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Effect of Incorporating a Biowax Derived from Hydroprocessing of Crude Palm Oil in a Facial Cream and a Blemish Balm Cream

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Abstract: Most waxes used as cosmetic ingredients are derived from the petrochemical industry. A modern alternative to this complex synthesis approach is the hydrotreatment of palm oil; thus, the aim of this study is to evaluate the effect of incorporating a biowax derived from hydroprocessing of crude palm oil as a new natural cosmetic ingredient in facial cream and BB cream. Therefore, two water in oil (W/O) emulsions, one including pigments, with five different weight percentages were developed and subjected to further sensory evaluation by a trained panel to estimate the level of acceptance. Moreover, resistance to centrifugation, pH, spreadability, phase separation, viscosity and storage modulus were the parameters evaluated in a preliminary stability study using thermal stress. Sensory analysis showed that the highest level of acceptance was obtained between 3% and 9 wt% biowax. For both prototypes, increasing biowax percentage led to a greater effect on stickiness, the viscosity increased, and extensibility decreased. The formulations were able to maintain their pH. The best stability for BB cream was observed at 9%, since the changes in the properties were slight. For facial cream, the emulsion was more stable at intermediate biowax content. It was observed that biowax exhibits favorable characteristics as an emollient or thickening agent. Finally, the formulations with the best stability and sensory characteristics were obtained at 9 wt% biowax.

Keywords: biowax; preliminary stability; sensory profile; level of acceptance

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1. Introduction

Currently, the global market shows a growing preference for natural cosmetics, with an estimated annual growth rate of 15%, compared to 5% for traditional cosmetics [1]. In fact, the skincare market is expected to contribute significantly to the growth of the industry, followed by hair care and, in third place, color makeup, such as blemish balm cream (BB cream), has great potential due to the protection it provides to the skin [1].

In Colombia, the cosmetics sector has had an annual growth rate of 8.7% [2], higher than Latin America, which has made it one of the most important producers and the one with the best projection. Furthermore, Colombia is one of the countries with the richest soil and a wide variety of climates, which favor the production of natural ingredients. Despite this, Asociación Nacional de Industriales de Colombia (ANDI) shows most raw materials are imported (85%) [3], and many of them are derived from palm oil of the *Elaeis Guineensis* tree. Therefore, ANDI emphasizes that if Colombia's palm oil sector ventures into the production of these raw materials, mainly emulsifiers and emollients, it would increase the country's competitiveness considerably [3].

Palm oil production increased by 8.2% in the last year, from 1.6 to 1.7 million tons [4]. Likewise, 27% of the crude palm oil produced in the country has certified sustainability, which is a differential factor and an added value due to the palm oil industry having a high environmental impact [4].

In addition, the availability of FDA-grade wax for the cosmetics industry is limited to imports from China, Brazil, Argentina and Germany. However, there is an opportunity to produce it from sources available in the national oleochemical industry of Colombia as palm oil is composed of triglycerides, combinations of glycerol and different fatty acids, which makes it a sensorially interesting cosmetic ingredient [5].

Waxes are commonly used as lubricants, adhesives, foodstuffs, pharmaceuticals and cosmetics. Seventy percent of waxes are made from petroleum; nevertheless, the depletion of reserves, fluctuating crude oil prices and sustainability concerns have led to the search for new alternatives. Thus, vegetable oils (VOs) represent a promising source for producing renewable and environmentally friendly wax alternatives [6].

Ecopetrol has been developing a technology that involves FDA-grade wax production from the hydroprocessing of crude palm oil [7,8], known as biowax. This technology has desirable characteristics for the cosmetics industry, especially as an emollient and thickening agent. biowax was obtained following the methodology described in Guzman et al., 2010 and 2013 [9,10], modifying the reaction conditions and catalysts used according to Olarte et al., 2023 [8], as follows: Biowax was obtained in a pilot-scale fixed-bed reactor using a nickel molybdenum catalyst ($NiMo/Al_2O_3$), with temperatures ranging from 240 to 260 °C, pressures between 800 and 1300 psig, and liquid hourly space velocity (LHSV) of 1 to 2 h-1. According to stability studies carried out by Laura Chaparro et al. 2023 [11], this biowax (BPW7) consists of a mixture of fatty esters (24.80%), paraffin (29.60%), triglycerides (TGs, 22.00%), diglycerides (DGs, 1.7%), fatty acids (FFAs, 14.30%) and fatty alcohols (7.60%). In addition, biowax has physicochemical characteristics that make it attractive for use in the cosmetics industry [12].

