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Research Article

Formulation and Evaluation of Lipstick with Brazilline Pigment of *Caesalpinia sappan L*

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ABSTRACT

Lipstick is one of the decorative cosmetics used to beautify the lips with attractive colors. A good lipstick preparation must be easy to apply, not irritating, not sticky, and can maintain the durability of the color that sticks to the lips. Secang wood contains pigments, tannins, brazilin, tannic acid, resin, resorcin, brazielin, sappanin, and gallic acid. This study aims to make a formulation of sappan wood lipstick (*Caesalpinia sappan* L.) as a colorant when applied with a concentration of 10%, 14%, 18% and 22% of the lipstick extract of sappan wood (*Caesalpinia sappan* L.) formula which produces color in the formulation. physically and chemically stable lipstick preparations. The preparation was carried out by maceration using 96% ethanol solvent for 3 x 24 hours and evaporated to obtain a thick extract of secang wood. Lipstick testing was carried out physically and chemically including organoleptic test, spreadability, melting point, preference test, irritation test and pH test. The results show different colors in the four formulas, the best formula is Formula II with a concentration of 14% seen from several favorite tests 73.33%, irritation test shows negative results, spreadability shows good results, melting point at 72°C, pH 5.9, and not irritating.

Keywords: *Caesalpinia sappan L, lipstick, natural product, pigmentation, brazillin*

INTRODUCTION

Lip color is a cosmetic preparation that is used to color the lips with an artistic touch so that it can improve the aesthetics of facial makeup (1) . Lip color comes in various forms, such as liquids, crayons and creams. Lipstick is a decorative cosmetic that is used to beautify lips with attractive colors (2,3). A good lipstick preparation must be easy to apply, not irritating, not sticky, and can maintain the durability of the color that sticks to the lips. Commonly used wax bases in lipstick, including carnauba wax, paraffin wax, ozokerite, beeswax, candelila wax, spermaceti and ceresine, all contribute to the hardness of the lipstick (4).

Traditionally, the use of secang plants by the community is quite extensive. The part of the sappan plant that is often used is wood. Until the 19th century, in Kalimantan sappan wood was used as a brownish red dye for food. The components of metabolites contained in *Caesalpinia sappan* L. are brazilin, brazilein, 3'O-methylbrazilin, sappanone, chalcone, sappancalchone and other components such as amino acids, carbohydrates, and palmitic acid with small concentrations (1,5).The content of brazilin in sappan wood gives a distinctive color to sappan wood, ethanol and methanol extracts of sappan wood produce a reddish brown color and produce a bright red color when dissolved in water (6). The results of a literature search showed that secang wood extract in 70% ethanol solvent (red color) was added to a buffer solution of pH 3.8-6.2 to become weak yellow (clear), pH 7.0-8.6 became pink, at pH 8.6-9.4 it is orange, and at pH 10.9-12 the solution is pink (7). The resulting color change is the advantage of sappan wood to produce color variations in decorative cosmetic preparations, especially lipstick preparations (8).

Based on these components the most interesting is the dye, produced by brazilin, which when dissolved in water will give a bright red color. The presence of a Brazilin component gives a characteristic of sappan wood, which is brownish red when oxidized or in an alkaline atmosphere (9). However, Brazilin is considered to have the effect of protecting the body from poisoning caused by chemical radicals (10). Secang wood which has a color pigment caused by the presence of brazilin compounds gives a color from dark orange to deep red. Based on its antioxidant activity. In the application of the use of sappan wood as a traditional medicine, some areas are more familiar with sappan wood with a pink color (11).

The concentration of *Caesalpinia sappan* L. wood extract used as a dye is 8%-22% (12). Based on the description above, the authors wish to make a lipstick formulation of Caesalpinia sappan L. extract with the dye concentration of Secang Wood in this study was 10%, 14%, 18% and 22%.

