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Oil incorporation in water-based solid formats using a new biodegradable polymeric emulsifier

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1. Introduction

Water-based formulations are commonly used in the cosmetic industry due to their lightweight and fresh sensory. This category of products includes a significant variety of textures, from liquid to solid formats. This study will be focused on water-based solid products, that can entail from frequently used deodorants to trending formats like eye patches or makeup sticks. While providing the desired refreshing sensory, these products present the limitation of containing an oil phase, which can lead to a lack of emolliency and skin moisturizing properties, potentially leading to skin dryness and itchiness overtime. They are therefore recommended to be used in combination with a moisturizer.

Nowadays, consumers are inclined to use a small, limited number of cosmetics to improve not only skin health and efficacy but to employ as decorative products as well. The purpose of this trend is to reduce multistep routines and focus instead on minimalist ones [1]. Furthermore, consumers are paying more attention to the ingredients, searching for more sustainable solutions as an alternative to the synthetic ones [2]. For formulators, creating products with multiple benefits can help meet consumers' needs and expectations.

Water-based solid formats enable easily the incorporation of humectants due to their solubility in the main solvent. Humectants are known to supply moisture by attracting water from the environment and the skin. However, when used alone, they tend to provide a sticky sensory and increase the transepidermal water loss (TEWL). They are, therefore, recommended to be used in combination with oil ingredients [3].

The addition of an oil phase into water-based formats can provide moisturization to the skin and improve skin flexibility. The oil ingredients work as occlusive agents by blocking the evaporation and loss of moisture as well as assisting in the maintenance of the skin barrier by

reducing the exfoliation of dead skin cells. In addition, they can provide sensory benefits during application and afterfeel, reducing any possible discomfort and irritation over time [4, 5].
Simultaneously, the addition of an oil phase in water-based solid formats, facilitates the incorporation of both water-soluble and oil-soluble active ingredients. The combination of both actives is expected to further improve the efficacy of the formula.
Our solution to incorporate oil-based ingredients into water-based solid formats is the use of Undecylenoyl Xanthan Gum, a naturally derived and biodegradable polymeric emulsifier. By including Undecylenoyl Xanthan Gum in water-based solid formats it is possible to incorporate relatively high concentrations of oily ingredients, without impacting the appearance, hardness or sensory of the formulas. Additionally, a reduction of the jelling agent can be accomplished when increasing the amount of oil phase, without affecting the appearance or sensory either. Therefore, by including Undecylenoyl Xanthan Gum in water-based solid formats, it is possible to deliver multibenefit and sustainable solutions.
In this study, two different formats will be presented: a blush and lips stick and an eyepatch, jellified by Agar and Carrageenan, respectively. In both formulations, these polymers lack emulsification and suspension properties when an oil phase is included. The addition of Undecylenoyl Xanthan Gum will improve the stability of both formulas by not only maintaining the structure provided by the polymers, but also by avoiding oil release during cool down. Furthermore, no additional emulsifiers or solubilizers will be needed, reducing potential skin irritation and avoiding HLB calculations to provide emulsion stability.

2. Materials and Methods

2.1. Blush and lips jelly stick

The jelling agent used is Agar from Hispanagar (Burgos, Spain) and the polymeric emulsifier used is Undecylenoyl Xanthan Gum from Lubrizol (Brecksville, Ohio, US).
The oil phase includes Triolein, Isostearyl Isostearate and Brassica Campestris Seed Oil & Tabebuia Impetiginosa Bark Extract & Polyglyceryl-3 Diisostearate from Lubrizol (Brecksville, Ohio, US).
The dye used is CI 17200 from Sancolor (Barcelona, Spain). Other ingredients are Glycerin from CQ Masso (Barcelona, Spain), Propanediol from CovationBio PDO (Tennessee, US), Phenylpropanol & Propanediol & Caprylyl Glycol & Tocopherol from Ashland (Delaware, US), Glyceryl Caprylate and Silica from Evonik (North Rhine-Westphalia, Germany) and Glycerin & Water (Aqua) & Prunus Armeniaca Fruit Extract from Lubrizol (Brecksville, Ohio, US).

2.2. Eyepatches

The jelling agents used are Carrageenan and Ceratonia Siliqua Gum from CP Kelco (Georgia, US). The polymeric emulsifier used is: Undecylenoyl Xanthan Gum from Lubrizol (Brecksville, Ohio, US).

The oil phase is based on Triolein and Brassica Campestris Seed Oil & Tabebuia Impetiginosa Bark Extract & Polyglyceryl-3 Diisostearate from Lubrizol (Brecksville, Ohio, US).

