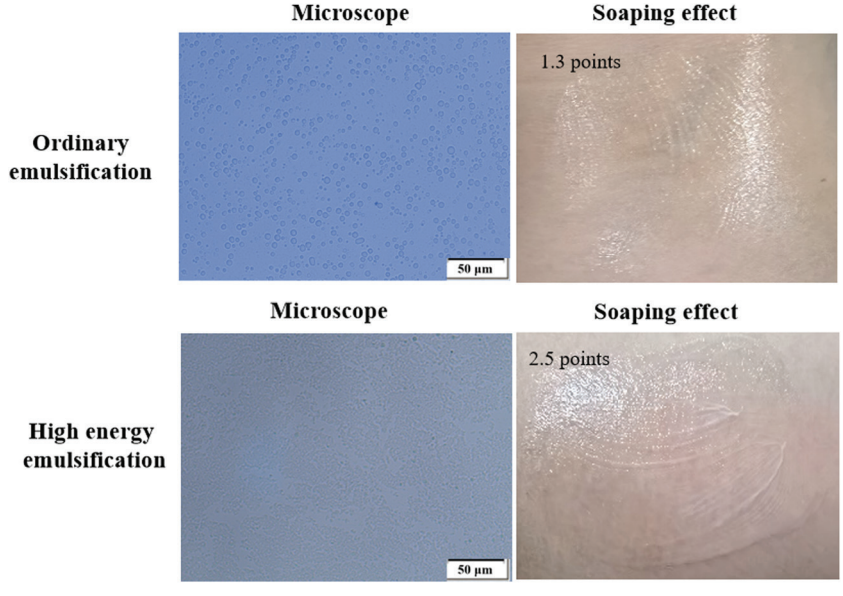


Figure 7. Micrographs and photographs of the soaping effect among samples prepared with different preparation processes.

a liquid medium. This cavitation effect produces instantaneous high-pressure impacts that break down particles through acoustic energy, thereby enhancing the dispersion of nanoemulsions and reducing the particle size of the emulsified droplets.[17] Under constant dispersed phase volume, a substantial reduction in emulsification particle size corresponded to a significant increase in the liquid–liquid interfacial area. Furthermore, due to their smaller size, nanoemulsions can distribute more uniformly on the skin surface and exhibit strong resistance to shear forces.[18] Consequently, the stability of the liquid–liquid interface was improved, leading to a noticeable enhancement of the soaping effect.

Solid–liquid interface. The emulsion comprises various solid components, including emulsifiers and solid grease. During the cooling and storage stage of the emulsion, these solid constituents undergo gradual solidification and precipitation, displaying characteristic crystallization behavior. Typically, under consistent storage conditions, solid substances with higher melting points exhibit faster crystallization rates and greater degrees of crystal regularity.[19] Thus, during the application process, the white traces that appeared due to the increase of the solid–liquid interface caused by the insufficient melting of solid substances

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Table X
Summary of the Soaping Effect Among Samples Prepared with Different Preparation Processes

Emulsification Method	Particle Size (nm)	Grading of the Soaping Effect	Score of the Soaping Effect
Low-Energy Emulsification	2500.00	Slight	1.3
High-Energy Emulsification	201.78	Small	2.5

were also recognized as one of the contributing factors to the soaping effect. Therefore, this study investigated the influence of the solid–liquid interface on the soaping effect by analyzing both the melting points of solid raw materials and the quantity of high-melting-point substances incorporated.

The differential scanning calorimetry (DSC) curves (Figure 8) were obtained from the heat profiles of different solid ingredients to reveal the influence of melting point on the soaping effect. The micrographs and the photographs of the soaping effect among samples prepared from different solid oils are shown in Figure 4, and the soaping effect is summarized in Table XI.

