

# Formulation and Evaluation of Natural Pigmented Lipstick from Roselle Flower and Secang Wood Extracts

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## Abstract

Lipstick is a cosmetic product for the lips consisting of pigments, oils, waxes, and moisturizers. Natural lipstick is safe to use and contains natural nutrients to maintain lip health. The use of synthetic dyes in lipstick can have adverse effects on the skin and health; hence, the use of natural dyes from various natural sources needs to be considered. Secang wood and Rosella flowers are plants with important compound contents that have antioxidant properties. Secang wood contains brazilin, alkaloids, flavonoids, saponins, tannins, phenylpropane, and terpenoids. Meanwhile, Rosella flowers that grow in tropical areas have anthocyanin pigments as part of flavonoids that function as antioxidants. This research aims to determine the formulation of lipstick containing extracts of Secang wood and Rosella flowers and to test the antioxidant activity of the preparation. Extraction was carried out by maceration using 70% ethanol solvent. Then, a phytochemical analysis was conducted to determine the extract content, and a lipstick formula was made by comparing the ratio of Rosella and Secang extracts. The optimal formula selection was performed using the Simplex Lattice Design method with quality testing parameters, including pH, melting point, hardness, and antioxidant activity of the preparation. The data analysis showed that the optimal ratio of the two extracts was 2.5% and 7.5% for Rosella and Secang extracts, respectively. At this composition, the optimum pH value was 5, the melting point was 57°C, the hardness was 1.87 seconds, and the antioxidant activity was classified as strong.

**Keywords:** secang; roselle; lipstick; natural pigment

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## INTRODUCTION

Cosmetics are materials or preparations intended to be used on the external skin of the human body, such as the epidermis, hair, nails, lips, and external genital organs, or the teeth and mucous membranes of the mouth, especially to clean, perfume, change the appearance,

and improve body odor or protect or maintain the body in good condition.<sup>1</sup> Lipstick or lip color is a cosmetic preparation that is used as a lip color to perfect the shape and give decorative color to the lips to support the appearance. Lipstick is very susceptible to being swallowed with saliva or food and drinks consumed, so it has a terrible impact if the

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lipstick contains harmful ingredients, including synthetic dyes. If dangerous synthetic dyes continue to be swallowed when using lipstick, it can cause poisoning, irritation, and liver disorders.<sup>2</sup> The dye is a significant factor in the preparation of lipstick. These dyes can be derived from natural dyes and synthetic or chemical dyes. The use of synthetic dyes in cosmetics, including lipstick, can cause adverse effects on human health. Some synthetic dye materials used in lipstick have been found to have carcinogenic potential or may cause cancer. In addition, the use of synthetic dyes can also increase the risk of irritation, inflammation, and allergic reactions on the skin, especially for individuals with high skin sensitivity.<sup>3</sup> The use of synthetic dyes also has negative effects on the environment. Synthetic dye materials that are discarded into the environment can disrupt the balance of the ecosystem and trigger serious environmental damage. Therefore, the use of natural dyes in lipstick has become a safer and more environmentally friendly alternative for consumers and the environment.

Natural dyes can be acquired from plants, animals, or mineral sources that have better biodegradability, and high compatibility with the environment.<sup>4,5</sup> Rosella plant is an ornamental plant that is a type of shoe plant. Indonesia, which has a tropical climate, causes rosella to thrive. Rosella plant (*Hibiscus Sabdariffa* L.) is very well known nowadays as Rosella flower petals can cure various diseases such as hypertension, diabetes, and diuretics.<sup>6</sup> The most active substances in rosella flower petals include gossypetin, anthocyanins, and hibiscus glucosides.<sup>7</sup> The red color of rosella flowers (*Hibiscus Sabdariffa* L.) is caused by the content of anthocyanins, peroxides, and antioxidants that can defend and provide protection

from the effects of free radicals. However, free radicals are more than defense capabilities. These natural antioxidants can interrupt so that they break the normal oxidation-reduction chain that causes oxidative damage.<sup>8</sup> *Caesalpinia sappan*, also known as Secang wood, is a plant species that has been widely used in traditional medicine due to its various bioactive compounds. The heartwood of this plant contains natural pigments, such as brazilin and brazilein, which have been shown to possess antioxidant, anti-inflammatory, and anticancer properties. Other bioactive compounds found in *Caesalpinia sappan* include flavonoids, terpenoids, and lignans, which have potential applications in the treatment of various diseases.<sup>9,10</sup> Moreover, *Caesalpinia sappan* has also been used as a natural dye and in the production of red pigment for various applications.<sup>11</sup> Secang wood, with its content of brazilin compounds, has been known to be a potential source of natural antioxidants.

