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"Research on influencing parameters of the soaping effect in skin care emulsions and its formation mechanism"

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1. Introduction

Whereas natural cosmetics are a major trend today, soaping effect is often cited as a drawback of such products. This phenomenon refers to the appearance of a white streak on the skin when a skin care product, generally an O/W emulsion, is applied. It is particularly disliked in Asian countries where transparency and quick absorption are preferred by consumers.

Often related to the presence of fatty alcohols in the formula, it is poorly documented, and formulators often use a test and trial method to avoid or decrease this phenomenon. Silicone oils are efficient to remove this unwanted effect, but natural cosmetics require solutions without their use.

The objective of this study is first to set up a method to evaluate the soaping on the skin, then to understand the influence of the formula composition on its formation and finally to explore the mechanism behind it.

2. Materials and Methods

Emulsifiers of natural origin have been selected to conduct this study. Their compositions are detailed in Table 1.

Table 1. Emulsifiers composition

Emulsifier	INCI name
A	polyglyceryl-6 distearate (and) candelilla/jojoba/rice bran polyglyceryl-3 ester
B	cetearyl alcohol (and) glyceryl stearate (and) jojoba esters (and) sunflower (helianthus annuus) seed wax (and), sodium stearoyl glutamate (and) water (and) polyglycerin-3
C	polyglyceryl-6 distearate
D	cetearyl alcohol (and) cetearyl glucoside
E	cetearyl olivate (and) cetearyl glucoside
F	polyglyceryl-3 methylglucose distearate

Others materials used in the study are fatty alcohols including cetearyl alcohol, stearyl alcohol and behenyl alcohol; emollients including octyldodecanol myristate, undecane (and) tridecane, camellia seed oil, jojoba oil, squalane, dicaprylyl ether, butylene glycol cocoate, caprylic/capric triglycerides; aqueous gelling agents including xanthan gum, carrageenan, sclerotium gum, carbomer; citric acid as pH adjuster; phenoxyethanol (and) ethylhexylglycerin as preservative system; and sodium stearoyl glutamate as co-emulsifier.

A basic formula has been set up as shown in Table 2.

Table 2. Basic formula

Phase	Ingredient	w/w (%)
A	Emulsifier	4.0
	Cetearyl alcohol	1.5
	Octyldodecyl myristate	8.0
	Dicaprylyl ether	3.0
	Caprylic/capric triglycerides	5.0
B	Water	q.s. 100.0
	Glycerin	5.0
	Xanthan gum	0.3
C	Citric acid (10% sol.)	Adjust to pH = 5.5
	Phenoxyethanol (and) ethylhexylglycerin	0.8

Two different manufacturing processes have been used:

- Classic emulsification method: oil and water phases were weighed and heated to 85°C. Phase A was slowly added to phase B at 560 rpm while stirring, and the stirring continued for 3 min. Subsequently, homogenization was performed at 12000 rpm in a water bath at 85°C for 3 min. Stir at 400 rpm to cool down to below 50°C, and then add phase C; Finally, stir and cool to room temperature.
- High energy emulsification method: oil and water phases were weighed and heated to 85°C. Phase A was slowly added to phase B at 560 rpm while stirring, and the stirring continued for 3 min. Subsequently, the ultrasonic dispersion instrument (Scientz-CP750) was used to sonicate at 85°C, the ultrasonic power was 600W, and the ultrasonic time was 15 min (ultrasonic 5 s, interval 5 s). Stir at 400 rpm to cool down to below 50°C, and then add phase C; Finally, stir and cool to room temperature.

Microscopic observation: an optical microscope (BX-53, Olympus) was used to evaluate the microstructure of the formula before application and the formula when the white streaks were most important upon application on the skin.

Determination of particle size: particle size of the emulsion was determined using a Zetasizer Nano ZS instrument (model 90PLUS PALS, Nano Brook, Brookhaven, USA) in dynamic light scattering (DLS) mode, and three sets of parallel experiments were set up for each formula to ensure the accuracy of the data.