Especially in cosmetics, waxes have been included in the formulation of many products, one of which is facial creams and BB creams. Both products are emulsified systems, typically in color cosmetics, such as water in silicone (w/si); however, silicones have been rejected due to their environmental impact and have been replaced by oils with similar sensory characteristics [13]. However, switching from silicone to oils brings with it the challenge of achieving stable emulsions with sensory profiles acceptable to the consumer; thus, it is important to include a stability prediction and evaluation of sensory parameters.

Sensory analysis is an important parameter for the cosmetic industries, as sensory evaluation data are used in marketing decisions and have shown to be important in the development of cosmetic products designed to delight the consumer's senses [14]. Another important parameter is the study of accelerated stability studies using thermal stress, which is a good tool for inducing emulsion alterations [15]. Some physicochemical parameters that help to predict stability are viscosity, storage modulus, pH and phase separation.

Despite the above, few studies have been found in which the stability and sensorial performance of facial creams using waxes of natural origin have been evaluated. And, consequently, biowax has not been produced at a pilot scale in Colombia. Therefore, the aim of this study is to evaluate the effect of incorporating a biowax derived from hydroprocessing of crude palm oil, as a new cosmetic ingredient of natural origin, in facial cream and BB cream.

2. Materials and Methods

2.1. Preparation of Formulations

The preparation of the cream began by melting the biowax (3, 6, 9, 12, or 15%) with Plantsil (phase B) at 60 °C, until a single phase is formed; subsequently, the Emulium Illustro (emulsifier), Cetiol 5C, Plantsil (emollients), silicone and, in the case of the BB cream, the pigments, (Phase A) were added to this container; this oily phase was homogenized using an Ultra Turrax (IKA T25 digital) at 3000 rpm for 5 min. Then, the aqueous phase (Phase C) consisting of water, salts and the chelating agent was slowly added to the container and mixed at 3000 rpm for 10 min using an Ultra Turrax and left to cool to 40 °C. Finally, additives such as tocopheryl acetate (antioxidant), Patch H_2O (wetting active), preservative, silica, fragrance and pH stabilizer (citric acid), if necessary, were added below 40 °C (See Table 1). This procedure was carried out for both W/O emulsion formulations (BB cream and facial cream) at 5 different biowax weight percentages (3, 6, 9, 12, and 15%).

Table 1. General formulation, raw materials distributors, and functions.

Phase	Raw Material	Company	% W/W [a]	Function
A	Emulium Illustro	GATTEFOSSÉ	5.239%	Emulsifier
	Natura-Tec Plantsil	NAURA TEC	13.388%	Emollient
	Mirasil C-DML	IMCD	3.492%	Silicone
	Cetiol 5C	BASF Care Creations	5.239%	Emollient
B	Natura-Tec Plantsil	NAURA TEC	5.821%	Emollient
	BIOWAX	--	0% [b]	Biowax
C	Water	Type 1	56.809%	Water
	Sodium chloride	NATIVUS S.A.S.	1.048%	Stabilizer
	Magnesium sulphate	NATIVUS S.A.S.	1.048%	Stabilizer
	Disodium EDTA	NATIVUS S.A.S.	0.117%	Chelating agent
D	Euxyl PE 9010	ASHLAND	1.048%	Preservative
	MSS-5003H	KOBO	3.143%	Powder
	Tocopheryl Acetate	NATIVUS S.A.S.	0.116%	Antioxidant
	Patch H_2O	BASF Care Creations	3.143%	Active ingredient
	Aqua Fragrance	--	0.349%	Fragrance
	TOTAL		100%	

[a]: All the components were normalized in each sample in function of biowax percentage; [b]: the biowax was added at 5 different weight percentages (3, 6, 9, 12, and 15%) for BB cream and facial cream.