Several previous studies have been conducted on the use of natural dyes in lipstick formulations. They found that natural dye from Secang, Rosella, Ruruhi fruit, and Kecombrang could be used as an alternative to synthetic dyes in lipstick, with promising results in terms of color stability and oxidation resistance.<sup>2,4,12-14</sup> However, those studies were limited to the use of only one extract. Therefore, this study used a combination of Secang and Roselle extracts by optimizing their combination to obtain a more attractive and synergistic formula in lipstick formulation.

## METHODS

The research was conducted using an experimental research design with the Design Expert method. The samples used in this study were Secang wood extract

(*Caesalpinia Sappan* L.) and Roselle flower (*Hibiscus Sabdariffa* L.), which had been made with the given ratio seen in Table 1.

**Table 1.** Formulas with Roselle and Secang Wood Extract Variation

Ingredients	Formula (%)							
	F1	F2	F3	F4	F5	F6	F7	F8
Roselle Extract	10	2.5	5	7.5	0	10	0	5
Secang Extract	0	7.5	5	2.5	10	0	10	5
Vaseline Alba	4	4	4	4	4	4	4	4
Cetyl Alcohol	2.83	2.83	2.83	2.83	2.83	2.83	2.83	2.83
Oleum Ricini	2	2	2	2	2	2	2	2
Cera Alba	2	2	2	2	2	2	2	2
Propylene glycol	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
BHT	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Nipagin	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Tween 80	3	3	3	3	3	3	3	3

The dried *Simplicia* was made by collecting raw materials obtained from Batu Materia Medika in the form of Secang wood and Rosella flowers, cleaning, sorting, aerating, drying, and processing them into powder. The drying process was done in the oven at a temperature of 50°C until dried thoroughly. The dried *Simplicia* was then ground to become powder, after which it was sieved using a 40-mesh sieve. The extract was made utilizing powder weighing approximately 1 kg of Rosella flower powder and sappan wood powder. Maceration was carried out using 6 liters of 70% ethanol solvent (comparison 1:6), soaked for 3x24 hours while stirring occasionally, then filtered to obtain a liquid extract. After being concentrated using a vacuum rotary evaporator at a temperature of 60°C and 120 rpm to produce a 75% yield of Secang and Rosella extracts obtained, then evaporated using a water bath with a temperature of 60°C until the extract became dry. They were weighed and then recorded.

### Phytochemical Screening

Phytochemical analysis was carried out to determine the class of chemical

compounds contained in *simplicia*. The analysis carried out included an alkaloid test, saponin test, and tannin test.

### Lipstick Manufacturing

The lipstick was manufactured by weighing the ingredients to be used and then the base (vaseline, cera alba, cetyl alcohol, BHT) was melted. Furthermore, the Rosella extract and Secang extract are mixed with oleum ricini and tween 80 (mixture a). PPG and nipagin (mixture b) were weighed, then mixed between (mixture a) with (mixture b) until homogeneous. After the base was completely melted, all the ingredients were mixed until homogeneous in one cup. Lubricate the lipstick mold first using paraffin so it doesn't stick. After that, it was put in the refrigerator until the next lipstick evaluated the lipstick.

### Organoleptic Test

The organoleptic examination was carried out by observing the lipstick preparations with Roselle flower extract and Secang wood visually in terms of color, smell, and texture. An organoleptic test was carried

out to determine the physical changes of the preparation.

