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Effect of the *Cera Carnaubae* and *Euphorbia Cerifera Cera* waxes in the Synthesization of Natural Lipsticks

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Abstract. Lipsticks are without a doubt one of the most beloved products, among many cosmetic products. The phenomenon of lip coloring has historical use and has served over the years for various purposes e.g., historically lip coloring has shown social status and a form of freedom (3). This work will elaborate in more detail various formulations for lipsticks based on the use of natural ingredients only, bringing to knowledge the effect of combining plant-based waxes such as ***Cera Carnaubae*** and ***Euphorbia Cerifera Cera*** with animal-based waxes such as ***Cera Flava***, in different amounts and formulations. Through a detailed and thorough analysis of the physical and chemical properties of the lipstick samples, this work will discuss in detail the effect of combining different natural ingredients to synthetize lipsticks.

Keywords: natural lipsticks, natural cosmetics, waxes, oils

1. Introduction

Cosmetic is any substance that is intended for external use in our body (skin, lips, nails, hair, teeth, etc.) which aims to keep clean, protect, nourish, beautify, or perfume the body [1]. The scientific development of cosmetics first began in the XIX century. As a concept cosmetic generally includes treatments for various skin diseases as well as the prevention and elimination of skin defects on the face, neck, legs, and hands.

There are thousands of different cosmetic products on the market, in the United States alone there are approximately 12,500 unique chemical ingredients approved for use in the production of personal care products. A typical product may contain from 15-50 ingredients. Given that the average user uses between 9 and 15 personal care products per day, studies have estimated that when combined with the addition of perfumes, users apply about 515 individual chemicals on their skin each day through cosmetic use [2]. Keeping these numbers in mind it is very important to know what kind of substances we expose our body to. With this work the aim was to create lipstick formulations that would contain as few ingridients as possible but still fulfill all of the nessesary properties for a practical application whilst having soothing and healing effects on the lips.

1.1 Lipsticks and what they are made of

The lipsticks used today are similar to the mass formulations that began in the mid-twentieth century and generally consist of waxes, oils, alcohols and pigments. Although during the industrial development the formulations have undergone some changes, the basis of the formulations has practically continued to be constant, and each ingredient has a very important role in the structural and aesthetic aspect of the lipsticks [3]. Commonly used waxes, beeswax, carnauba wax and candelilla wax give the final product the desired shape and firmness. It is therefore very important to find the exact balance of the wax mixture during the formulations. The melting point of the waxes affects the softness of the final product, waxes with low melting points can give a soft final product which is difficult to put into molds and apply on the lips. A very strong consistency of lipsticks causes difficulty in application and spreading [3]. Depending on the current trends, the ideal lipstick can be glossy, matte, transparent, intense colored, etc. An optimal lipstick has a melting point of 41-55 ℃ [4], so that it does not melt at high temperatures and is stable enough not to break during application. At the same time, it should be soft enough to be easily applied and leave behind a soft sensation [5]. Lipsticks contain approximately (62%) Oils, (25%) Waxes, (5%) Lanolin, (5%) Pigments and (3%) other ingredients such as preservatives [5].

2. Materials and Methods

For this work, ten lipstick formulations with different amounts of ingredients have been selected and their physical and chemical properties have been analysed and discussed.

Table 1. Laboratory equipment and reagents used

Laboratory equipment	Reagents (Solvents)
Electric Stove	Ethanol
Laboratory Glasses	Chloroform
Spatula	Dichloromethane
Hygienic Napkins	Acetone
Erlenmeyer	Diethyl ether
Water bath	
Electric Scale	
Lipstick Mold	
Lipstick Tubes	

Table 2. shown below lists all the ingredients that were used to create each lipstick formulation. Overall, 10 samples were prepared in the laboratory and each one of them contained different ingredients in various combinations and amounts. The waxes used Beeswax, Carnauba Wax and Candelilla Wax added to the stability and solid structure of the lipsticks whilst the Oils that were added to the formulations provided the final products with a light application and soothing properties.