2.2. Sensory Analysis

The sensory analysis of the developed prototypes was performed by a sensory panel composed of 19 members, aged between 18 and 25 years. Each member was previously trained according to ASTM E1490-19 [16]. During training, they were introduced to cosmetics and to the general concept of the study, followed by a detailed explanation of the sensory panel and the test that comprises it. Raw materials defined by the standard as sensory extremes for each property to be evaluated were used; these properties are presented in Table 2. The training began with 30 people, and, as indicated in the standard, a test was performed on each of the trainees, which consisted of a sensory evaluation of creams and raw materials. Those who answered correctly were considered suitable for the sensory panel and went on to evaluate the prototypes.

Table 2. Sensory properties evaluated in the prototypes.

Property	Maximum Value
Stickiness	The force required to separate fingertips is minimal
Spreadability	Product spreads very easily on the skin
Absorbency	The number of rubs at which the product loses its wet, moist feel and the resistance perceived is minimal
Gloss	The amount of reflected light from the product is minimal
Softness	The degree to which the skin feels soft is maximum
Coverage	The coverage of color imperfections (freckles, facial spots) is maximum
Uniformity	Maximum product continuity on the skin
Adherence	The product is perfectly adhered to the area of application
Amount of residue	The amount of product remaining on the skin is minimal

The evaluation of each of the prototypes followed the methodology of Renata Moschini Daudt [15] and the standard used during the training. The panelists performed a process of washing the area before each application of the sample, applied an equal amount of product on the forearm for each test and, finally, gave a score from 0 to 10 for each aspect evaluated, with 10 being the score with the highest level of acceptance.

2.3. Stability Tests

When the formulations reached room temperature, they were packaged in transparent glass containers of 10 mL for organoleptic tests and 30 mL for physicochemical tests, which were taken to the stability test by thermal stress. The samples were subjected to 6 cycles of cooling and heating. Each cycle consisted of 24 h in an oven at 40 °C, followed by 24 h in the refrigerator at 4 °C [15]. The parameters analyzed for each sample before and after the cycles were resistance to centrifugation, pH, spreadability, phase separation, viscosity curve and storage modulus.

2.4. Resistance to Centrifugation

The resistance to centrifugation test was performed in a centrifuge (SL8R THERMO SCIENTIFIC) using 10 mL samples (3000 rpm, for 30 min, a 20 °C) [15]. At the end of the test, if there was a separation of the cream at the bottom or the top, this value was measured for subsequent analysis.

2.5. pH

The pH values were measured at room temperature directly in the formulation using a previously calibrated digital pH meter (SI Analytics HandyLab 100).

2.6. Spreadability

The spreadability was evaluated by depositing 0.1 g of sample in the center of a flat glass dish. This container was positioned on a sheet of graph paper with the axis in the center of the sample, then a flat glass dish (45.4 g) was placed on the sample. After 1 min, the surface covered was measured with the diameter at 8 different points. The spreadability was calculated as the area covered by the sample.

2.7. Rheological Characterization

The rheological parameters measured were viscosity curves and storage modulus. The measurements were carried out in a rheometer (Anton Paar MCR72) using parallel plates geometry (50 mm diameter, gap of 1.5 mm). All measurements were performed at 25 °C.

Viscosity curves were obtained using a rotational test, recording shear stress values when shearing the samples at increasing shear rates from 0.01 to 100 s^{-1}. The storage modulus and loss modulus were measured with an oscillatory test at a constant frequency (1 Hz), and the strain was swept from 0.001% to 10%.

2.8. Phase Separation

The observed separation of the creams at the top and bottom of the 10 mL vial was measured before and after the heat stress cycles; the cream was left to stand for the entire duration.

2.9. Statistical Analysis

To determine whether biowax percentage affects the parameters studied above, an analysis of variance (ANOVA) and Tukey HSD test were performed for the result of the sensory panel; for the stability study, a multifactorial analysis of variance was applied. The software used for these studies was Statistica™ V. 14.