Thermodynamic analysis of solid raw materials (as depicted in Figure 8) indicated a substantial positive correlation between the melting points of solid components and the soaping effect in samples. Specifically, the intensity of white traces escalated with

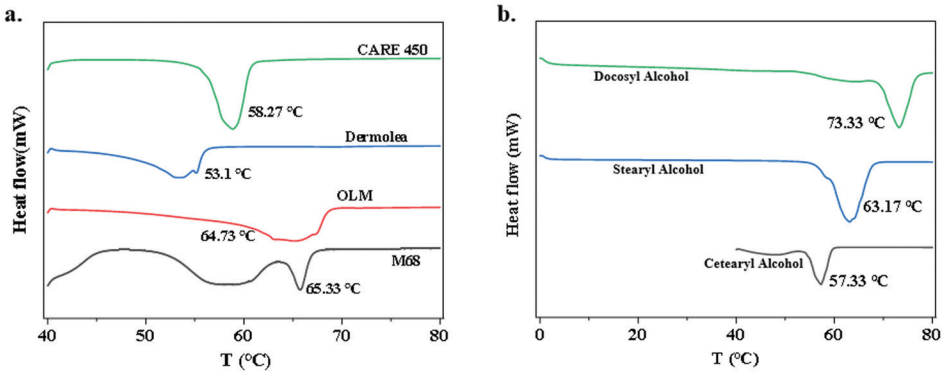


Figure 8. DSC curves of different solid raw materials, including (A) emulsifier and (B) fatty alcohols.

Table XI
Microscopic and Macroscopic Images of Samples Prepared With Different Melting Points

Solid Raw Materials	Fusing Point (°C)	Grading of the Soaping Effect	Score of the Soaping Effect
DM	53.10	Small	2.3
CARE 450	58.27	Small	3.0
OLM	64.73	Large	7.6
M68	65.33	Extra Large	8.2
CA	57.33	Slight	1.6
SA	63.17	Small	3.8
BA	73.33	Large	7.0

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an increase in melting points. This phenomenon could be ascribed to the differential absorption kinetics during the application of the product. During the process, liquid oils were gradually absorbed by the skin, whereas solid components remain unabsorbed until they reach their melting points.[20] Under continuous shear stress, the proportion of solid materials on the skin surface raised, thereby expanding the solid–liquid interface area. Macroscopically, this expansion was manifested as the soaping effect. Solid components with higher melting points exhibited slower absorption rates, which ultimately amplified the soaping effect. The underlying mechanism primarily stems from optical scattering induced by incompletely melted solid–phase particles within the system. This scattering effect is progressively amplified through phase separation kinetics during product application. The detailed mechanism is outlined as follows. (1) In systems containing solid particles larger than 400 nm (eg, partially solid-state components), significant Mie scattering occurs due to refractive index differences between these particles and the surrounding continuous phase. This results in the immediate appearance of visible white streaks upon initial application and persists throughout the cooling and solidification phase of the formulation. (2) During the application process, this phenomenon is further enhanced through a synergistic interplay of multiple factors: rapid migration of liquid oil into the stratum corneum increases resistance to particle movement; progressive water evaporation leads to an elevated volume fraction of the solid phase; and shear forces generated during application promote the aggregation of solid-phase particles into larger clusters. Collectively, these dynamic processes culminate in the formation of persistent white residues on the skin surface. Consequently, the inclusion of solid fats emerged as a crucial factor that influences the manifestation of the soaping effect.

THE DOSAGE OF HIGH–MELTING–POINT RAW MATERIALS

To further validate the influence of high-melting-point substances on the soaping effect, CA was selected as a representative. This study employed SQ as the fixed emulsifier at a concentration of 4 wt.%; CA was employed at concentrations of 0, 1.5, and 3 wt.%; and the rest of the formulation composition is shown in Table II. The resulting variations in soaping effect were systematically compared and are detailed in Figure 9 and Table XII.