#### **pH test**

The pH test was carried out using a universal pH indicator. The measurement aims to determine the level or alkalinity of lipstick. The pH value of skin products for various kinds of lipsticks was between 4.5-6.5.<sup>15</sup>

#### **Melting Point Test**

The melting point was tested by putting the lipstick on the waterbath at 60°C. It was left and then observed whether the lipstick has started to melt. It was observed at what temperature the lipstick began to melt.<sup>16</sup>

#### **Hardness Test**

Hardness test using a strength tool utilizing a lipstick rod in the middle of the tool plate, then added weight, every 30 seconds added until the lipstick breaks. This is done to determine the resistance of lipstick preparations in the face of tensions obtained both during the packaging process and in storage.<sup>17</sup>

#### **Homogeneity Test**

The homogeneity test for the 8 types of lipstick was carried out by placing the lipstick stick on the surface of the slide and then leveling it using another object with a certain pressure.<sup>2</sup>

#### **Spreadability Test**

The spreadability was determined by applying the lipstick 10 times and then observing the color of the lipstick attached to the arm. Lipstick preparations are said to have a smearing power if the color sticks to the skin of the arm a lot and evenly.<sup>15</sup>

#### **Hedonic Test**

The hedonic test was carried out randomly with 20 panelists by making 4 category

scales ranging from very dislike, dislike, like, and like to know the level of liking for the product. The researcher then applied lipstick on the back of the hand that has been made with different concentrations. They calculated the percentage of preference for each preparation.<sup>2</sup>

#### **Irritation test**

This test was carried out once a day for 3 consecutive days. A positive irritant reaction is characterized by redness, itching, or swelling of the skin of the forearm.<sup>15</sup>

#### **Antioxidant Activity Test**

Preparation of DPPH (1,1-Diphenyl-2-Picrylhydrazyl) solution

To initiate the experiment, the required equipment and resources must be gathered. The primary standard solution was then produced by precisely measuring 2 mg of DPPH powder, which is subsequently transferred into a 100,0 ml volumetric flask and dissolved with pro-analytical grade ethanol until the volumetric mark was reached. The resulting solution was then subjected to UV Vis spectrophotometry at a wavelength of 516 nanometers.

#### **Preparation of Vitamin C Solution**

To produce a primary standard solution, 25 mg of ascorbic acid was precisely measured and transferred into a 25 ml volumetric flask. Subsequently, the solid was dissolved in ethanol until the volumetric mark is reached, thus generating a mother solution with a concentration of 1000 ppm. Next, aliquots of 0.1, 0.15, 0.2, and 0.3 milliliters of the primary solution were taken, and each aliquot was diluted in a 50-milliliter volumetric flask using ethanol to yield solutions with concentrations of 2, 3, 4, 5, and 6 ppm.

### Inhibition Measurement Test

The evaluation of the antioxidant activity of the lipstick under investigation was performed using a spectrophotometer. The experimental protocol involved the preparation of the necessary materials and tools. Initially, 25 mg of each formulation of the lipstick was weighed and dissolved in pro-analysis ethanol, and the solution was then transferred to a 25 ml volumetric flask to obtain a concentration of 1000 ppm. Subsequently, 0.1 ml, 0.3 ml, 0.5 ml, 0.7 ml, and 0.9 ml of the prepared solution were separately mixed with pro-analysis ethanol in 10 ml volumetric flasks, resulting in concentrations of 10 ppm, 30 ppm, 50 ppm, 70 ppm, and 90 ppm, respectively. In the next step, 0.2 ml of each test solution, including the lipstick sample and the ascorbic acid comparison solution, were mixed with 3.8 ml of DPPH solution. The mixture was then transferred to an aluminum foil-wrapped vial and incubated for 30 minutes at each

concentration. The absorbance at the maximum wavelength and the blank solution was observed using a UV-Vis spectrophotometer at a wavelength of 516 nm. The percentage of DPPH inhibitory activity was calculated using the following equation.<sup>18</sup>

$$\% \text{inhibition} = \frac{(\text{Sample Abs} - \text{NC Abs})}{(\text{PC Abs} - \text{NC Abs})} \times 100\%$$

### RESULTS AND DISCUSSION

Extracts obtained from maceration technique is then evaporated with rotary evaporator and further using a water bath at 50°C until it forms a thick extract. For analysis, 2 g of the extract is weighed and dissolved with an appropriate reagent according to the test to be performed in a 100 ml volumetric flask. The phytochemical screening result can be seen in table 2.