2.10. Decision Matrix

For the selection of biowax percentage with the best sensory and physicochemical properties, a decision matrix was employed according to the methodology used by Medina Godoy and Diana Gabriela [17]. For this purpose, with the support of a group of experts, the weights for each evaluated aspect were defined.

The score (St) obtained by each aspect is given by Equation (1).

$$S_t = S * V \quad (1)$$

where S is the weight assigned to the criterion, being negative if its desirability is minimum and positive if it is maximum, and V is the value obtained for the property being weighted.

The total obtained for each biowax percentage is given by Equation (2). The best score was compared with a commercial product (moisturizing cream and BB cream Natura Faces)

$$T_i = \sum S_{ti} \quad (2)$$

3. Results and Discussion

3.1. Sensory Analysis

The skin sensory performance of personal care products is an important factor for the sales potential of any cosmetic product [18]. The *p*-value statistics of analysis of variance for every sensory characteristic studied in both formulations are shown in Table 3.

Table 3. Table ANOVA, *p*-value for facial cream and BB cream with respect to the amount of biowax. (p-value < 0.05 is statistically significant).

Property	Facial Cream	BB Cream
Spreadability	0.0001	0.0581
Absorbency	0.172	0.1237
Softness	0.0705	0.0149
Gloss	0.4805	0.1971
Stickiness	0.0018	0.0000
Amount of residue	0.0631	0.0000
Coverage	N.A.*	0.0870
Uniformity	N.A.*	0.1509
Adherence	N.A.*	0.0521

N.A.*: Not applicable.

The *p*-value presented in Table 3 reveals whether biowax percentage affects the parameters studied above; if it is less than 0.05, there is a statistically significant difference between the means. Thus, regarding facial cream, it is observed that biowax percentage influences stickiness and spreadability, whereas, for BB cream, biowax percentage affects the parameters softness, stickiness and amount of residue. The differences in the statistically significant properties are attributed to the fact that the pigments increase the viscosity of the cream, making it less extensible in all its percentages of biowax; in addition, due to the layer that leaves, the BB cream on the skin increases the amount of residue and the sticky sensation of the cream when absorbed. As a result, biowax percentage is not statistically significant for gloss, absorbency or aspects related to color.

As a verifying factor, Tukey's test was performed for the variables evaluated, and the results are shown in Tables 4 and 5, which confirms that there are statistical differences for both formulations in the aspects mentioned between the means of the biowax percentages of 3% and 15%.

Increasing biowax percentage also increases stickiness and the amount of cream residue on the skin but decreases spreadability and softness (Figure 1), which demonstrates that the biowax significantly influenced in the formulations, agreeing with previous studies [19] that demonstrated that the constituents of the oil phase affected the sensory aspects of emulsions. Figure 1 has a division at 9%; this is because at lower percentages there is a better acceptance, since the cream is perceived as softer and more spreadable and less viscous and sticky and leaves less residue.

Table 4. Tukey's test for stickiness and spreadability for significant different factors for facial cream (if the studied parameter has the same "X" or "XX", its average is similar).

%Biowax	Stickiness		Spreadability	
	Average	Homogeneous Groups	Media	Homogeneous Groups
3%	1.47	X	8.42	X
6%	1.84	XX	7.74	XX
9%	2.26	XX	6.89	XX
12%	2.74	XX	7.26	XX
15%	2.95	X	6.26	X

Table 5. Tukey's test for stickiness and spreadability for significant different factors for BB cream. (if the studied parameter has the same "X" or "XX", its average is similar).