METHOD

Research Materials and Tools

The ingredients used were thick extract of secang wood, vaseline alba, cera alba, cetyl alcohol, carnauba wax, -tocopherol, oleum ricini, oleum rosae, propylene glycol, methyl paraben, 96% ethanol. The tools used include a 100 ml beaker glass (pyrex), measuring cup (pyrex), stirring rod, evaporating dish, watch glass, spoon, maceration vessel, gauze, dropper pipette, analytical balance, water bath, gas stove, incubator, rotary vacuum evaporator, glass slide, thermometer, pH meter and lipstick container.

Sample Preparation

A number of dried simplicia of sappan wood was put into a maceration container, 96% ethanol was added until the simplicia was submerged, the filter fluid was changed for 3 × 24 hours.

After the maceration process is complete, the maserate is filtered with flannel. The maserate is then concentrated using a rotary vacuum evaporator. Then it is poured into an evaporating cup to make a thick ethanol extract, concentrated in a water bath until a thick extract is formed (13,14).

Specific quality parameters include organoleptic test of extracts which include odor, color, taste and phytochemical screening (identification of alkaloids, flavonoids, tannins, saponins, steroids). Use of the five senses to describe shape, color and smell.

Formula Design

Lipstick Formula Design using Caesalpinia sappan L extract dye. It can be seen in Table 1.

Table 1. Formula Design

Formulation	Formula (%)				Function	Range (%)
	F1	F2	F3	F4		
Caesalpinia sappan L.	10	14	18	22	Coloring	8-22
Carnauba Wax	4	4	4	4	Coating agent	3-5
Vaseline alba	15	15	15	15	Enchances gloss	5-20
cetyl alcohol	3	3	3	3	Emulsifying agent	2-5
Propylene glycol	5	5	5	5	Eluent	5-10
α – tocopherol	0,05	0,05	0,05	0,05	Antioxidant	0,001-0,05
Methyl Paraben	0,1	0,1	0,1	0,1	preservative	0,02-0,3
Ol. Ricini	30	30	30	30	Emolient	30-60
Ol. Rosae	0,05	0,05	0,05	0,05	fragrance	-
Cera alba	Ad 100%	Ad 100%	Ad 100%	Ad 100%	Controlled release agent	1-20

Procedure

Prepare the tools and materials needed, then weigh each material according to your needs. Methyl paraben is dissolved in propylene glycol, after the methyl paraben is dissolved. α - tocopherol is added to oleum ricini. Then added to the mixture of methyl paraben and propylene glycol, then stirred until homogeneous (mixture A). Carnauba wax, vaseline alba, cera alba and cetyl alcohol, were put into a vaporizer cup, then melted over a water bath with a maximum temperature of 87°C (Mixture B). After that, the mixture B is removed from the water bath and then put the mixture A into the mixture B, stir until homogeneous in the cup using a stirring rod while heated. Then add the extract and add oleum rosae, stir until homogeneous. While it is liquid, pour it into the lipstick mold, then packed.

Physical Evaluation

1. Organoleptic

The organoleptic test of the preparation was carried out on any changes in the texture, color and odor of the lipstick preparation, carried out on each preparation during storage (15).

2. **Spreadability Test**
   Apply the product to a glass slide to be visually observed to observe the uniformity of the coating formed, and see if there is any fragmentation, or breakage of the product during application (16).
3. **Hedonic Test**
   The preference test was carried out to determine the panelists' level of preference for the lipstick preparations made. This preference test was conducted visually on 20 panelists. Participants who are used as panelists in this hedonic test are women who are used to using lipstick or who often use lipstick and this is done indoors (16).
4. **Melting Point**
   The melting point method of lipstick used in this study was by placing the lipstick in an oven with an initial temperature of 50°C for 15 minutes, observing whether it melted or not, after that the temperature was increased by 1°C every 15 minutes and observed at what temperature the lipstick started to melt (10,15,17).
5. **Patch Test**
   The technique used in this irritation test is an open patch test on the inner forearm of 10 panelists. An open patch test is carried out by applying the preparation made at the attachment location with a certain area (2.5 x 2.5 cm), leaving it open and observing what happens (15,18,19).