**Table 2.** Data on Phytochemical Screening Results of Rosella and Secang Extracts

Secondary Metabolite	Extract	Result	Description
Alkaloid	Roselle	+	White-colored sediment
	Secang	-	No sediment
Saponin	Roselle	-	No bubbles
	Secang	+	White bubbles formed
Tanin	Roselle	+	White-colored sediment
	Secang	-	Brown colored sediment

In conducting alkaloid test, 2 g of the extract was mixed with 1% 10 ml of hydrochloric acid in a test tube and heated for 3 minutes in a water bath. The resulting solution was filtered with cotton into test tube A and test tube B, followed by adding 3 drops of Mayer reagent to each solution. A positive reaction was indicated by the presence of a white precipitate in the Mayer reagent. Phytochemical screening showed that the reaction was positive in the Rosella extract. The saponin test involved adding 300 mg of the extract to a

test tube, followed by adding 10 ml of water and shaking vigorously for 30 seconds. The tube was then left upright for 5 minutes, and the presence of foam up to 3 cm from the surface indicated the presence of saponins. Phytochemical screening indicated a positive result for the Secang extract, as indicated by the formation of foam on the surface of the liquid. The tannin test involved adding 3 grams of extract to a test tube, followed by adding 15 ml of water and heating for 30 minutes in a boiling water bath to separate

tannins from the sample. The resulting filtrate was mixed with 1% gelatin containing 2% NaCl, and the formation of a white precipitate indicated a positive result. Phytochemical screening indicated a positive result for rosella extract and no precipitate for Secang. The principle of the tannin test involves the deposition of protein in gelatin when the sample contains tannin compounds.

The variation concentration level of Roselle and Secang wood extract was determined using design expert application with simplex lattice design method. A design expert is an application

of optimization techniques that are used to determine the best formulation by using data from the evaluation results of the preparation. Optimization can be interpreted as an approach to get the best combination of a product that has certain characteristics. After the lipstick is made, its quality characteristics are tested to ensure that the product meets the desired quality requirements.

**Organoleptic Test**

Parameters assessed in this organoleptic test are the color, odor, shape, and texture of the preparation.

**Table 3.** Organoleptic Test Result Data

Formula	Color	Odor	Texture	Result
F1	Maroon	Odorless	Soft	
F2	Maroon	Odorless	Soft	
F3	Maroon	Odorless	Soft	
F4	Maroon	Odorless	Soft	
F5	Reddish-brown	Odorless	Soft	
F6	Maroon	Odorless	Soft	
F7	Reddish-brown	Odorless	Soft	
F8	Maroon	Odorless	Soft	

The results of the organoleptic test showed that there were differences in the color of each formula, as can be seen in Table 3. In general, the results obtained were red, as expected. Preparations were produced from each formula that had no difference in smell, shape, or soft texture. At extract concentrations of less than 7.5%, the resulting product was red, but at a concentration of 10%, the lipstick had a

reddish-brown color. A slightly purplish color was produced by Rosella extract.

**pH test**

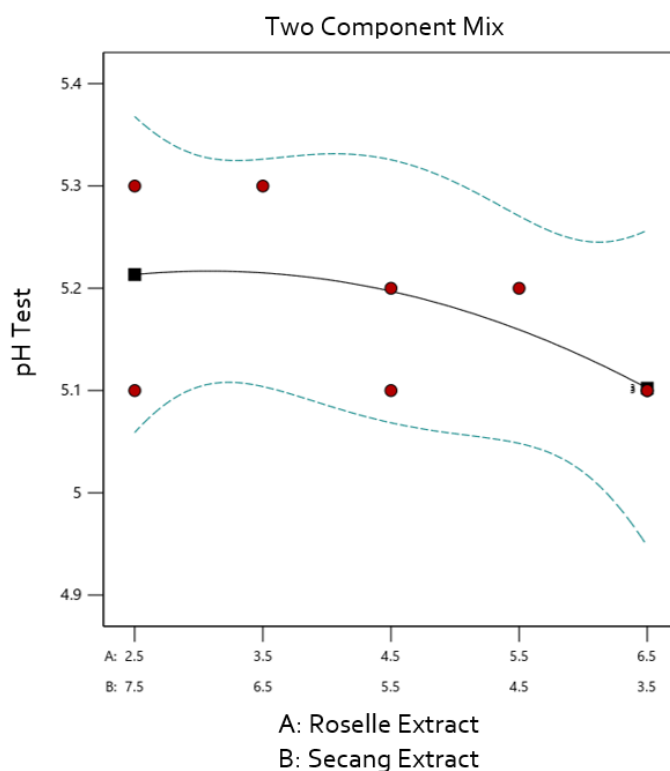
The pH test results showed that the lipstick preparation had a pH of 5. The pH value was still in the normal pH range for the skin as the skin pH for the lipstick ranges from 4.5 to 6.5. If it has a pH lower than 4.5, it can irritate the skin.

