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Beeswax increases moisture in lipstick formulation

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Abstract. Beeswax and Carnauba waxes' mixture can potentially increase the quality of anthocyanin natural dye lipstick. This study aimed to obtain formulas of different beeswax and carnauba wax mixtures' contain. Jati leaves was extracted using maseration method. Lipsticks made from Jati leaves extract were added with variations of beeswax and carnauba waxs' concentration. Each lipstick formula was evaluated for its physical quality, such as homogeneity, melting point, smearing agent, shape, color, and odor stability test, and pH during 4 weeks storage at room temperature, and hedonic and irritation test in rabbit. Jati leaves extract contains anthocyanin proven by qualitative test result using KLT with R_f value 0,1143 and stable at pH 3. Homogenous, orange, and stable lipsticks with melting point at 73-77°C, soft, easy to apply and does not cause irritation against rabbit test animals were obtained. Lipstick's mixture was increasing along with the increasing of beeswax concentrations.

Keywords: Red, Ectraction, spectrofotometry, Carnauba wax, Beeswax

1. Introduction

Cosmetics are ingredients or preparations intended to be used on the outside of the human body (epidermis, hair, nails, lips, and external genital organs) or teeth and oral mucosa, especially for cleaning, giving perfume, changing appearance, repairing body odor, protecting and / or maintain the body in good condition .Lipstick is a solid mold based molded material that contains dissolved and / or suspended dyes that meet the criteria / requirements as a dye. Lipstick must provide a pattern that meets the criteria of fashion and market needs. Lipstick must be applied easily and evenly on the lips without causing oiliness or unpleasant taste [2].

The use of natural dyes in the formulation of lipstick preparations aims to minimize the use of harmful synthetic dyes. Natural dyes are dyes obtained from plants, animals, or from mineral sources. Anthocyanins are secondary metabolites of the flavonoids contained in plants. Anthocyanin is a color pigment that gives the colors orange, red, purple and blue [12].

Plants that contain anthocyanin dyes are quite high, one of which is teak plants (*Tectona grandis* L, f). Teak leaves (*Tectona grandis* L.f) especially young ones contain pigments of pheophiptin, β-carotene, chlorophyll and some anthocyanin derivatives, namely pelargonidin 3-glucoside, pelargonidin 3,7-diglucoside. The use of teak leaves as a coloring source can increase the economic value and use value of the leaf. The use of anthocyanin compounds in teak leaves will produce natural dyes that are safe for health and the environment [5].

The thing that attracts consumers of lipstick users is not only in terms of color but also in terms of their physical. The consistency and physical form of lipstick is influenced by the base used. Comparison of base composition plays an important role in producing quality lipstick. Carnauba Wax and Beeswax are one of the bases commonly used in lipstick preparations.

This study aims to utilize teak leaves as natural coloring in lipstick preparations and determine the effect of Carnauba Wax and Beeswax concentration variations on the characteristics of lipstick preparations.

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2. Methodology

2.1. Material

The plant material used in this research is *Tectona grandis* leaves (*Tectona grandis* L.f) obtained from the Cimaragas region, Tasikmalaya West Java.

The chemicals used are Mayer reagents, Dragendorff, Lieberman – Burchard reagents, ether, amyl alcohol, hydrochloric acid 2N, hydrochloric acid dilution, vanillin 10% in sulfuric acid conc (vanillin-sulfate), 1% gelatin, ammonia, ferric chloride 1%, ethanol 96% and citric acid 3%, carnauba wax, beeswax, lanolin, vaselin, cetyl alcohol, oleum ricini, glycol glycol, methyl paraben, propyl paraben, tween 80, butyl hydroxitoluene (BHT) and strawberry oleum from Merck.

Tool

The tools used in this study are: maserator, rotary evaporator (Eyela®), oven, distillation device, test tube (Pyrex®), drop pipette, porcelain cup, filter paper, funnel, glass beaker (Pyrex®), erlemeyer (Pyrex®), arlogi glass, stirring rod, mortar, stamper, analytical balance (Excelient®), pH meter, water tank, furnace (WiseTherm®), UV-Vis spectrophotometer (Genesys 10S®) .

2.2. Method

2.2.1. Plant determination

Plant determination is aimed at ensuring the identity of the simplicia that will be used in this study. Determination is carried out at the Biology Laboratory of the Faculty of Mathematics and Natural Sciences, Padjajaran University.

2.2.2. Pigmen extraction

The process of processing the first *Tectona grandis* leaves simplicia was carried out by sorting, namely washing the *Tectona grandis* leaves using aqudest until clean. The leaves are clean and then dried using an oven with a temperature of 40-60ºC to dry which is marked with leaves easily broken. The next process is a refining process using a blender, and finally sieving with 50 mesh to obtain a fine simplicia powder.