The pigments included are Calcium Aluminum Borosilicate & Titanium Dioxide & Silica & Tin Oxide from Merck (Hesse, Germany). Other ingredients are: Propanediol from CovationBio PDO (Tennessee, US), Levulinic Acid and Pentylene Glycol & Glyceryl Caprylate from Jover Scientech (Barcelona, Spain) and Water (Aqua) & Tetrapetide -1 & Caprylyl Glycol from Lubrizol (Brecksville, Ohio, US).

2.3. Formulation and procedure: blush and lips jelly stick

The water-based jelly sticks were prepared using the formulations shown in Table 1.

The procedure followed was: Mix and disperse ingredients of phase A. Add phase B slowly while mixing. Mix for 15-20 minutes to ensure complete dispersion, avoid air incorporation. Add phase C and heat to 90-95ºC. Then, add phase D, one by one, ensuring incorporation. Premix phase E and add it to batch while maintaining the temperature at 80-85ºC until the end. Add phase F one by one and compensate the water that has been lost through evaporation to reach 100%. To finish, fill the molds while hot and let it cool down at room temperature.

Table 1. Composition of blush and lips jelly stick

		Formulas					
Phase	**Ingredients**	**1**	**2**	**3**	**4**	**5**	**6**
A	Water	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.
	CI 17200	0.30	0.30	0.30	0.30	0.30	0.30
B	Undecylenoyl Xanthan Gum	0.50	0.50	0.50	0.50	0.50	0.50
C	Agar	2.00	2.00	2.00	1.50	1.50	1.50
D	Glycerin	5.00	5.00	5.00	5.00	5.00	5.00
	Propanediol	10.00	10.00	10.00	10.00	10.00	10.00
	Phenylpropanol & Propanediol & Caprylyl Glycol & Tocopherol	0.80	0.80	0.80	0.80	0.80	0.80
	Glyceryl Caprylate	0.30	0.30	0.30	0.30	0.30	0.30
E	Triolein	8.00	13.00	18.00	18.00	-	23.00
	Isostearyl Isostearate	-	-	-	-	18.00	-
	Silica	0.50	0.50	0.50	0.50	0.50	0.50

F	Brassica Campestris Seed Oil & Tabebuia Impetiginosa Bark Extract & Polyglyceryl-3 Diisostearate	2.00	2.00	2.00	2.00	2.00	2.00
	Glycerin & Water (Aqua) & Prunus Armeniaca Fruit Extract	2.00	2.00	2.00	2.00	2.00	2.00

2.4. Formulation and procedure: eyepatch

The water-based eyepatches were prepared using the formulations shown in Table 2.

The procedure followed was: Mix ingredients of phase A. Add phase B slowly while mixing. Mix for 15-20 minutes, ensure complete dispersion and avoid air incorporation. Then while mixing, add phase C one by one. Heat to 75-80ºC and continue mixing until complete dispersion of both polymers. Add phase D, one by one. Stop the heating and continue mixing. Adjust to pH 5.0-6.0 with phase E and add phase F, consecutively, while the formula is still warm. Pour the formula into the molds while warm and let it cool down at room temperature.

Table 2. Composition of eyepatches

		Formulas	
Phase	**Ingredients**	**7**	**8**
A	Water	To 100	To 100
	Propanediol	5.00	5.00
	Pentylene Glycol & Glyceryl Caprylate	3.15	3.15
	Levulinic Acid	0.30	0.30
B	Undecylenoyl Xanthan Gum	0.20	0.20
C	Ceratonia Siliqua Gum	0.30	0.30
	Carrageenan	0.60	0.60
D	Triolein	2.00	3.00
	Calcium Aluminum Borosilicate & Titanium Dioxide & Silica & Tin Oxide	0.10	0.10
E	Sodium Hydroxide 20%	0.20	0.20
F	Water (Aqua) & Tetrapetide -1 & Caprylyl Glycol	2.00	2.00
	Brassica Campestris Seed Oil & Tabebuia Impetiginosa Bark Extract & Polyglyceryl-3 Diisostearate	-	2.00

2.5. Oil phase stabilization

All formulas were done with and without Undecylenoyl Xanthan Gum to demonstrate its effectiveness in oil stabilization.

2.6. Appearance and structure

Appearance was compared between formulas to determine the appropriate ratio of oil phase needed to obtain homogeneous formulas.

To spot differences on the structure, hardness was measured for the blush and lips jelly stick by using a Texture Analyser equipment (TA.XT Plus, Stable Micro Systems, UK). The test was performed using a 2mm diameter cylinder probe to puncture the sticks, determining the higher force needed for penetrating 5mm of the formula. The higher the force value obtained, the harder the sample will be [6]. Results were obtained for two replicates of each formulation.