Meanwhile, if the pH is greater than 6.5, it can cause scaly skin as it is too acidic.<sup>15</sup> Based on previous research, the smallest degradation of anthocyanin occurs at pH 4, which is 55.83%, and it continues to increase with rising pH. The increasing degradation is attributed to the rise in the number of carbinol bases. Carbinol bases continue to increase as the shift towards a more alkaline pH occurs. Flavylum hydration turns into colorless carbinol bases at the C-2 position in the anthocyanin structure due to the loss of an H<sup>+</sup> ion and the addition of H<sub>2</sub>O.<sup>8</sup> The pH value can also affect the organoleptic color of the lipstick; pH 5 produces a red color in the Secang extract and becomes more

yellowish as the pH becomes more acidic. However, in this case, the pH of all formulas is the same, resulting in a tendency towards a red color.

**Table 4.** Data of pH Test Results

	Average pH Value
F1	5
F2	5
F3	5
F4	5
F5	5
F6	5
F7	5
F8	5



**Figure 1.** Plot of pH Test Contour

In the ANOVA test, the p-value was  $0.1000 > 0.05$ , indicating no significant difference. It showed that Rosella flower extract and sappan wood extract had the same pH, so there was no difference or comparison between the two. It can be seen in the contour plot above that a

straight-line curve is obtained, indicating that the pH between Rosella flower extract and sappan wood extract was the same. The following antioxidant test results were obtained by using the quadratic model.

$$Y = 5.10229(A) + 5.2134(B) + 0.156863(AB)$$

Information:

Y = Lipstick pH Test Results  
A = Rosella Extract

B = Secang Extract

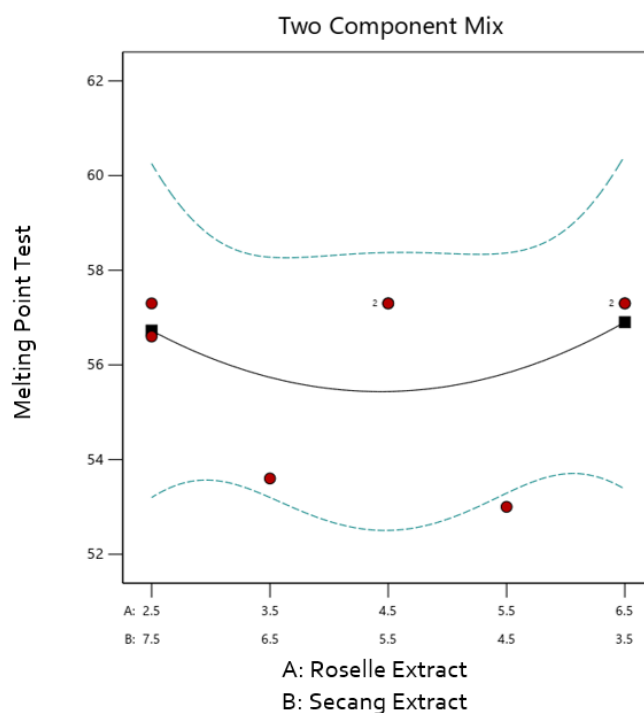
### Melting Point Test

Melting point test results showed that the lipstick preparations from 8 formulas had a melting point range between 50-57°C. All formulations made had good melting points and met the requirements of SNI

16-4769-1998, namely, the melting point was above 50°C for tropical country.<sup>17</sup>

**Table 5.** Data of Melting Point Result

Formula	Melting Point
F1	53±2.64 °C
F2	56±0.57 °C
F3	57±0.57 °C
F4	53±3.21 °C
F5	57±0.57 °C
F6	57±0.57 °C
F7	57±0.57 °C
F8	57±0.57 °C