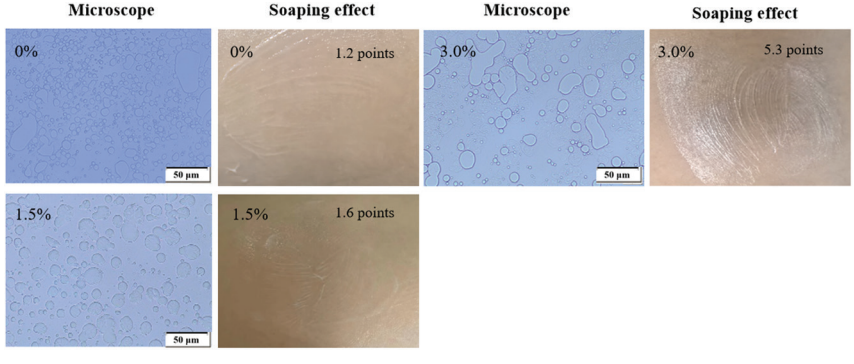


Figure 9. Micrographs and photographs of the soaping effect among samples prepared with different dosages of CA.

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Table XII
Summary of the Soaping Effect of Samples Prepared With Different Dosages of Cetearyl Alcohol

CA/%	Grading of the Soaping Effect	Grading of the Soaping Effect
0.00	Slight	1.2
1.50	Slight	1.6
3.00	Medium	5.3

As shown in Figure 9 and Table XII, the incorporation of high–melting–point CA initially decreased and subsequently increased the emulsion particle size. However, the soaping effect showed a progressive linear increase with increasing dosage, rising from an initial slight level (1.2) to a medium level (5.3). Therefore, in the formulation of natural cosmetic products, it was recommended to minimize the use of high-melting-point substances with long carbon chains, lower phase transition temperatures, and restrict the formation of solid–liquid interfaces.

Gas–liquid/solid interface. During the application of the emulsion, as the liquid spreads across the skin surface, the solid–liquid interface gradually replaces the original solid–gas interface. Continued application of the sample introduces entrained air, leading to the formation of a new gas–liquid/solid interface. The presence of entrained air alters the phase state, which visually manifests as the soaping effect. The addition of excessive surfactants or a substantial quantity of rheological modifiers can enhance the stability of the gas–liquid/solid interfacial film. Accordingly, the experiment examined the correlation between the gas–liquid/solid interface and the soaping effect from these two perspectives.

Emulsifiers. In emulsion systems, excess emulsifiers were commonly added to ensure system stability. Emulsifiers stabilized both gas–liquid and liquid–liquid interfaces by reducing their respective interfacial tensions. Consequently, free emulsifiers present in the system typically exhibited both foaming and foam-stabilizing properties, which the industry frequently associated with the soaping effect observed by consumers. Based on this understanding, the present study selected sodium hydrogen N-(1-oxooctadecyl)-L-glutamate (SG), an anionic emulsifier with pronounced foaming capacity, as the experimental variable. Single-factor experiments were conducted by controlling the SG concentration at 0, 0.5, and 1 wt.% to investigate the influence of emulsifiers on the soaping effect.

As shown in Figure 10 and Table XIII, with the increase of the SG concentration, the soaping effect intensified. Additionally, the images reveal that as the SG concentration increases to 1 wt.%, the number of bubbles in the white traces formed by the application gradually increased. This indicates that a greater volume of air was entrained and stabilized, forming an increased number of gas–liquid/solid interfaces. Consequently, the phase state undergoes modification, resulting in a more pronounced visual manifestation of the soaping effect.

RHEOLOGY MODIFIERS

During the application of the emulsion, air was entrained under shear stress, forming gas–liquid/solid interface with the sample. The incorporation of rheological modifiers substantially altered the viscosity of the emulsion system, resulting in enhanced air entrainment and stabilization, as well as a decreased absorption rate during application.