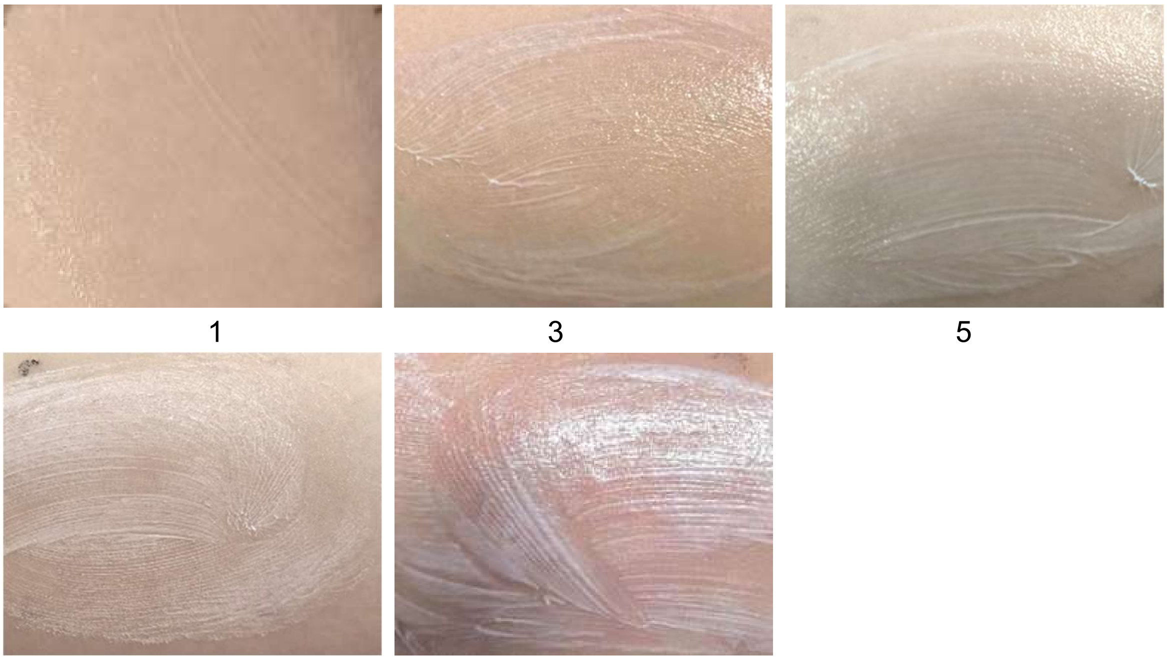
3. Results and discussion

3.1. Design of white streaks evaluation method

Preliminary work has been done to set up the proper method. The use of synthetic membranes and application device were tested but it does not simulate skin absorption and real application conditions. That's the reason why a human panel has been used.

Test have been performed to determined the ideal quantity of cream to apply, the size of the area of application, the way and speed of application, and then how to evaluate the white streaks.

Finally, to ensure an objective and accurate evaluation of the quantity of white streaks, the following method was determined: wash the inside of the arm with facial cleanser, let it stand for 30 min at constant temperature and humidity, take 0.08ml of sample in the test area formed by a square of 4cm, apply it at a constant speed of 5 cycles/s with a circular motion with the index finger to observe the formation and absorption of white streaks. When the maximum amount of white streaks is formed, a picture is taken with a mobile phone in a standard light box to ensure consistent lightning. The white streaks of the formula are scored according to the following score table from 0 with no white streak to 10 with large amount of soaping effect.



1 3 5

7 9

Figure 1. White streaks score scale

Skin type and hydration level have been also investigated. The more oily is the skin, the more soaping effect will be produced. On the other hand, there was no significant correlation between skin moisture content and the quantity of white streaks.

3.2. Effect of formula composition on white streaks

3.2.1. Emulsifiers

The microstructure and macroscopic aspect of the formulas prepared with the different emulsifiers are shown in Figure 2, and the white streak scores are summarized in Table 3.