**Figure 2.** Contour of Melting Point Test Plot

In the ANOVA test, the p-value was  $0.0027 > 0.05$ , which showed significant results. It indicated that a concentration comparison between Roselle flower extract and Secang wood, which gave real and significant results for the melting point test. By using the quadratic model, the equation for the antioxidant test results is as follows:

$$Y = 56.9036(A) + 56.7258(B) - 5.50588(AB)$$

Information:

Y = Melting point test result

A = Rosella Extract

B = Secang Extract

Based on the above equation, it is known that the positive values in A and B influence increased the melting point test results. The contribution of rosella extract concentration in increasing the melting



point value was greater than that of Secang extract.

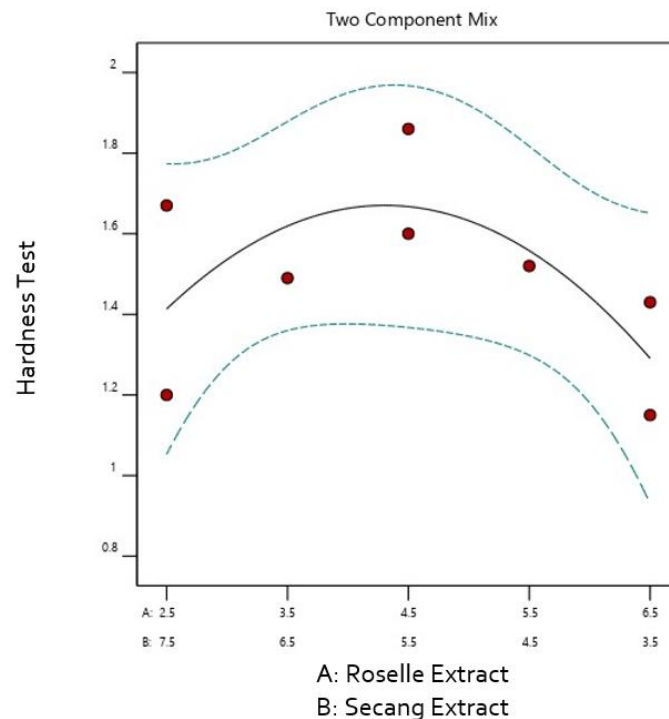
### Hardness Test

If the lipstick is lower than the standard hardness, it will cause the lipstick to break easily and be unable to maintain its shape, making it difficult to apply to the lips. Meanwhile, if the lipstick is too hard compared to the standard, it will be difficult to apply it. <sup>2</sup>.

**Table 6.** Hardness Test Result Data

Formula	Hardness (min)
F1	1.52±0.42
F2	1.20±0.09
F3	1.15±0.05
F4	1.49±0.01
F5	1.60±0.36
F6	1.43±0.49
F7	1.67±0.30
F8	1.86±0.31

The results of the hardness test in table 6 show that Roselle flower extract and Secang wood extract will produce lipstick with proper hardness in all formulations. All lipstick preparations can have an acceptable hardness, which is between 120 and 3600 seconds.



**Figure 3.** Hardness Test Plot Contour

The following antioxidant test results were obtained by using the quadratic model.

$$Y = 1.29095(A) + 1.41317(B) + 1.26118(AB)$$

Information:

Y = Hardness test result

A = Rosella Extract

B = Secang Extract

Based on the above observations, it is known that the positive values in A and B influence increasing the hardness test value. The contribution of the concentration of Secang extract in increasing the hardness test value was greater than that of rosella extract.








### Homogeneity Test

This test was carried out by applying the sample to a glass object.<sup>19</sup> The results of the homogeneity test were that the preparation showed a formula with a homogeneous texture; the base and other

additives were evenly mixed when the stockpile did not show coarse grains, indicating the formula was not homogeneous.