2.2.3. Qualitative test of Tectona grandis leaf extract

The qualitative test of the extract aims to determine the chromatogram pattern and to determine the components of the compounds contained in the ethanol extract of teak leaves (*Tectona grandis* L.f). Where the principle of thin layer chromatography is the separation of compounds that occurs due to differences in the absorption capacity of the compound to the adsorbent and its solubility in the elution liquid. In this thin layer chromatography, the stationary phase used is silica gel GF_{254} and the mobile phase of acetone: methanol: isopropyl alcohol with a ratio of 5: 4: 1 [4].

2.2.4. Tectona grandis leaf extract

The color extraction process of teak leaves is done by maceration method. The simplicia powder was soaked with 96% ethanol with the addition of 3% citric acid solution. The addition of acid in this extraction process aims to make the extracted stable anthocyanin compounds red. Because anthocyanin compounds will be red if in an acidic atmosphere. The extraction process is carried out for 3 days with solvent replacement every 1x24 hours to prevent solvent saturation.

After obtaining the liquid extract, then concentrated using a rotary evaporator at a temperature of 60ºC until a thick extract is obtained [3].

2.2.5. Stability test for Tectona grandis leaf extract dyes

Dyestuff stability test from teak leaf extract aims to determine the stability of the dyes to changes in pH and temperature. Before testing the extract, it was first determined the maximum wavelength of the extract by dissolving the extract in 96% ethanol with a concentration of 500 ppm then measured at a wavelength of 400-800 nm. pH stability test was carried out on pH 2, 3, 5 and 7 with a time range of 0 minutes, 15 minutes, 30 minutes, 45 minutes and 60 minutes. While the temperature stability test was carried out at room temperature, 50ºC, 75ºC, 100ºC, and 120ºC. Stability seen from changes in absorbance values measured using UV-Vis spectrophotometry.

2.2.6. Formulation of lipstick

In making these lipstick preparations, the ingredients are divided into 2, namely mixture A and mixture B. Mixture A is made by dissolving methyl paraben and propyl paraben into propylene glycol, after dissolving then adding thick teak leaf extract until homogeneously mixed. Add butyl hydroxylolene which has been dissolved in the oleum ricini in the first mixture and stir until homogeneous. Mix B is made by melting the wax base which consists of carnauba wax, beeswax, vaselin, lanolin, and cetyl alcohol on a water bath until it fuses perfectly. A mixture and B mixture are mixed slowly, then added tween 80 and strawberry oleum, stirring until homogeneous. In a liquid state the mixture is put into a mold and left to freeze. After freezing the mass is removed from the mold and put in a container [9].

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Table 1. Formulation of lipstick

Composition	Dosage in (%)		
	Formula 1	Formula 2	Formula 3
Tectona grandis leaf extraction	15	15	15
Carnauba wax	20	15	10
Beeswax	10	15	20
Oleum jarak	6,8	6,8	6,8
Lanolin	10,3	10,3	10,3
Vaselin	28,6	28,6	28,6
Cetyl alcohol	8	8	8
Propilenglikol	5	5	5
Tween 80	1	1	1
Butil hydroksitoluen	0,1	0,1	0,1
Metyl paraben	0,18	0,18	0,18
Propyl paraben	0,02	0,02	0,02
Oleum Strawberry	Qs	Qs	Qs

2.2.7. Evaluation of lipstick formulation

2.2.7.1. Organoleptic test

Organoleptic tests of lipstick preparations include visual observation of the physical appearance of the preparation including texture, aroma, and color [1].

2.2.7.2. Homogeneity test

Homogeneity test is done by applying a certain amount of preparation to a transparent glass. Preparations must show a homogeneous arrangement and there is no visible grain of roughness [9].

2.2.7.3. Test the melting point

Lipstick is put in an oven with an initial temperature of 50°C for 15 minutes, observed whether the lipstick melts or not, then the temperature is raised by 1°C every 15 minutes and observed at what temperature the lipstick starts to melt [9]. Good lipstick requirements have a melting point of 50-70°C.

2.2.7.4. Smear test

The topical test is done visually by applying lipstick to the back skin of the hand and then observing the number of colors attached to the 5 times the treatment. Lipstick preparations are said to have good smearing power if the color is attached to the back skin of the hand with a visible and even color with several times of application [9].