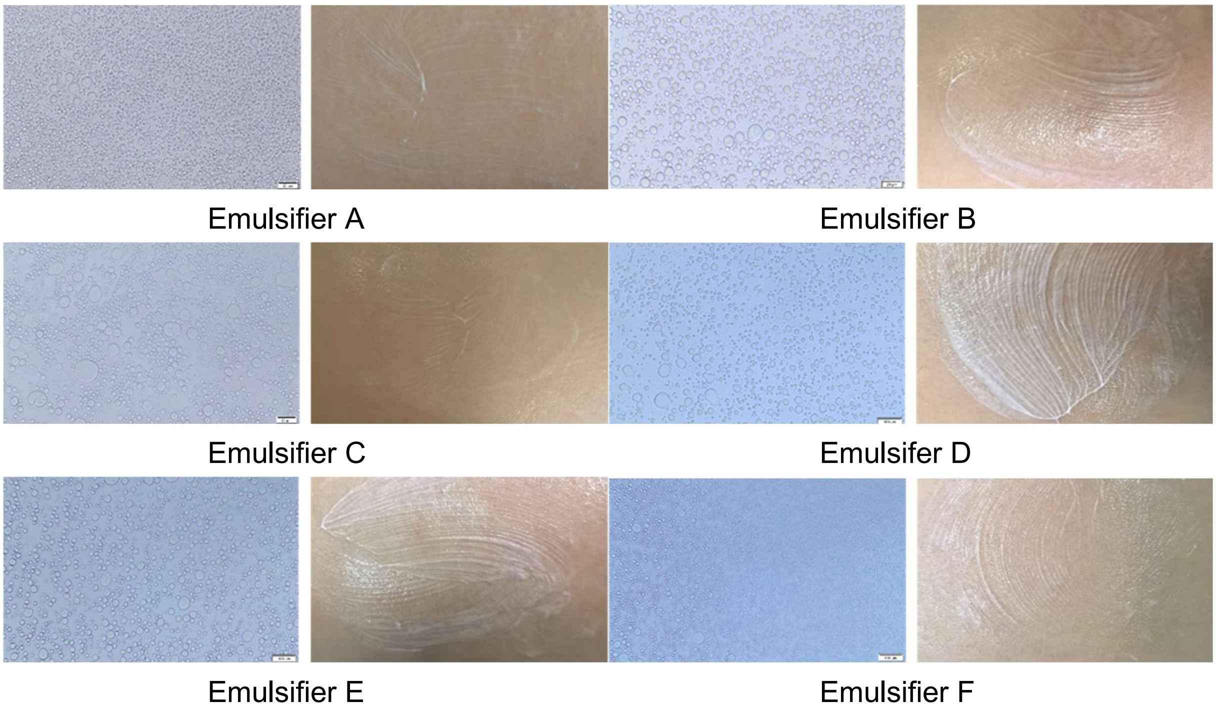


Figure 2. Microscopic and macroscopic pictures of formulas prepared with the different emulsifiers

Table 3. Summary of the white streak score of formulas prepared with the different emulsifiers

Emulsifier	INCI name	White streaks score
A	polyglyceryl-6 distearate (and) candelilla/jojoba/rice bran polyglyceryl-3 ester	2.3
B	cetearyl alcohol (and) glyceryl stearate (and) jojoba esters (and) sunflower (helianthus annuus) seed wax (and), sodium stearoyl glutamate (and) water (and) polyglycerin-3	4.5
C	polyglyceryl-6 distearate	1.6
D	cetearyl alcohol (and) cetearyl glucoside	8.2
E	cetearyl olivate (and) cetearyl glucoside	7.6
F	Polyglyceryl-3 methylglucose distearate	3.0

As can be seen from Figure 2 and Table 3, the emulsification of all formulas were good, and the white streaks were weak with Emulsifiers A, C and F. Emulsifiers B, D and E exhibit more important white streaks. By comparing the composition and structure of the raw materials, it can be seen that the main component of Emulsifiers A, C and F is a polyglycerol ester, while Emulsifiers B, D and F contain all cetearyl alcohol. Therefore, it is speculated that cetearyl alcohol may have a direct contribution to the white streaks. In the follow-up study, Emulsifier A with a relatively simple composition and a weak white streak effect was selected as the main emulsifier to investigate the effect of other components on the soaping.