%Biowax	Softness		Stickiness		Amount of residue	
	Average	Homogeneous Groups	Average	Homogeneous Groups	Average	Homogeneous Groups
3%	7.11	X	1.41	X	2.41	X
6%	7.17	X	3.05	X	2.29	X
9%	6.41	XX	3.47	XX	2.23	X
12%	5.35	X	3.58	X	4.35	X
15%	5.88	XX	4.64	X	5.17	X

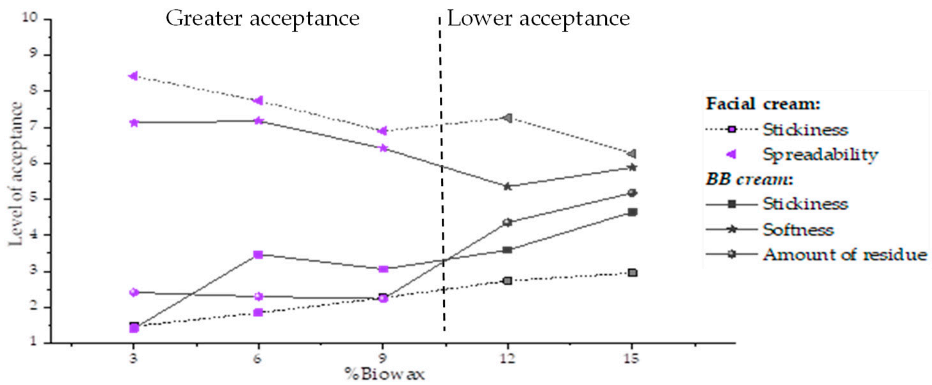


Figure 1. Statistically significant properties with respect to biowax percentage for the facial cream and the BB cream.

Hence, it is possible to affirm that biowax derived from the hydroprocessing of crude palm oil has important effects on the cosmetics formulation developed and could be considered as an emollient, as it is closely related to the sensory properties of emulsions and its performance on the skin [20]. Moreover, emollients have a major impact on physico-chemical properties of cosmetic emulsions such as consistency and spreadability, properties that are important to achieve adequate efficacy and user acceptance of the products [21]. Particularly in cosmetics, waxes are formulated in numerous personal care products due to their excellent emollient behavior [22], which is consistent with the results of this study. Regarding the sensory characteristics desired for these cosmetic prototypes, the highest level of pleasure is obtained by incorporating biowax between 3 and 9% (Figure 1), as it is perceived as an easy-to-spread, smooth cream with little residue attributes that are of great importance for its application on areas of the skin, especially on the face.

3.2. Resistance to Centrifugation

After performing the test, the length of each of the differentiated phases in each of the prototypes was measured; the results after stability are shown in Table 6. It is important to note that before thermal stress, prototypes showed no phase separation. As shown in

Table 6 and Figure 2, for facial cream, the highest clarification of the oily phase (top) was presented for concentrations lower than 6%. On the other hand, at concentrations higher than 12%, in addition to the separation of the oily phase, coalescence of water free (bottom) was observed (Figure 2).

In the case of BB cream, a higher phase separation was observed at low biowax values; above 9%, there was no perceptible difference in phase separation. This result can be attributed to the amount of biowax, as mentioned by Norton and Norton [23], because in a W/O emulsion the addition of waxes provides greater connections in the continuous phase of the emulsion, increasing the resistance to flow of the aqueous phase. The difference in the result for the face cream and the BB cream is attributed to the increase in viscosity that occurs when pigments are incorporated into the formulation, and a higher viscosity also increases the resistance to flow of the dispersed phase.

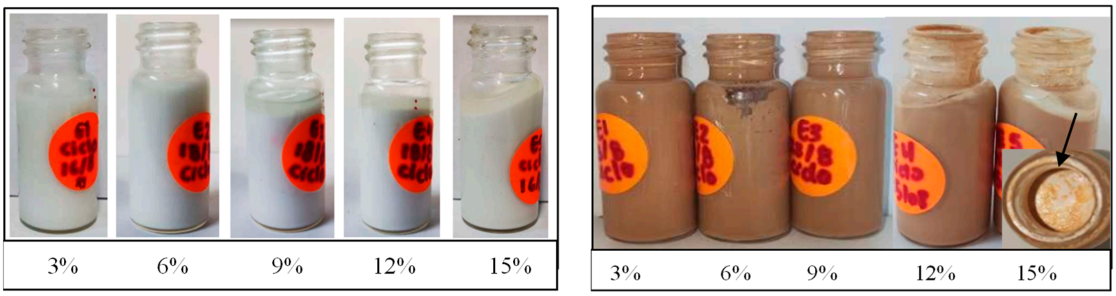
Table 6. Average pH values, centrifugation and phase separation after stability.