3. Results

3.1. Blush and lips jelly stick

Formulas 1 and 2 from Table 1 were reproduced in the laboratory following the procedure described. The resulting sticks were homogeneous, and the desired appearance and texture was obtained. In both formulations, Agar was incorporated at 2% to deliver the jelly structure, while the oil phase, stabilized with Undecylenoyl Xanthan Gum, was included at 10% and 15%, respectively (Figure 1).

Figure 1. Left to right: Formula 1, Formula 2

The most significant difference detected between formulations 1 and 2 was during emulsification. Due to the higher proportion of oil phase included in formula 2, an increase in viscosity was easily perceived. However, no further complexity was added during the procedure. Alternatively, depending on the packaging used, particularly if it is too small, a hampering of the filling process by obstruction or jellification beforehand could occur.

Subsequently with formula 3, when 20% of oil phase was used, a further increase in viscosity could be observed during emulsification process. In this situation, the viscosity change did not have an impact on the procedure either. Nevertheless, the packaging filling was more challenging. This resulted in a less homogeneous and a rougher appearance than the obtained in the previous formulas.

To decrease viscosity during emulsification process and improve the appearance of the stick, a reduction of 0.5% in the use level of Agar was done, obtaining formula 4. Unfortunately, no variation on the viscosity was perceived, and a similar appearance to formula 3 was obtained

(Figure 2). Yet, by checking the appearance on a 100ml glass jar, no difference between samples was detected, implying that the final aspect will substantially depend on the packaging used. The stick used in this study has a 6 ml capacity. Presumably, by using a stick packaging of bigger size, a more homogeneous appearance could be obtained.

Figure 2. Left to right: Formula 1, Formula 3, Formula 4.

When a further increase of 5% of Triolein was applied, rising the oil phase to 25% in formula 6, similar viscosity during emulsification was achieved. However, a slight oil release was observed during cool down and although it was possible to obtain a stick, higher exudation was easily detected (Figure 3). This determined that 20% is the maximum amount of oil phase that Undecylenoyl Xanthan Gum can stabilize in this water-based jelly stick.

Figure 3. Formula 6

With the maximum amount of oil phase established, a change of the main emollient was performed, substituting 18% of Triolein for 18% of Isostearyl Isostearate, while maintaining Agar at 1.5%. A better emulsification was perceived during the procedure when using Isostearyl Isostearate, obtaining a stick with a more homogeneous appearance (Figure 4). The viscosity during emulsification was similarly high as the formula with Triolein.

Figure 4. Left to right: Formula 4, Formula 5

When the same formulations were done excluding Undecylenoyl Xanthan Gum, an oil release was easily perceived during cool down. This led to non-stable formulas from the start, determining the need to incorporate Undecylenoyl Xanthan Gum for stabilization (Figure 5).

(a) (b)

Figure 5. Formula without Undecylenoyl Xanthan Gum and dye. (a) Formula 1 (b) Formula 4

To further evaluate differences between the sticks obtained, hardness values were measured using Texture Analyser equipment. Results are captured in Figure 6.

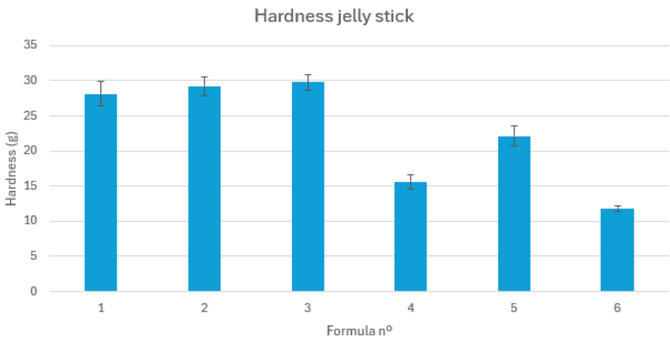

Figure 6. Hardness results (formulas 1-6)

From this analysis it was detected a direct influence of Agar's concentration on the hardness of the sticks. By comparing formula 3 with formula 4, a significant impact on the hardness was noticed on the latter when Agar was reduced from 2% to 1.5%. Regardless, this reduction in hardness did not impact the jelly appearance nor the application of the formula on the skin.

Moreover, it was identified that the emollient used in the oil phase could also influence the hardness of the stick. When comparing formulas 4 and 5 with 18% of Triolein and Isostearyl Isostearate, respectively, an increase in hardness of 41.7% was observed when including the latter. By providing better emulsification, Isostearyl Isostearate was not only capable of providing a more homogeneous appearance but also harder stick, without effecting the jelly texture. Additionally, the concentration of emollient did not appear to have a significant impact on the hardness of the sticks. When analyzing the first three formulations, no substantial differences in hardness were observed. However, if an excessive amount of emollient was added, as in formula 6, where oil release was observed, a considerable impact on the hardness could be measured, decreasing it nearly a 25% when compared to formula 4.