2.2.7.5. pH test

pH checks are carried out using a pH meter. The pH meter tool that will be used before is calibrated first by using a standard neutral buffer solution (pH 7.01) and an acid pH buffer solution (pH 4.01) until the device shows the pH price. Then the electrode is washed with distilled water, then dried with a tissue. Samples were made in 1% concentration, namely by weighing 1 gram of preparation and melting in Beaker glass with 100 ml of distilled water on a water bath. After cold then the electrode is dipped in the solution. Let the tool show the pH price to be constant. The figure shown by the pH meter is a preparation. Determination of pH was carried out three times on each preparation. Good lipstick requirements have a pH of 4.5-7.5 (SNI 16-4769-1998).

2.2.7.6. Irritation Test

In the primary skin irritation test, rabbit test animals were used, the test was carried out by means of the test compound applied to the skin of the test animal that had previously been shaved. The skin reaction to the test compound is then observed and recorded at certain intervals (minimum 3 days). The irritation observed was the presence of erythema and edema in the tissues [6.7].

2.2.7.7. Hedonic Test

This test was conducted to determine the level of panelists' preference for the preparation of lipstick made. Visual test of preference for 30 panelists with criteria used were women, aged 20 years and over, did not have sentitive or allergic skin. Observations are made on texture, odor, color and smear.

3. Results and discussion

3.1. Determination

The results of the determination carried out at the Biology Laboratory of the Faculty of Mathematics and Natural Sciences, Padjajaran University, Bandung showed that the plants used in this study were true teak leaves (*Tectona grandis* L f).

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3.2. Qualitative test of ethanol extract of teak leaves

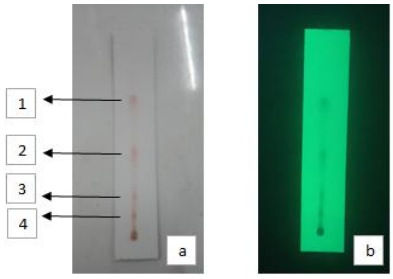

Fig 1. Thin layer chromatogram of teak leaf ethanol extract. a. seen directly, b. seen in UV lamp λ 254 nm.

Based on the results of thin layer chromatography, it can be seen that the target compound, anthocyanin, is found in spots 4 with a Rf value of 0.1143. This is in accordance with the literature which states that the range of Rf values for anthocyanins is 0.06-0.12 [4].

3.3. Extraction

From the results of this maceration, a liquid extract of teak leaves was obtained which was then concentrated with a rotary evaporator until a thick extract of teak leaves was obtained. In this extraction process, extract yield of 30,649% was obtained.

3.4. Stability test for teak leaf extract dyes

3.4.1. Determination of Maximum Wavelength

In determining the maximum wavelength of teak leaf extract a maximum wavelength of 479 nm was obtained with an absorbance value of 0.676. This shows that the ethanol extract of teak leaves containing anthocyanins according to the literature has a wavelength of 475-550 nm [1].

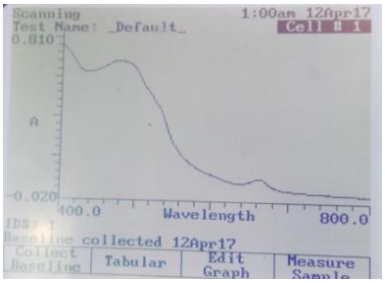

Fig 2. The λ maximum curve of Teak Leaf Extract

3.4.2. Stability Test Against pH

Based on the pH stability test results obtained that the extract was stable at pH 3 because the standard deviation was the smallest. Where the smaller the standard deviation, the sample becomes more constant because the difference in the absorbance value gets smaller. Whereas based on the results of the temperature stability test results obtained that the higher the heating temperature, the lower the absorbance value of the sample due to the bleaching of the color due to heating. Then the extract is most stable at room temperature (27°C) [10].

Table 2.Test results of the Extract Stability Test on pH

pH	Absorbance					Standard Deviation
	0’	15’	30’	45’	60’	
2	0,534	0,538	0,538	0,537	0,543	0,0032
3	0,525	0,532	0,531	0,528	0,527	0,0029
5	0,534	0,538	0,543	0,536	0,535	0,0036
7	0,687	0,785	0,806	0,825	0,835	0,0594

3.4.3. Stability Test Against Temperature

Temperature stability tests were carried out at room temperature, 50°C, 75°C, 100°C and 120°C with a heating time of 15 minutes. This temperature stability test aims to determine the stability of the dye against changes in temperature where this stability affects the quality of the preparation during storage.