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Table XIII
Summary of the Soaping Effect Among Samples Prepared with Different Concentrations of SG

SG concentration (%)	Grading of the Soaping Effect	Grading of the Soaping Effect
0.00	Slight	1.6
0.50	Small	3.2
1.00	Medium	4.5

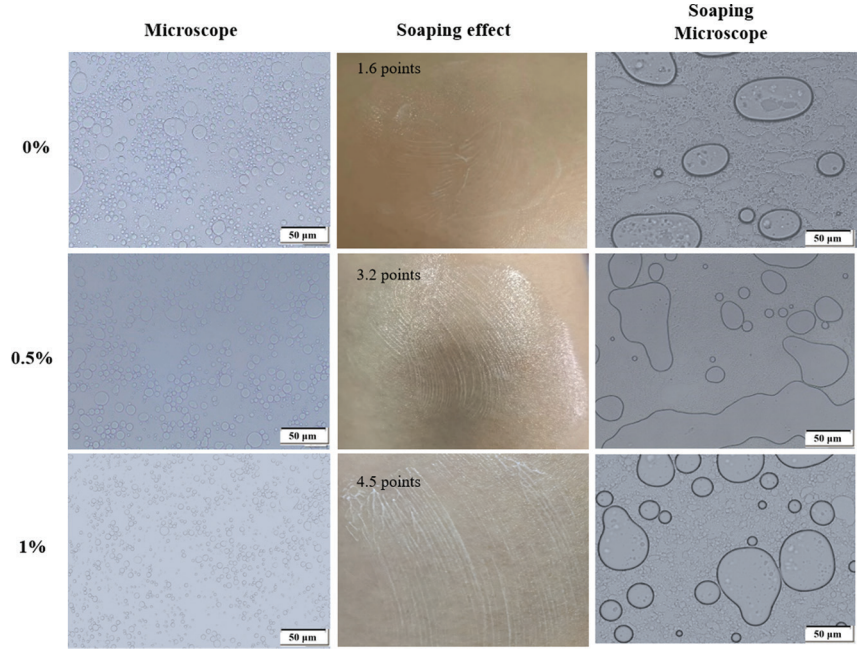


Figure 10. Micrographs and photographs of the soaping effect among samples prepared with different concentrations of SG.

These modifications influenced the properties and interfacial strength of both gas–liquid/solid interfaces, thereby affecting the soaping effect. Accordingly, this study selected DC as the primary emulsifier and prepared samples containing 0.3 and 0.5 wt.% xanthan gum (CGT) to investigate the impact of rheological modifiers on the soaping effect through their influence on the gas–liquid/solid interface stabilization.

As shown in Figure 11, Figure 12 and Table XIV, the viscosity of the samples increased with the incorporation of CGT, accompanied by a marked increase in the soaping effect from slight to large levels. This indicates a positive correlation between system viscosity and the intensity of the soaping effect. CGT, a naturally derived rheological modifier with a high molecular weight, primarily consists of heteropolysaccharides. Upon incorporation into the aqueous phase, it formed an interconnected three-dimensional network structure through intermolecular interactions such as hydrogen bonding, thereby significantly enhancing the system's viscosity.[21] During the application process, the original network structure was disrupted by shear forces, the aqueous phase gradually evaporates, and the liquid oil is absorbed over time.[22] The presence of CGT increases the viscosity of the system, which

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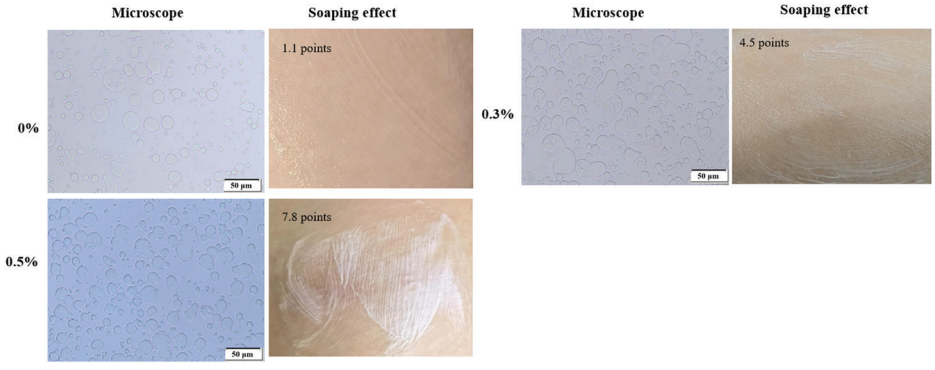