% of Biowax	Cream Type	Resistance to Centrifugation (mm)	pH		Phase Separation (mm)
			Initial	Final	
3%	Facial cream	18	5.22	5.81	1
	BB cream	16	5.11	4.20	0
6%	Facial cream	14	5.29	5.73	0
	BB cream	7	5.30	4,35	0
9%	Facial cream	9	5.21	5.88	3
	BB cream	48	5.29	4.30	0
12%	Facial cream	27	5.26	5.76	3.5
	BB cream	48	5.21	4.30	1
15%	Facial cream	17	5.24	5.55	4
	BB cream	28	5.34	4.40	3

Figure 2. Facial cream subjected to centrifugal test after accelerated stability.

3.3. Phase Separation

The phase separation values were recorded and presented in Table 6. It is observed that at concentrations higher than 12% of biowax, the BB cream presents a mostly white supernatant, which has the texture and consistency of biowax mixed with the oily phase. In the case of the facial cream, this supernatant is opaque white, but sensorially it has the same characteristics as the other supernatant. Figure 3a,b show the results.



(**a**) (**b**)

Figure 3. Emulsions after preliminary stability: (**a**) facial cream, (**b**) BB cream.

As has been demonstrated in some studies, a higher viscosity value allows the formulation to remain stable for a longer time because the sedimentation of droplets is slower, and, in some systems, it is also the liquid film drainage; however, the presence of the supernatant indicates that there is a maximum concentration at which the biowax, during the cooling and heating cycles, shows a substantial separation from the emulsion and is located on the surface of the cream. This behavior is due to the crystallization of the biowax as explained by Supratim Ghosh and Dérick Rousseau [24] due to the expansion and compression cycles of the wax crystals, contributing to the coalescence of the dispersed phase.

3.4. pH

The pH of the formulations was measured directly in the creams before and after stability. The results are presented in Table 6, from which it is concluded that the creams are able to maintain their pH because the values obtained for the formulations are located within the range established for a facial cream and a BB cream, 4.2 to 6.5 [25].

The pH for the facial cream after preliminary stability increased as shown in Table 6, which is consistent with the results presented in previous research on stability [25]. On the other hand, the pH of the BB cream decreased (Table 6) after the test. This is a behavior presented in other works [26]. This difference in pH response is attributed to the presence of the pigments either due to the possible oxidation or the coating they present, since it is the only variation between the two formulations developed.

3.5. Spreadability

Spreadability of all the formulations was evaluated, and for both prototypes a similar behavior was obtained, showing a dependence between the amount of biowax and the spreadability value, which means a high concentration makes the formulation less spreadable. This same behavior is shown by commercial waxes whose function in cosmetic creams is to act as emollients and rheological modifiers, such as carnauba wax or castor wax.

This result is congruent with the spreadability determined through the sensory analysis performed by the trained panelists. On the other hand, the facial cream spreads 1.2 times more on the skin than the BB cream; this result indicates a greater distribution of the emulsion per application area, which is desirable for this prototype.

3.6. Rheology

All tests showed a non-Newtonian and viscoplastic behavior, as shown in Figures 2 and 3, a desirable result in this type of formulations because the cream shows shear thinning, for example, when applied on the skin, being a desirable rheological property in cosmetic formulations because it improves the application and dispersion, providing a pleasant sensation [19]. Viscoplasticity is also a desired behavior since provides cosmetic products with consistency and stability.

Additionally, the results obtained in the mentioned studies on the relationship between a high wax concentration and the high viscosity values of the formulation are corroborated, as shown in Figure 4a,b, confirming the action of this raw material as a thickening agent.