Chemical Evaluation

Lipstick Preparation pH

Determination of pH using a pH meter. The instrument was first calibrated using a neutral standard buffer solution (pH 7.01) and an acidic pH buffer solution (pH 4.01) until the instrument showed the pH value (17,19,20).

RESULT AND DISCUSION

Based on the extraction results in the concentration process, the extract was obtained as much as 27.09 grams, and the yield was 13.5%. Then the organoleptic test and pH test of the extract. The results of the sappan wood extract test (*Caesalpinia sappan* L.) can be seen in the Tabel 2:

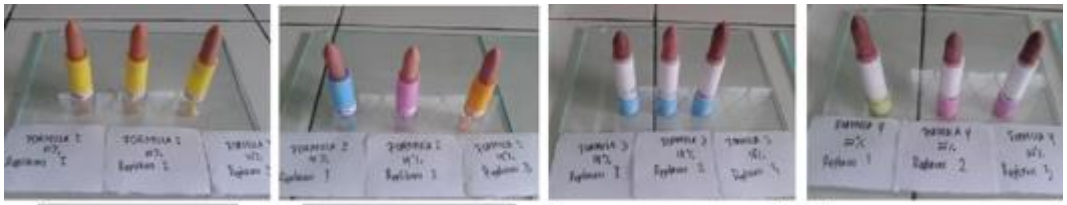
Table 2. Organoleptic Test Results Extract

Organoleptic	Result
Shape	Viscous liquid
Color	Reddish yellow
Fragrant	Typical sappan wood
pH	4,7

Based on the table above, sappanwood (Caesalpinia sappan L.) produces a red pigment due to the presence of brazilein compounds. This pigment has a sharp and bright red color at neutral pH (pH 6-7) and shifts towards purplish red with increasing pH. At low pH (pH 2-5) brazilein has a yellow color (19). Quality parameters indicate the types of pigments found in wood/plant stems are tannins, flavonoids and quinones.

BLANKO

Figure 1. Blank Formulation of Lipstick Preparations



FORMULA I **FORMULA II** **FORMULA III** **FORMULA IV**

Figure 2. Results of the Lipstick Preparation of *Caesalpinia sappan* L. Extract

The concentration of the dye extract of Caesalpinia sappan L. resulted in color differences in the lipstick preparation which had previously been aged for 30 days. Formula I with an extract concentration of 10% on days 0,3,7,15 and 30 produces a pink color that has a solid texture and smells of oleum rosae, Formula II with a concentration of 14% on the 0, 3, 7, 15, and 30 days produces a purplish red color, has a solid texture and smells of oleum rosae, Formula III with a concentration of 18% on the 0,3,7,15 and 30th days produces a brick red color, solid texture and smells of oleum rosae, and Formula IV with a concentration of 22% on the 0,3,5,7,15 day produces a brick red color while the 30th day produces a brownish red color, solid texture and smells of oleum rosae.

The difference in color at each concentration is influenced by the amount of extract used, The greater the amount of extract concentration used, the darker the resulting color, but the greater the concentration of extract used causes damage to the lipstick due to the reduced amount of cera alba base used. While the brick red color change that occurred in Formula IV with a concentration of 22% on the 30th day to brownish red this was due to the difference in polarity between the sappan wood extract which was polar while the lipstick base used was nonpolar, to produce a homogeneous preparation, it is necessary to add a surfactant such as tween 80. It can be concluded that formulas I and II are better than formulas III and IV because they are more stable until the 30th day.

Based on the Figures and Tables above, the dispersion test on the formulations of Formula I, II, III and IV lipsticks with various concentrations showed the results with Good Criteria influenced by homogenous sappanwood extract so that the preparations that were applied spread evenly, did not leave fragmentation and application perfect.

Table 3. Results of the Spreadability Test for *Caesalpinia Sappan L*. Lipstick.