### Spreadability Test

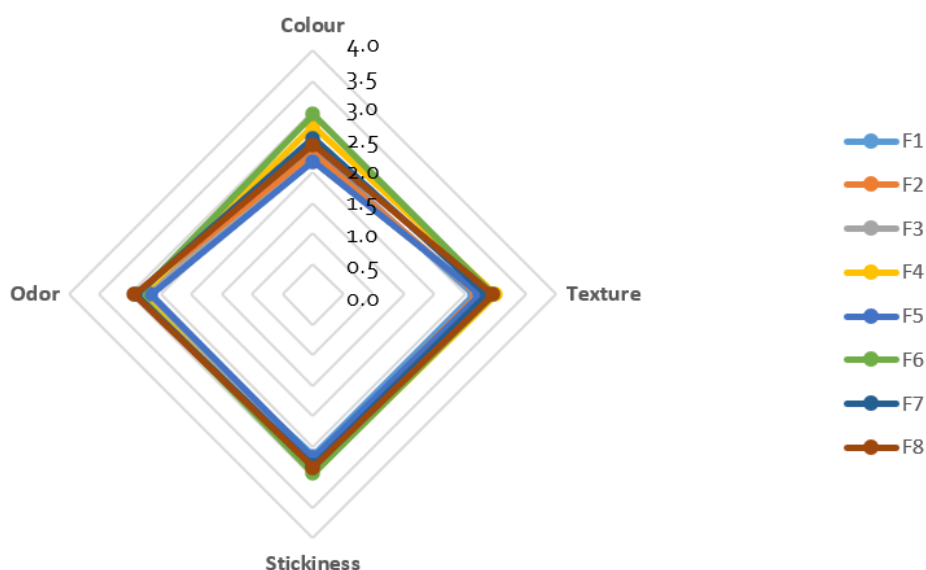
**Table 7.** Data on Spreadability Test Results

Formula	Result	Figure
F1	(+)	
F2	(+)	
F3	(+)	
F4	(+)	
F5	(+)	
F6	(+)	
F7	(+)	
F8	(+)	

It can be seen in Table 7 that the results obtained indicate that the lipstick preparation is easy to apply since the color

appears and looks shiny when applied. The 8 formulas showed different colours in each variation.

## Hedonic Test



**Figure 4.** Hedonic Test Results

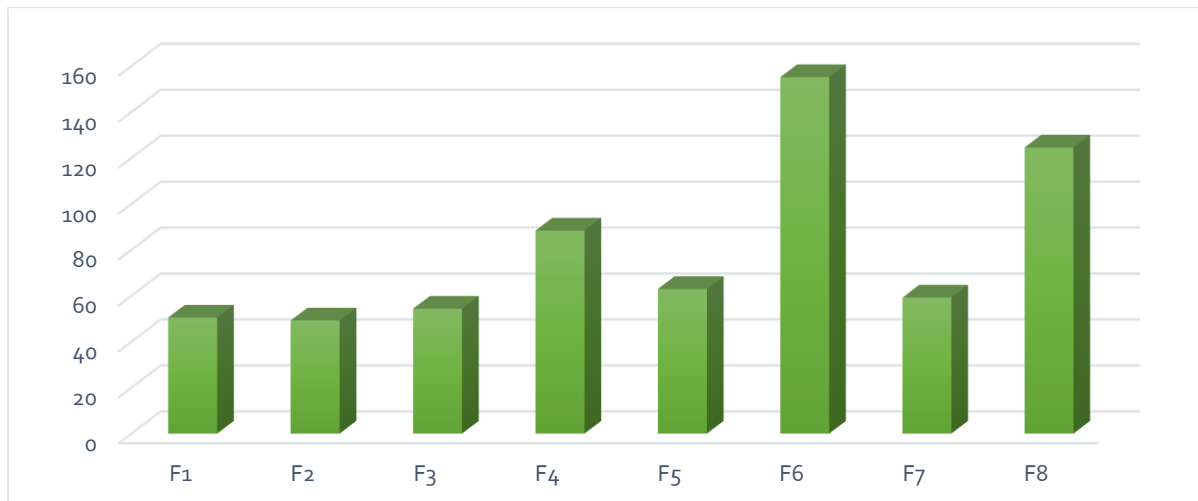
In the figure 4, the results of the hedonic test are obtained. These results were obtained from 32 respondents with predetermined parameters. After the questionnaire's results were collected, the researcher grouped the existing data and then took the average of all respondents in each formulation and each assessment parameter. The results demonstrated that most respondents were interested in all lipstick preparations made, indicating that respondents loved the color, texture, aroma, and appropriate stickiness of the 8 lipstick formulations so that the lipstick stockpile can be accepted among the public. It was shown that formula 6 obtained the most preferred results by respondents in terms of color, scent, stickiness, and texture, where formula 6 consisted of 10% rosella extract.

there was no redness, itching, or swelling of the skin.