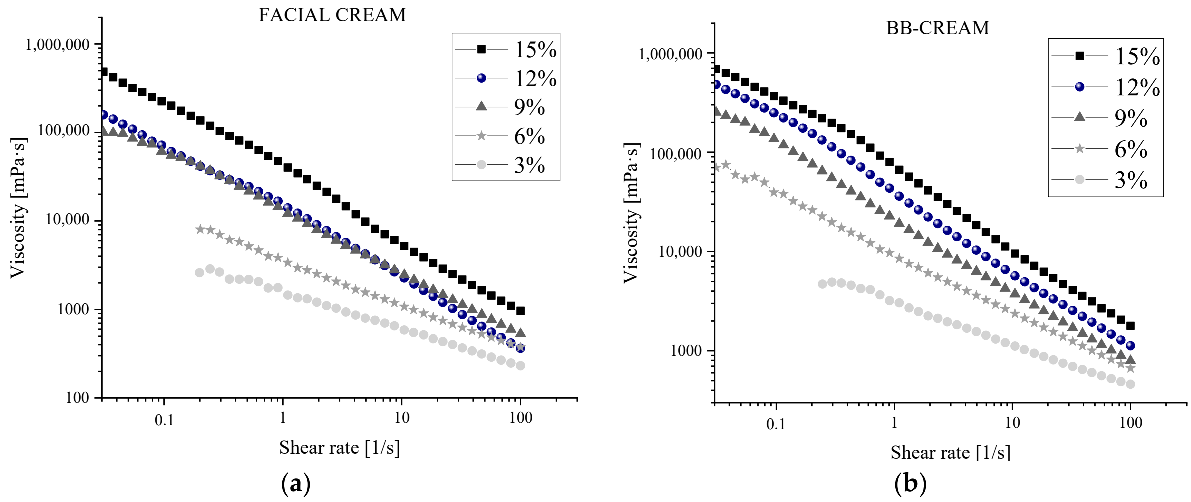


Figure 4. (**a**) Viscosity profile for BB cream after preliminary stability. (**b**) Viscosity profile for the facial cream after preliminary stability.

With respect to the viscoelastic modulus, it is observed that all formulations exhibit an elastic modulus (G′) higher than the viscous modulus (G″), as shown in Figure 5a,b. This is a key parameter of stable emulsions since it can be considered as a viscoplastic fluid. In other words, this fact indicates the viscoelastic character of the emulsions. As G′ > G″, the sample shows a solid-like structure and can be termed as a viscoelastic solid material. Likewise, after subjecting the formulations to thermal stress, the change in the modulus G′ for each prototype is not significant, as in the case of Moschini et al. [15], which means that the prototypes present stability in their rheological behavior.