Formulation	Replication	Spreadability Test Category		
		Good	***Intermediate***	***Bad***
Formula I	1	✓	-	-
	2	✓	-	-
	3	✓	-	-
Formulasi II	1	✓	-	-
	2	✓	-	-
	3	✓	-	-
Formula III	1	✓	-	-
	2	✓	-	-
	3	✓	-	-
Formula IV	1	✓	-	-
	2	✓	-	-
	3	✓	-	-

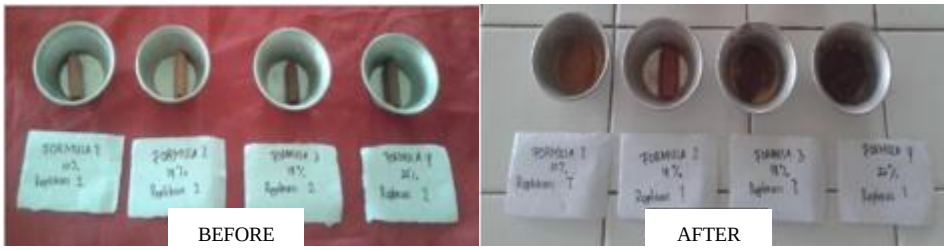


Figure 3. Melting Point Test Results of Secang Wood Lipstick

Based on the observations in Table 4, it can be seen that the best lipstick melting point is the formulation of formula II lipstick with an extract concentration of 14% which melts at an average temperature of 72°C with a melting time of 334 minutes 5 seconds. Meanwhile, 10% extract concentration melts at an average temperature of 70°C with a melting time of 300 minutes, lipstick preparations with an extract concentration of 18% melt at an average temperature of 70°C with a melting time of 304 minutes 5 seconds, Then the lipstick preparation with a concentration of 22% melts at an average temperature of 70°C with a melting time of 300 minutes.

The melting point of the preparation is influenced by the ingredients contained in the formula, where carnauba wax has a melting point of 80°C - 86°C, cetyl alcohol 45°C - 52°C, vaseline alba 38°C - 56°C, Cera alba 62°C - 65°C and -tocopherol 37°C - 41°C (15). The ideal lipstick melting temperature is actually set to a temperature close to the lip temperature, varies between 36-38°C. But because it must pay attention to the resistance factor to the surrounding weather temperature, especially the temperature of the tropics, The melting temperature of the lipstick is made higher, which is considered more suitable to be set at a temperature of about 62°C, usually in the range of 55-75°C (2,4).

Table 4. Melting Point Test Results for Lipstick Preparations *Caesalpinia sappan* L.

Formulation	Replication	Result	
		Temperature (°C)	Time (Minutes)
Formula I	1	70	300
	2	70	300
	3	70	300
Averages		**70**	**300**
Formula II	1	72	330
	2	72	330
	3	73	345
Averages		**72**	**334,5**
Formula III	1	70	300
	2	70	300
	3	71	315
Averages		**70**	**304,5**
Formula IV	1	70	300
	2	70	300
	3	70	300
Average		**70**	**330**

Figure 4. pH Test Results

The results of pH testing on lipstick preparations showed good results in the four formulas, namely using Caesalpinia sappan L. extract with a concentration of 10% was 5.8, extract concentration 14% indicates pH 5.9 extract concentration 18% indicates pH 5.7, and the extract concentration of 22% showed a pH of 5.6. The pH of the lipstick preparation is influenced by the ingredients contained in the formula, where the pH of the secang wood extract is 4.7 pH of carnauba wax 2-7 pH of cetyl alcohol 1 pH of methyl paraben 4-8 and pH of oleum ricini 6. If the pH of the lipstick is compared to the normal pH, it can be said that the pH of the preparation is appropriate and will not cause discomfort to the lips and reduce the risk of skin irritation. Based on the results of the pH test, it can be concluded that the four formulas whose physiological pH values are included in the lip skin pH range are 4.5-6.5 (4).