3.2.2. Fatty alcohols

Fatty alcohols are one of the commonly used oily raw materials in cosmetics, and their physicochemical properties and their effects on the emulsification effect vary according to the length of the carbon chain. In the basic formula described in Table 2, cetearyl alcohol is used at 1.5% w/w. A variation in the carbon chain length and on the level of fatty alcohols have been investigated. The results are presented in Table 4.

Table 4. Summary of the white streak effect of formulas prepared with the different fatty alcohols

Type of fatty alcohols	% w/w in the formula	White streaks score
Cetearyl alcohol (C16/C18)	1.5	2.3
Cetearyl alcohol (C16/C18)	3.0	3.0
Stearyl alcohol (C18)	1.5	3.0
Behenyl alcohol (C22)	1.5	3.0

Results show that a slight increase of the soaping effect is measured when increasing the carbon chain length from C16 to C18, but then no more increase is visible with behenyl alcohol. However with the use of behenyl alcohol, we can notice a lower quality of emulsion.
An increase of the content of fatty alcohol will also increase slightly the soaping effect.

3.2.3. Emollients

The test of different emollients, both different polarity and chemistry, did not really help to rank them regarding their effect on the soaping, except silicone oils which induce a decrease of the white streaks, and vegetable oils which tend to increase the soaping.
Nevertheless, evaluation of the microscopic aspect of the emulsion show that an emollient which create a nice fine emulsion has more chance to enhance the soaping effect, than an emollient which leads to a poor emulsion structure (see Figure 3).

a/

b/

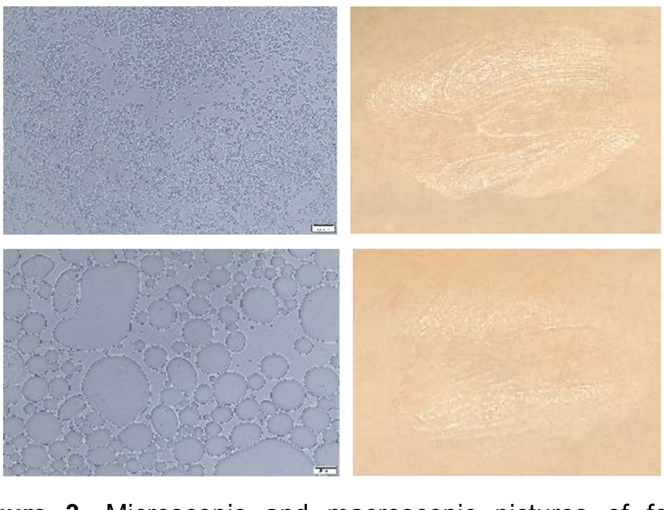


Figure 3. Microscopic and macroscopic pictures of formulas prepared with (a) caprylic/capric triglycerides and (b) butylene glycol cocoate

3.2.4. Aqueous gelling agents

Aqueous gelling agents are often used in emulsions to adjust the viscosity of samples, and their rheological properties and effects on emulsification and absorption properties vary according to their sources and structures. Four gelling agents have been tested at 0.3% w/w in the basic formula.

Table 5. Summary of the white streak effect of formulas prepared with the different aqueous gelling agents

Aqueous gelling agents	White streaks score
Xanthan gum	2.3
Carrageenan	3.2
Sclerotium gum	2.3
Carbomer	1.2

Results in Table 5 show that there are a small differences amongst the natural gelling agents regarding their effect on soaping, whereas the use of carbomer leads to lower white streaks. Most of the natural gelling agents are high molecular weight and polysaccharide substances, which can form interconnected three-dimensional network structures through intermolecular interactions such as hydrogen bonding to increase the strength of interfacial films. Compared with other thickening mechanisms, the shear thinning rate is slower, which increases shear action required to destroy the network structure and emulsion interfacial film during the application on the skin, resulting in a slower absorption rate and a lag in phase change.
Further tests have shown that increasing the amount of xanthan gum will also increase the viscosity of the formula together with the soaping effect.