## Irritation Test

Observations were made 3 days in a row by applying lipstick on the hands. It showed that all formulations gave negative results to the irritation reaction parameters carried out. The 8 lipstick formulations can be said to be non-irritating to the skin as

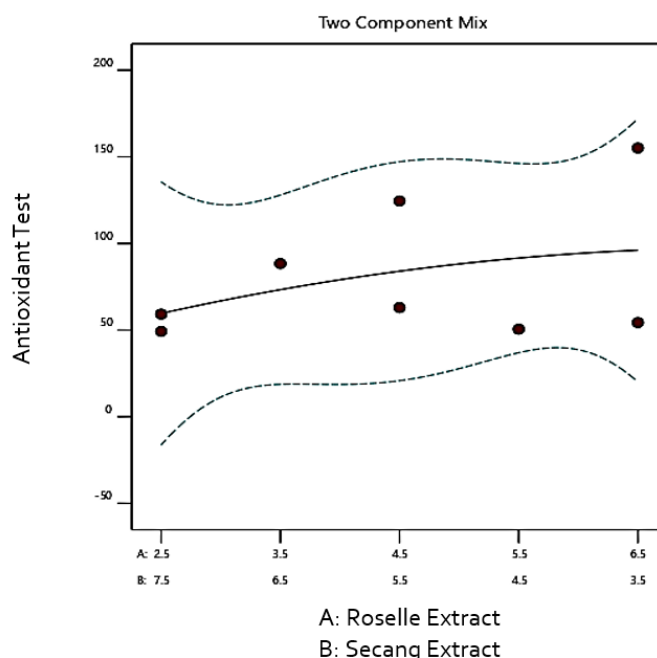
### Antioxidant Test



**Figure 5.** Results of IC<sub>50</sub> Antioxidant Test

The research findings indicated that the average IC<sub>50</sub> values for each formula were considered strong. The IC<sub>50</sub> results, measured in ppm units, were relevant to the typical use of lipstick, where the average woman used 24 mg per day.<sup>20</sup> However, there was a formulation with a different IC<sub>50</sub> value; it can occur due to

damage to bioactive compounds that have a role as antioxidants. Damage to antioxidant compounds in lipstick preparations is caused by several factors, namely the length of contact time given between the active substance and the solvent used or at high temperatures due to prolonged heating.



**Figure 6.** Contour of Antioxidant Activity Test Plot

By using the quadratic model, the following antioxidant test results were obtained.

$$Y = 96.09 (A) + 59.5982 (B) + 24.51 (AB)$$

Information:

Y = Antioxidant test result

A = Rosella Extract

B = Secang Extract

Based on the above equation, it is known that positive values in A and B affect increasing antioxidant yield. The contribution of rosella flower extract concentration in increasing antioxidant value was superior to that of secang wood extract. It can be seen on the above equation, where the coefficient of rosella extract is 96.09, which is greater than the coefficient of the secang extract, which is 59.5982, and the combination of secang and rosella extract can increase the antioxidant with a coefficient value of 24.51.

Design Experts produced the most optimal type of formulation, namely with a concentration of 2.5 rosella extract and 7.5 sappan wood extract. The desirability value in table 12 is 0.902, which is close to the value of 1. The desirability value is an optimization value where a value of 0 means that the target response is not following the desired optimal conditions, and a value of 1 means that the target response is following the desired optimal conditions. The predicted characteristic of the optimal formula is pH 5, melting point 56.73°C, hardness 1.41 minutes, and IC<sub>50</sub> 59.60 ppm.

## CONCLUSION

Based on the result of this research, it can be concluded that the lipstick formulation had an optimal value in the concentration of Rosella extracts at 2.5% and Secang

wood extract at 7.5% with a desirability value of 0.902. At this composition, the optimum pH value was 5, the melting point was 57°C, the hardness was 1.87 seconds, and the antioxidant activity was classified as strong.

A recommendation for further research is to apply polymer encapsulation techniques to the extract to improve its stability and color under various pH, temperature, and light exposure conditions.

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