Table 5. pH Test Results for Lipstick Extract *Caesalpinia Sappan* L.

Formulation	Replication	Result (pH)	Averages
Formula I	1	5,8	5.8
	2	5,8	
	3	5,8	
Formula II	1	5,9	5,9
	2	5,9	
	3	5,9	
Formula III	1	5,7	5,7
	2	5,7	
	3	5,7	
Formula IV	1	5,6	5,6
	2	5,6	
	3	5,6	

Formula I with an extract concentration of 10% with a pH of 5.8, formula II with an extract concentration of 14% with a pH of 5.9, formula III with an extract concentration of 18% with a pH of 5.7 and formula IV with an extract concentration of 22% with a pH of 5.6.

Table 6. Preference Test Results for Lipstick Preparations *Caesalpinia Sappan* L. Extract.

Formulation	Category	Result			Averages
		Texture	Color	Odor	
Formula I	1	2	1	0	5%
	2	10	13	15	63,33%
	3	8	6	5	31,67%
Formula II	1	1	0	0	1,67%
	2	4	5	6	25%
	3	15	15	14	73,33%
Formula III	1	1	2	1	6,67%
	2	15	10	12	61,67%
	3	4	8	7	31,67%
Formula IV	1	12	11	3	43,33%
	2	7	6	13	43,33%
	3	1	3	4	13,33%

Score Value Assigned Category:
3: Like Very Much
2: Like Moderatly
1: Neither like nor dislike

The panelist's preference test for testing the texture, color and smell of lipstick made from sappan wood in formula II with a concentration of 14% Neither like nor dislike 1.67% Like Moderatly 25%, and Like Very Much 73.33% higher than other formulas. Formula I with a

concentration of 10% sappan wood extract, 5% Neither like nor dislike category, 63.33% Like Moderatly and 31.67% Like Very Much, formula III with 20% sappan wood extract concentration, Neither like nor dislike category 6.67% Like Moderatly 61.67% and Like Very Much 31.67%, while formula IV with a concentration of 22% sappan wood extract in the Neither like nor dislike category 43.33%, Like Moderatly 43.33% and Like Very Much 13.33% very much.

Figure 5. Patch Test Results of Secang Wood Lipstick

Table 7. Patch Test Results for Lipstick Extract *Caesalpinia Sappan* L.

Formulation	Replication	Result			
		0	+	++	+++
Formula I	1	✓	-	-	-
	2	✓	-	-	-
	3	✓	-	-	-
Formula II	1	✓	-	-	-
	2	✓	-	-	-
	3	✓	-	-	-
Formula III	1	✓	-	-	-
	2	✓	-	-	-
	3	✓	-	-	-
Formula IV	1	✓	-	-	-
	2	✓	-	-	-
	3	✓	-	-	-

Description:
(0) = Not Irritating
(+) = Red Skin
(++) = Itchy rash
(+++) = Udem
(√) = Positive
(-) = Negative

The results of the irritation test of Caesalpinia sappan L. lipstick on the skin of the inner arm showed a negative result (-) in the category of red, itchy and swollen skin in the four lipstick formulations made of sappan wood, it can be concluded that lipstick preparations from sappan wood extract are safe when used.

CONCLUSION

Based on the characteristic data of the Caesalpinia sappan L. extract lipstick formulation, the results of the organoleptic test, spreadability, melting point, pH, preference, and irritation test of the best lipstick preparation were Formula II with an extract concentration of 14%. based on the organoleptic test, it has a stable odor on day 0 to day 30, which is the smell of oleum rosae, dispersion which has good criteria, namely spreading evenly, leaving no fractures and perfect application, melting point has the highest temperature of 72°C. The resistance factor to the surrounding weather, especially the tropics, the lipstick melting temperature is made higher which ranges from 55-75°C, the preference test with the highest percentage is 73.33% and the irritation test which shows negative (-) results and does not cause irritation.

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