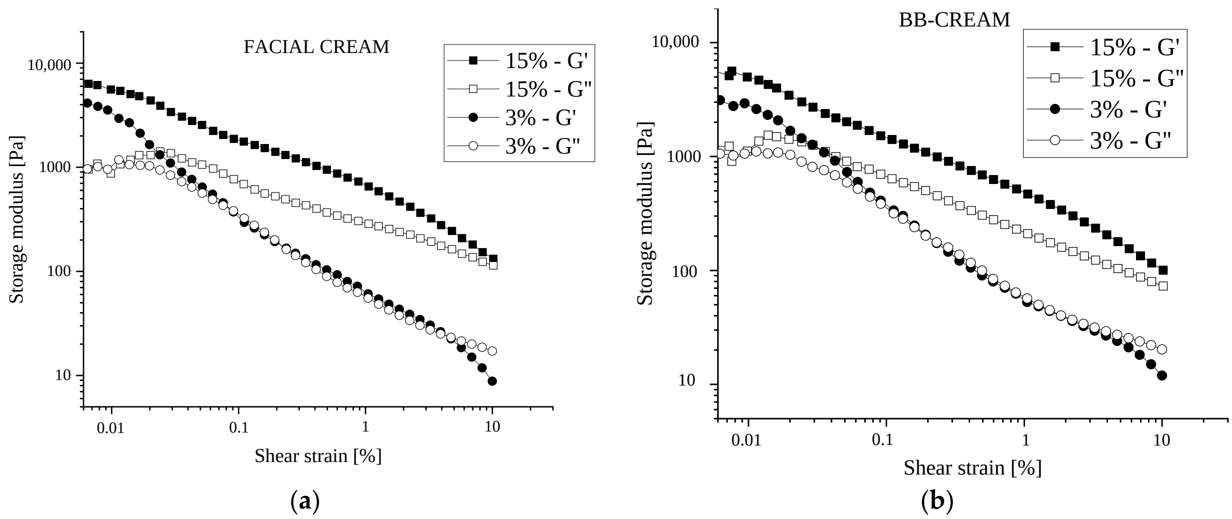


Figure 5. (**a**) Viscoelastic profile for BB cream after preliminary stability. (**b**) Viscoelastic profile for facial cream after preliminary stability.

3.7. Decision Matrix

The result for each prototype (calculated as described in the methodology) is shown in Table 7. Hence, biowax percentage which obtained the best score was selected, 9% for both. This is the suggested dosage for its sensory profile and stability. The formulations selected were compared sensorially with a commercial reference product (moisturizing cream and BB cream of Natura FACES). Figure 6 shows that the property that was the least close was gloss, being higher for the prototypes developed, which decreases the level of user acceptance, as facial creams usually leave a matte finish.

Table 7. Decision matrix results for sensory and stability analysis.

%Biowax	Result	
	Facial Cream	BB Cream
3%	201	285
6%	210	272
9%	233	287
12%	171	257
15%	185	252

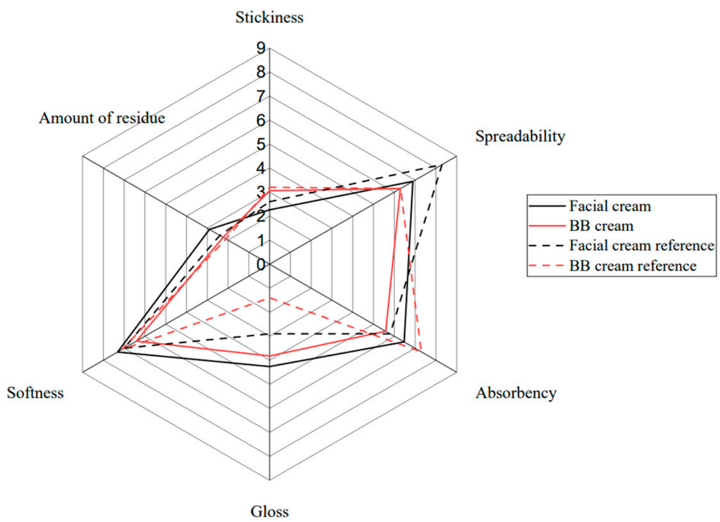


Figure 6. Sensory panel of facial cream, BB cream and two commercial creams.

4. Conclusions

The result of this work reveals that the biowax derived from hydroprocessing of crude palm oil presents high potential as a cosmetic ingredient, firstly as an emollient as it modifies the sensory properties of the formulations developed and secondly as a thickening agent, since the viscosity and the spreadability present a high dependence of biowax percentage used in the formulations studied.

Related to the stability, it is concluded that the formulations are able to maintain their pH. On the other hand, the cream shows phase separation in the prototypes with lower viscosity; however, the optimum value of biowax found where the separation decreases and avoids the formation of a supernatant is 9%. Furthermore, all tests showed a non-Newtonian and shear-thinning behavior, being a desirable rheological property in cosmetic formulation. In addition, the obtained emulsions can be considered as stable solid-like fluids since $G' > G''$.

Finally, the percentage that presented the best stability and sensory profile was 9% of biowax for both creams, which is expected to be easily accepted in the market because, compared to a reference product, its sensory properties are very similar.

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Institutional Review Board Statement: The study was conducted in accordance with the Declaration of Helsinki and approved by Ethics Committee of the Industrial University of Santander, Bucaramanga, Colombia, act N.2 from 21 February 2020.

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Data Availability Statement: The data presented in this study are available upon request from the corresponding author. Data are not publicly available due to privacy restrictions.

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