3.3. Research on the formation mechanism of white streaks

The soaping effect is an optical effect, and the enhancement of the white streaks will be accompanied by the formation of new phases. The emulsion system itself has a liquid-liquid interface and a solid-liquid interface generated by solid raw materials. During application on the skin, with the occurrence of shear and absorption, water is gradually volatilized, oil is gradually absorbed or volatilized for some of them, and the proportion of solid-state substances in the system increases. At the same time, during application, we can also incorporate some air resulting in a new air-liquid/air-solid interface. With the formation of a new interface, the white streaks gradually appeared. Therefore, the formation mechanism of soaping was studied from three dimensions: the liquid-liquid interface, the solid-liquid interface, and the air-liquid/air-solid interface.

3.3.1. Liquid-liquid interface

After adding the emulsifier in the oil-water system, the emulsifier adsorbs on the interface and forms an interfacial film, which plays a role in reducing the interfacial tension. At the same time, energy is applied to the oil-water system by means of stirring and homogenization, so that the originally immiscible oil phase and water phase form a new interface, that is, the liquid-liquid interface. As the emulsion is applied on the skin with shear, the original structure of the

emulsion is destroyed, the phase state of the oil-water interface changes, and the optical effects of the system are different, resulting in potential white streaks.
In order to modify the liquid-liquid interface, tests have been performed by increasing the proportion of the internal phase in the basic formula in which only one emollient has been used, caprylic/capric triglycerides from 0 to 25% w/w. The results show a gradual increase of the soaping effect with the increase of the dispersed phase. In this case, the size of emulsion droplets amongst the trials has been kept identical.
In the following trials, the impact of the size of the emulsion droplets has been investigated. To do this the same basic emulsion has been done using two processes, one with low energy emulsification and one with high shear emulsification.

Table 6. Summary of the white streak effect of formulas prepared with different emulsification processes

Process	Particle size (nm)	White streaks score
Low energy emulsification	2500	1.3
High energy emulsification	200	2.5

As can be seen from Table 6, the amount of white streaks increases as the emulsified particle size decreases. This can be due to the fact that when high energy is added, the emulsion particle size of the emulsion is smaller, forming a huge interfacial film. This interfacial film has greater strength, stronger shear resistance, and the interface is not easily destroyed, so the white streaks increase.
A hypothesis here is that more significant the demulsification (or the worse the emulsification effect) during the application process, the less the soaping effect. This can be correlated with the result obtained with behenyl alcohol in 3.2.2. and with butylene glycol cocoate in 3.2.3, which both lead to a lower emulsion quality in our example.

3.3.2. Solid-liquid interface

In an emulsion, we can have many solid substances, such as emulsifiers, thickeners, butters, etc. During the preparation process, these solid substances are usually solubilized at high temperature in the liquid oil phase, followed by an emulsification step during which the interface is formed and gradually stabilized. After cooling, the solid substances can remain solubilized in the emulsion or partially crystallized. However, during application on the skin, absorption and potential volatility of liquid emollients can lead to more crystallization, meaning an increase of solid-liquid interface. Investigations have been conducted to evaluate the correlation with the appearance of white streaks.

Emulsifiers and thickeners with different melting points were tested and preliminary results indicate that the amount of white streaks increase with the increase of the melting point of these solid substances. The hypothesis of higher crystallization on the skin leading to more soaping still needs to be confirmed with further tests. Indeed as seen in 3.2.2 an increase of the chain length and also the melting point of the fatty alcohols shows an increase of soaping when switching from cetearyl alcohol to stearyl alcohol, but then no increase of white streaks is visible when switching to behenyl alcohol. In the case of use of cetearyl alcohol at different levels, we have seen also that when a higher content is used, a visible increase of white streaks is noticed.

3.3.2. Air-liquid/Air-solid interface

During application on the skin, air can be entrapped creating so far a new interface in the system, air/liquid and/or air/solid. This phenomenon can be emphasized in case of the use of foaming ingredients such as anionic surfactants.

Investigations have been carried out with sodium stearoyl glutamate (SSG) a common co-emulsifier. Results are shown on Figure 4.