Table 2. Ingredients and quantities used for each sample

Ingredients	Quantity
Sample 1:	
Beeswax	1.0 g
Castor Oil	2.0 g
Coconut Oil	2.0 g
Sample 2:	
Beeswax	2.0 g
Coconut Oil	4.0 g
Sample 3:	
Beeswax	2.0 g
Coconut Oil	2.0 g
Sample 4:	
Beeswax	0.5 g
Candelilla Wax	0.5 g
Shea Butter	0.5 g
Coconut Oil	0.5 g
Castor Oil	2.0 g
Sample 5:	
Carnauba Wax	0.5 g
Candelilla Wax	0.5 g
Coconut Oil	1.0 g
Castor Oil	2.0 g
Sample 6:	
Carnauba Wax	0.5 g
Beeswax	1.0 g
Jojoba Oil	0.5 g
Castor Oil	2.0 g
Sample 7:	
Carnauba Wax	0.5 g
Beeswax	0.3 g
Candelilla Wax	0.2 g
Shea Butter	0.5 g
Lanolin	0.5 g

Castor Oil	2.0 g

Sample 8:

Carnauba Wax	0.5 g
Beeswax	0.3 g
Candelilla Wax	0.2 g
Shea Butter	0.5 g
Lanolin	0.5 g
Castor Oil	2.0 g

Sample 9:

Beeswax	1.0 g
Carnauba Wax	0.25 g
Candelilla Wax	0.25 g
Shea Butter	1.5 g
Castor Oil	1.5 g
Jojoba Oil	1.0 g

Sample 10:

Beeswax	1.5 g
Carnauba Wax	0.25 g
Candelilla Wax	0.25 g
Shea Butter	0.5 g
Castor Oil	1.0 g
Jojoba Oil	0.5 g

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3. Experimental Procedure

In general, the experimental work procedure is identical for each formulation. For each sample we have taken three times (3x) the amount of the ingredients given in **Table 2**. For demonstration purposes we will take **Sample 1** as an example:
The experimental work consists of two ingredient phases:

- The Solid phase
- The Liquid phase

1) For the solid phase, the solid ingredients were first measured separately with an analytical scale. 3g of Beeswax and 6g of Coconut Oil were placed together in a laboratory beaker.

2) The laboratory beaker with the mixture of solid ingredients was then set for melting in a water bath. Given that the waxes used during the formulations have relatively high melting points, melting can take up to several minutes. The melting process was constantly monitored, and the mass was mixed from time to time.

3) When all the solids melted and formed a liquid mas, the liquid phase was added to the mixture, for the first sample the liquid phase was 6g of Castor Oil.

4) After adding the liquid phase, the laboratory beaker with the mixture was placed back into the water bath, where the two phases mixed well with each other to form a homogeneous mass.

Fig 1. Steps 1 – 4 of the Procedure

The second step involves shaping and placing the lipsticks in the appropriate tubes:

5) The lipstick mold was filled with the liquid mass. For this purpose, we used an aluminium lipstick mold with a diameter of 12.1 mm. After filling it the liquid mixture stayed in the aluminium mold till hardened, about 15-20 min, depending on the room temperature.

6) After the samples hardened, they were taken out of the aluminium shaper and placed in the respective lipstick tubes, placement in the tubes was done by inserting the head of the solidified samples in the lipstick tubes and extracting them whilst pulling upwards.

Fig 2. Steps 5 – 6 of the Procedure

4. Results and Discussion

The best results obtained during the experimental work are presented in **Table 3**, the parameters were measured in the same conditions for all ten samples.

Table 3. Results of the experimental measurements

Evaluated Parameters	**Results**
pH	6.5 – 7.0
Melting Point	51.2 ℃ - 69.9 ℃
Breaking Point	1.06 N - 3.25 N
Solubility	Dichloromethane
Temperature Stability	Normal, Good

4.1. pH Value

The pH value was determined by using two methods:

1) Digital pH meter
2) Indicator letters

For the first method the pH meter electrode was immersed in 1g of each molten sample and then the pH value was read. For the second method, the indicator paper was dipped directly into each molten sample and based on the color of the indicator paper, the pH interval of the samples was determined. As a conclusion the average pH value of the samples was between 6.5 and 7.0, i.e., in the neutral zone. According to the references the ideal pH of a lipstick is between 7.45 -7.75 [6].

4.2. Melting Point

To measure the melting point, about 0.5 g of each sample was placed in the respective capillary tubes. The capillary tubes were then placed one after the other in the apparatus and the melting point was measured.

Reference temperatures:
● Start = 45 ℃
● Grade = 2 ℃
● Max Temp. = 75 ℃

The ideal lipstick should have a melting point between 40-55 ℃ [5]. According to the measurements made, the first sample with a melting point of 51.2 was very close to the ideal melting point. The highest melting point of 69.9 ℃ was determined in sample 8, a phenomenon which can be attributed to the higher amount of Carnauba wax present in this sample, out of the three waxes present Carnauba wax has the highest melting point.