Table 3. Test results of the Extract Stability Test on Temperature

Temperature	Absorbance
Room temperature(27°C)	0,233
50°C	0,226
75°C	0,212
100°C	0,211
120°C	0,198

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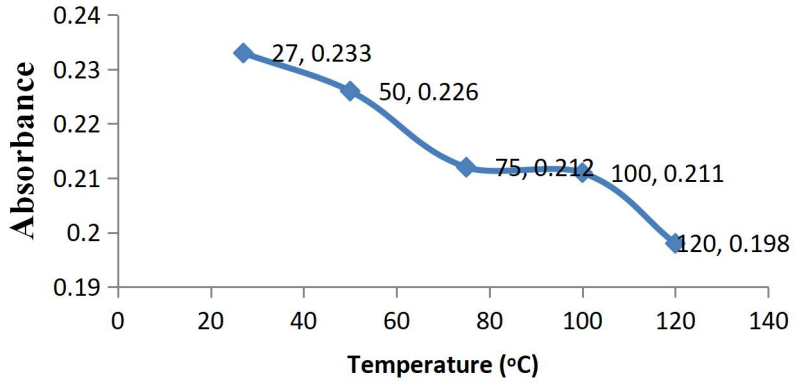

Fig 3. Absorbance curve to the influence of temperature

Based on Figure 3 shows that the higher the heating temperature, the lower the absorbance or stability of the color so that the red color will fade. This decrease in absorbance is caused by damage to the chromophore pigment group in anthocyanins due to pigment degradation and polymerization. The color degradation in anthocyanins is caused by the red flavillium cation changes into carbinol bases and eventually becomes colorless chalcone which ends in brown-colored degradation products [10].

3.5. Formulation of lipstick preparations

In determining the amount of teak leaf extract that will be used beforehand, first optimization is carried out with the results obtained by 15% extract concentration to produce the best color and texture of the lipstick. The difference in concentration between carnauba wax and beeswax of the three formulas produces different characteristics of lipstick. In formulation 1 produced lipstick that is hard, dry, non-sticky and has a good smear. Then in formula 2 it produces a hard lipstick, a little sticky but the smear is bad. While the formula 3 produces lipstick that is not so hard, sticky and has good smear.

Fig 3. Preparation of teak leaf extract lipstick

3.6. Evaluation of lipstick preparations

3.6.1. Organoleptic test

Organoleptic test results showed that formula 1 was dark orange, formula 2 and 3 were orange, the three formulas had a solid texture with strawberry smell. After 4 weeks of observation, the texture, color and smell of the three formulas were stable.

3.6.2. Homogeneity test

The homogeneity test results indicate that the three lipstick preparations do not show any coarse particles when applied to transparent glass. This shows that the preparation is homogeneous and meets the requirements [9].

3.6.3. Test the melting point

Melting point examination results showed that formula 1 had a melting point of 77ºC, formula 2 had a melting point of 76ºC and formula 3 had a melting point of 73ºC. From these results it can be seen that the greater the concentration of carnauba wax, the higher the melting point. These results have met the requirements according to [11]. which says that a good melting point of lipstick is> 50ºC.

3.6.4. Smear test

Formula 1 has a harder texture so that when applied it gives color but is dry so it requires more pressure when applying. Formula 3 has a texture that is not too hard so it is easier to apply and gives a moist and softer impression when applied. While the formula 2 has a texture that is not too hard but the color is difficult to stick when applied, so the formula 2 gives a more faded color.

3.6.5. pH test

Based on the pH test results, it can be seen that all three formulas have a pH of 6 during 4 weeks of storage. It shows that formula 1, 2 and 3 is stable because there is no change in pH during storage. This shows that the preparation meets the pH requirements of the lipstick which has been determined by SNI, namely 4.5-7.5 (SNI 16-4769-1998).

3.6.6. Skin Irritation test

The results of the irritation test showed that during 72 hours of testing on rabbit test animals, the three non-

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irritating lipstick preparation formulas were erythema and edema of rabbit test animals so that lipstick preparations could be said to be safe to use [7].

3.6.7. Hedonic test

From the test results of Friedman test, it was found that in terms of the texture formula 2 panelists liked the most with a mean rank of 2.27, in terms of the formula 3 odor the panelists liked the most with the mean rank of 2.15, in terms of the color of the formula 3 panelists preferred the value mean rank 2.50 and in terms of the formula 3 smudge panelists are most preferred with a mean rank of 2.17.

4. Conclusion

Teak leaf extract can be used as a dye in lipstick preparations. The concentration of teak leaf extract that gives the best color in the preparation is 15%. The different concentrations of carnauba wax and beeswax affect the texture of the lipstick preparation. The greater the concentration of carnauba wax, the harder the texture of the preparation.

Physical quality inspection results showed that all three formula preparations were stable in storage for 4 weeks, did not show any changes in shape, color, odor, pH, good homogeneity and did not cause irritation.

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