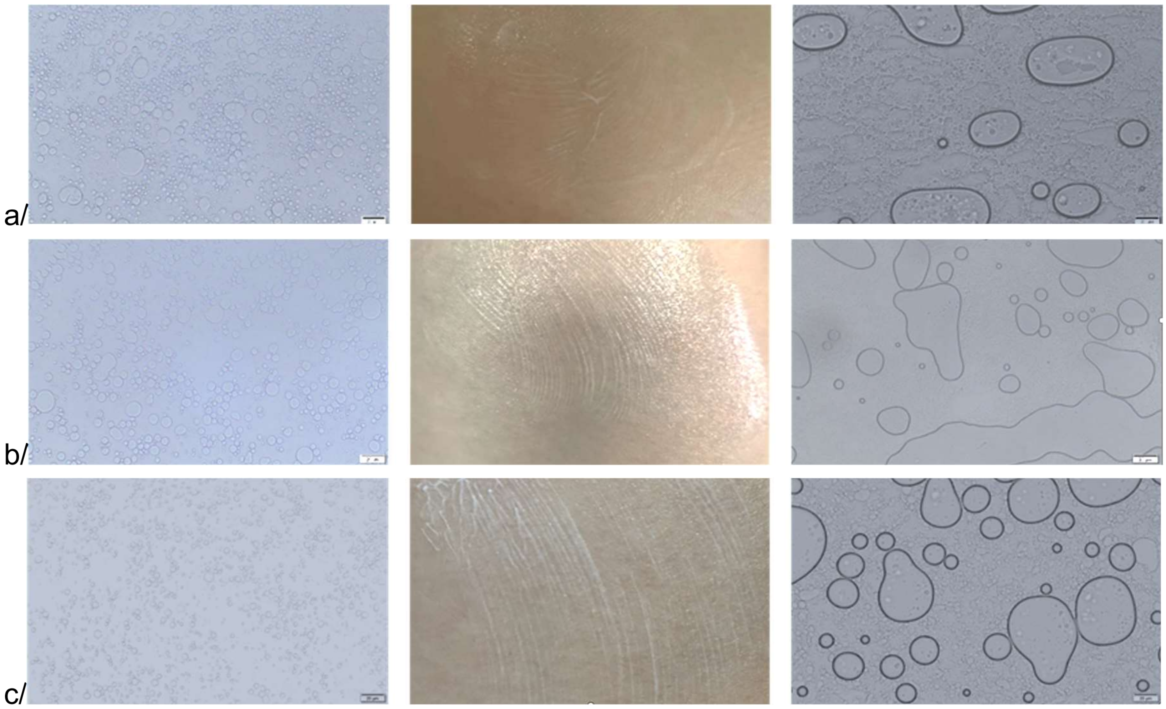


Figure 4. Microscopic and macroscopic pictures of formulas prepared with different levels of SSG (a) 0% (b) 0.5% (c) 1% w/w, and microscopic pictures of the formulas after application on the skin

From the microscopic images of the white bars (right images), it can be seen that with the increase of SSG content to 1% w/w, the air bubbles produced by application gradually increase together with an increase of the white streaks. The microscopic images of the formulas (left images) also indicate that an increased amount of SSG leads to a thinner emulsion, which is also known to produce more soaping as seen in 3.1.1.

Other trials have shown that air incorporation during application on the skin is also present in the case of non-ionic emulsifier for instance, but also that white streaks are not always linked to air incorporation.

4. Conclusion

Soaping during application on the skin is an undesirable optical effect for the consumers.

Due to the complexity of emulsion compositions, the formation of white streaks is controlled by a variety of factors, which is fundamentally caused by phase changes that happen during

application due to shear, absorption and evaporation. This provides a certain theoretical basis for the preparation and application of high-performance emulsions in the future.
An objective sensory method was designed with a precise application protocol and a visual reference scale to score accurately the white streaks.
Different factors have been evaluated to see their influence on the white streaks formation. It's clear that presence of fatty alcohols is an important parameter but is not the only one.
Further work is needed to confirm some hypotheses, however we can summarize several rules that can help the formulator to reduce or even eliminate white streaks in their formula development:

- Decrease the oil phase or at least decrease vegetable oils and increase silicone oils if allowed by their formulation charter.
- Decrease the solid lipophilic components to keep a ratio liquid/solid in the oil phase as high as possible once applied on the skin.
- Improve the demulsification during application on the skin. This can be done for instance by increasing the droplet size, using less emulsifier, decreasing the viscosity or using aqueous gelling agent with high sensibility to shear.