4.3. Breaking Point

In laboratory conditions, by means of a laboratory improvisation the force which the lipsticks can withstand was measured. A 250 ml glass beaker was taken and was tied around with a pendant, the respective lipstick and a burette were strengthened to the same metal holder and afterwards the burette was filled with water. The glass beaker was placed above about 3 mm away from the tip of the lipstick, the tip of the lipstick was then placed down against the glass beaker and slowly the glass was filled with water from the burette.

The filling continued until the lipstick broke. After lipstick breakage, the water from the burette was stopped and the weight of the glass at the moment of breakage was measured. The force in Newton was then found by substituting the corresponding weight value in the Force formula:

$$\mathbf{F = m \times 9.8\ m/s^2}$$

Compared to the reference values, the lipstick samples showed lower values of hardness than the commercial samples, sample 3 showed a hardness of 3.25 N close to the commercial sample REVLON - Kiss with Coral, with a hardness of 3.65 N [7].

4.4. Solubility

Given that we worked with non-polar organic ingredients respectively oils, fats and waxes it was expected that the solubility of the samples in organic solvents would be good. Out of the five solvents that were used the best solubility was shown by Dichloromethane and Chloroform, which left an almost clear solution with no residue. Acetone and Diethyl ether have shown moderate solubility. Cold ethanol, which is rather known as a polar solvent, left a lot of undissolved sample residues in the test tubes being therefore the most unsuitable solvent.

4.5. Temperature Stability

This parameter determines the stability of lipstick samples at three different temperatures:
• At a temperature of ≈ 4 °C
• At room temperature ≈ 25 °C
• At a temperature of ≈ 45 °C

For the measurement of temperature stability at ≈ 4 °C, the samples were placed in the refrigerator for 24 hours, after extraction as expected a more solidified form was observed but no significant changes appeared.

At room temperature ≈ 25 °C, there were also no significant changes observed.

To measure the temperature stability at ≈ 45 °C, the samples were inserted into an Erlenmeyer and sealed with a tap. The Erlenmeyer was then immersed in a water bath for 3 hours without interruption, the temperature in the water bath was measured from time to time to make sure it was in the right settings and as a result we concluded that most of the samples were stable at ≈ 45 °C, with sample 10 showing the highest stability and sample 4 and 5 the lowest.

Fig 3. Measurement of temperature stability ≈ 45 ℃

5. Thin Layer Chromatography (TLC)

To perform the Thin Layer Chromatography six samples were taken, three of which were the natural lipstick samples prepared in the laboratory and the other three were samples of industrial lipsticks. About 0.5 g of each sample was dissolved with chloroform in the respective test tube, a solvent that was selected after showing the best results in the solubility test. The samples were also used in the undissolved state for the chromatography. In Fig 4. we see 12 dots on the chromatograph, starting from right to left we always have one undissolved dot and one dissolved dot of each respective sample. Based on the obtained chromatograph in general the undissolved samples 2 and 3 have shown the highest purity since in the chromatogram they have appeared with only one moving point or with only one spot. An increase in the number of spots can be observed in the dissolved samples. In general, industrial samples have shown lower purity, the number of spots of these samples in both dissolved and undissolved form is generally higher. From the measurements made Rf was found for each moving spot of the sample components.

$$\mathbf{Rf = dK / dT x 100}$$

The retention factor (Rf) represents the ratio between the travel distance of the components from the starting point and the travel distance of the solvent from the starting point. The solvent we used traveled 18 cm along the chromatographic paper.