Figure 11. Micrographs and photographs of the soaping effect among samples prepared with the same rheology modifier at different viscosities.

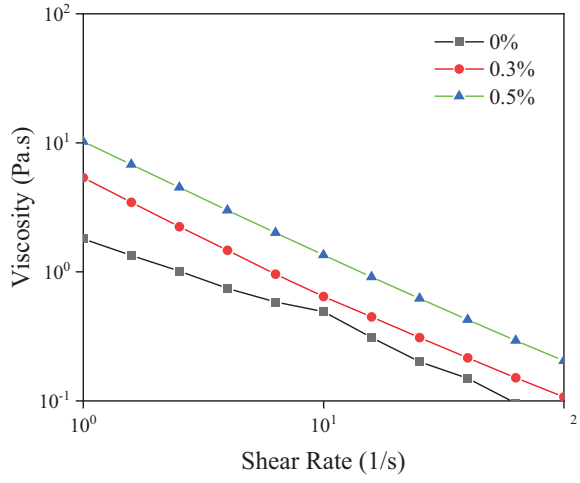


Figure 12. Viscosity curves of samples with different concentrations of rheological modifiers added.

Table XIV
Summary of the Soaping Effect Among Samples Prepared With the Same Rheology Modifier at Different Viscosities

CGT additions (%)	Grading of the Soaping Effect	Grading of the Soaping Effect
0.00	Slight	1.1
0.30	Medium	4.5
0.50	Large	7.8

enhanced the stability of the gas–liquid/solid interfacial film.[23] Consequently, this led to a more pronounced soaping effect, particularly when combined with high–melting–point solid ingredients on the skin surface. Moreover, the intensity of the soaping effect increased with higher concentrations of CGT, although it gradually diminished as both CGT and the solid components were eventually absorbed.

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CONCLUSION

The soaping effect is an optical effect, the intensification of which is frequently associated with the formation of new phase states. Through a systematic investigation into the underlying mechanisms of white traces formation, the following conclusions have been established. (1) A single-factor analysis of raw material composition within emulsion systems revealed that the presence of CA in emulsifiers intensified the soaping effect. The extent of this impact increased with the carbon chain length of fatty alcohols, and the quantity of visible white traces raised proportionally with their concentration. (2) During the preparation and application of emulsions, interfacial phase transition analyzed demonstrated that, under equivalent dispersed–phase ratios, the soaping effect became more pronounced with an increased number of emulsified particles and an expanded liquid–liquid interface area. Comparative studies on the influence of solid components with varying melting points indicated a dual mechanism by which these components affect the soaping effect, through both the generation of emulsified particles and the modulation of emulsion absorption rates. The inclusion of solid raw materials, naturally derived rheological modifiers, and high–melting–point substances all contributed to the enhancement of the soaping effect to differing extents. During application, as shear forces act upon the system and partial absorption occurs, the aqueous phase evaporated and liquid oil was gradually absorbed by the skin. This resulted in an increasing proportion of nonabsorbable solid constituents and rheological modifiers remaining on the skin surface. These components spread during application, became entrained into the gas phase, and give raised to new phase states, thereby producing the observable soaping effect. (3) Given the complex nature of emulsion compositions, the emergence of the soaping effect was influenced by multiple interacting factors, with phase transitions serving as the fundamental driving mechanism. These findings provide a theoretical basis for the formulation and application of high-performance natural emulsions in future research and development.

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