3.2. Eyepatches

Eyepatches were studied to demonstrate the effect of Undecylenoyl Xanthan Gum at a lower use level in a different water-based solid chassis. Formulations 7 and 8 from Table 2 were prepared following the procedure from section 2.

Formula 7 was developed by incorporating only 2% of Triolein as oil-based ingredient, obtaining a homogeneous eyepatch. No oil release was detected (Figure 7).

Figure 7. Formula 7

In formula 8, Triolein was increased to 3% and an oil-based active ingredient was added at 2%. This increase in the oil phase did not have impact on the procedure or the appearance of the final product, resulting in a similar eyepatch to formula 7.

As identified in the stick, if Undecylenoyl Xanthan Gum was not incorporated, an oil release was easily perceived during cool down, leading to a non-stable formula from the initial point (Figure 8).

(a) (b)

Figure 8. Eyepatches without Undecylenoyl Xanthan Gum. (a) Formula 7 (b) Formula 8

4. Discussion

Undecylenoyl Xanthan Gum is a naturally derived and biodegradable polymeric emulsifier that provides stabilization in emulsions by not only thickening the water phase but also through surface activity due to its hydrophobic modification. In this study, Undecylenoyl Xanthan Gum is used to stabilize oil-based ingredients in water-based solid formulations in combination with other structuring rheology modifiers capable of providing jellification. In these systems, the absence of Undecylenoyl Xanthan Gum results into an oil release.

It is commonly known that the use of oil-based ingredients can offer multiple benefits to the skin. The emollient incorporated in this study consists predominantly of Triolein, a microbiome friendly ingredient, used due to its efficacy studies and skin moisturizing benefits. In previous research, it was demonstrated that by adding Triolein on the forearm of 14 volunteers, an improvement of skin moisturization by 15% and a reduction of TEWL by 28% in 24h could be achieved. Furthermore, by combining water-based and oil-based active ingredients an improvement in efficacy could also be accomplished. The oil-based active ingredient used in this study consists in the mixture of Brassica Campestris Seed Oil & Tabebuia Impetiginosa Bark Extract & Polyglyceryl-3 Diisostearate. In previous in vivo analysis it was determined that this material could improve the appearance of wrinkles, skin radiance and skin moisturization.

By including both Triolein and the oil-based active ingredient on the jelly stick, a homogeneous appearance could be achieved when the oil phase represented a sum of 10% to 15%. However, an increase in viscosity during emulsification was perceived when the oil phase exceeded 10%. Furthermore, the aspect was impacted when the concentration reached 20%. In simple skin care chassis, it has been studied that by using 0.7% of Undecylenoyl Xanthan Gum and 20% of Triolein viscosities around 4000 mPa·s could be obtained. By including not only Undecylenoyl Xanthan Gum but Agar as well, relatively viscous samples could be anticipated, specifically when an internal phase increase occurred.

Regardless, if a higher amount of emollient concentration is desired, a change in oil phase would be recommended. For instance, at 20% oil-phase use level, the replacement of Triolein for Isostearyl Isostearate could lead to a better appearance. As exhibited, the variation of emollients will have an influence not only on the homogeneity of the stick but its hardness as well. Additionally, the hardness can be affected by the jellifying ingredient and its concentration. The selection of the appropriate emollient, jellifying agent and their respective concentrations, together with the packaging, will determine whether the formula presents the appropriate homogeneity, hardness and appearance or not.

Subsequently, when a reformulation was done, obtaining the eyepatch, successful stabilization was also achieved. By including Undecylenoyl Xanthan Gum into the eyepatch, it was not only possible to corroborate the results obtained with the jelly stick, but also to replicate them in a different chassis. Therefore, it can be established that the stabilization provided by the polymeric emulsifier is independent of the jellifying ingredient or the formula.

5. Conclusion

The aim of the study was to demonstrate the incorporation of oil-based ingredients into water-based solid formulations by adding Undecylenoyl Xanthan Gum as emulsifying and stabilizing agent. Based on the results reported, the effectiveness of incorporating Undecylenoyl Xanthan Gum into these formats was established, while if excluded, oil release occurred before jellification. Therefore, by utilizing this polymeric emulsifier it was possible to not only stabilize up to 20% of oil-based ingredients without significantly impacting the appearance, structure or application of the formulas on the skin but to potentially improve the efficacy as well, by including both water and oil-based active ingredients.

6. References

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