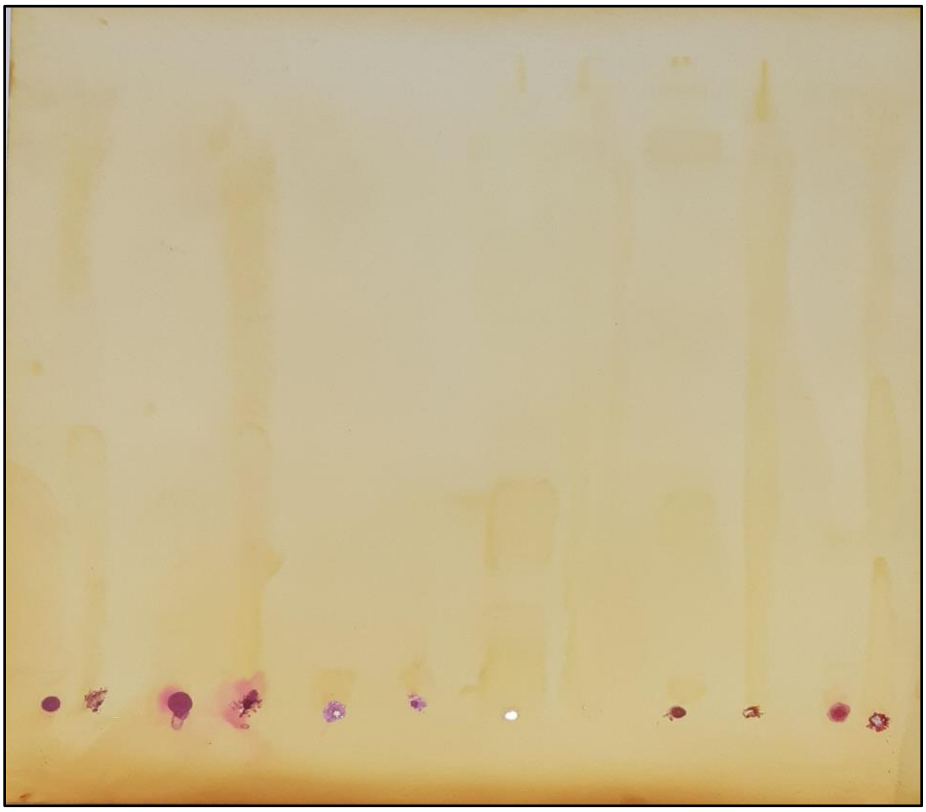

Fig 4. Chromatograph of the performed TLC

Table 4. Samples and results of the TLC

Natural lipstick samples

1. Pigment Carmine (Sample 1)

Undissolved sample	Dissolved sample
1. Rf = 27.7 (5cm)	1. Rf = 5.5 (1cm)
2. Rf = 52.7 (9.5cm)	2. Rf = 93.3 (16.8cm)
3. Rf = 89.4 (16.1cm)	

2. Pigment Fe2O3 (III) (Sample 8)

Undissolved sample	Dissolved sample
1. Rf=96.6 (17.4cm)	1. Rf=9.4 (1.7cm)
2. Rf = 52.7 (9.5cm)	2. Rf=36.1 (6.5cm)
	3. Rf=86.1 (15.5cm)
	4. Rf=92.7 (16.7cm)
	5. Rf=96.6 (17.4cm)

3. Without Pigment (Sample 9)

Undissolved sample	Dissolved sample
1. Rf=96.1 (17.3cm)	1. Rf=20.5 (3.7cm)
	2. Rf=37.7 (6.8cm)
	3. Rf=96.6 (17.1cm)

Table 5. Samples and results of the TLC

Industrial lipstick samples

4. Makeup revolution Enchant

Undissolved sample	Dissolved sample
1. Rf=5.5 (1cm)	1. Rf= 11.6 (2.1cm)
2. Rf=84.4 (15.2cm)	2. Rf=85.5 (15.4cm)

5. Catherine Arley (Matte) M04

Undissolved sample	Dissolved sample
1. Rf=27.7 (5cm)	1. Rf=35.5 (6.4cm)
2. Rf=44.4 (8cm)	2. Rf=69.4 (12.5cm)
3. Rf=84.4 (15.2cm)	

5. Wycon Cosmetics 207

Undissolved sample	Dissolved sample
1. Rf=44.4 (8cm)	1. Rf=38.3 (6.9cm)
2. Rf=91.1 (16.4cm)	2. Rf=52.7 (9.5cm)
	3. Rf=92.7 (16.7cm)

6. Conclusion

During this experimental work many features have been defined and many others have been improved. Through the measurements and the end results we were able to conclude that the lipsticks that had a mixture of plant and animal-based waxes as their main ingredients generally showed better results in their stability and applicability.

Lipstick samples that contained only plant-based waxes in their formulation provided a very solid structure which was not as practical for the applicability. On the other hand, lipstick samples that contained beeswax as their main ingredient showed much better results for a smooth and easy applicability but did not have a very stable and solid consistency. In conclusion, we can say that most of the lipstick samples that were created in the laboratory showed qualitative and suitable properties for application with sample 1 and 8 showing the best results in comparison to the remaining samples.

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