The Utilization of Secang Wood (*Caesalpinia sappan* L.) Extract Nanoemulsion as Natural Pigment in Lip Cream Preparations

Eriska Agustin*1,2, Aknes Pratiwi1

Faculty of Pharmacy, University of Kader Bangsa, Palembang, Indonesia

**Abstract**

Lip cream is a semi-solid lipstick preparation. One of the important ingredients in lipcream preparations is a variety of colorants. Secang wood contains the active compound brazilin which gives it a yellow to purple color which can be used as a natural dye. The conventional lip cream preparations from extracts have low homogeneity and unstable, so it is necessary to increase the stability of the preparation by making a nanoemulsion of secang wood extract which is incorporated into the lip cream preparation. The preparation of 10% secang wood extract nanoemulsion preparation using Tween 80, Virgin Coconut Oil (VCO), and PEG 400 with particle size reduction using a bath sonicator. The concentration of the nanoemulsion preparation was 1% (F3) and 3% (F4) incorporated into the lip cream base and conventional lip cream preparations with the same concentration (F1 and F2). The test results for the size of the 10% secang wood extract nanoemulsion globules were 127.54 ± 0.45 nm (PI: 0.3 ± 0.013). The test results for the F3 and F4 nanoemulsion lipcream preparations had a pink to purple color, pH 6-6.4, spreadability of 5.9-6.8 cm, adhesion of 5.5-6 seconds, the preparation was homogeneous and non-irritating, compared with conventional lip cream preparations F1 and F2 are not homogeneous, the color of the lip cream is pake, and the irritating properties are very mild in both formulas. The conclusion of this research is that secang wood extract can be formulated into a nanoemulsion preparation and incorporated into a lip cream preparation.

**Keywords:** Brazilin, Color, Dye, Sonicator Bath

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**INTRODUCTION**

Cosmetics are materials or preparations used for use on the external parts of the human body (skin, hair, lips, nails and external genital organs) specifically used to beautify, perfume, clean, eliminate body odor or maintain good body condition. One of the cosmetics intended to beautify the lips is lip cream. Lip cream is a semi-solid lipstick preparation, where the most important ingredient used is a coloring agent. Coloring agents are a determining factor in cosmetic preparations, especially for lip use. The use of synthetic or chemical dyes such as Rhodamine B can cause side effects if used in food or cosmetics, one of which can cause cancer and impaired liver function if exposed in large quantities.

The use of alternative dyes for the problems above is the use of natural dyes contained in secang wood (*Caesalpinia sappan* L.). The red color produced by secang wood comes from an active compound called brazilin. Natural ingredients have low side effects, in fact there are no dangerous side effects as long as they are not used excessively. The brazilin compound found in secang wood has high levels of antioxidants and anthocyanins which are good for use as natural dyes. The formulation of lip cream preparations in nanoemulsion form aims to develop the formulation so that the preparation can be more homogeneous and absorb the active ingredients more optimally. In addition, making nanoemulsion preparations can increase the stability of the preparation because the active ingredients are protected from the nanoemulsion base when in contact with the lip cream base. Research conducted by Rusdi (2017) examined the manufacture of nanoemulsion preparations of secang wood extract as a coloring agent with a comparison of various emulsifiers producing particle sizes below 100 nm and a polydispersity index of 0.187-0.782.

Cosmetics with nanoemulsion size have advantages compared to conventional preparations, where nanoemulsion has a transparent color and has a very small size between 20-500 nm. Nanoemulsion preparations have long-term stability. Therefore, in this research, optimization of the manufacture of secang wood extract nanoemulsion which is incorporated into lip cream preparations will be carried out and evaluation tests will be carried out on the preparations.

**MATERIAL AND METHODS**

1. Materials and Tools

The materials used are seccang wood (*Caesalpinia sappan* L.) (Central Java), Ethanol pa (Bragaco), Bees Wax (Bragaco), White Wax (Bragaco), Cetyl Alcohol (Bragaco), Kaolin (Bragaco), Dimetichone (Bragaco), Propyl paraben (Bragaco),...
Methyl paraben (Brataco), Titanium Dioxide (Brataco), Castor Oil (Brataco), Tween 80 (Brataco), PEG 400 (Brataco), and VCO (Brataco).

The tools used in this study include an analytical balance (Mettler Toledo® XS205), Hekdoph® rotary evaporator RV 10 (Germany), magnetic stirrer (IKA® RW 20 Digital, Germany), sonicator bath (Nagoya S Ultrasonic Cleaner GB-928), pH meter (Mettler Toledo® S20), Particle Size Analyzer (Delsa™ Nano C Particle Analyzer, Beckman Coulter, USA), and laboratory glassware.

2. Preparation of Secang Wood Extract

The secang wood extraction process is carried out using the maceration method. Secang wood powder was extracted using ethanol pa with a ratio of 1:10 for 3x24 hours (3 cycles). Maceration is carried out by soaking the simplicia in ethanol solvent and stirring continuously every day for 10 minutes. The filtrate is separated from the residue and stored in a container, then prepared using a rotary evaporator at temperature 70°C until a solid extract is obtained.

3. Preparation of Nanoemulsion Sappan Wood Extract

Nanoemulsion formulation is carried out by weighing all the ingredients. thick extracts of secang wood, tween 80, and VCO were mixed in a 50 ml beaker glass and homogenized with a magnetic stirrer at a speed of ±500 rpm for 15 minutes. Next, PEG 400 was added to the mixture and homogenized again with a magnetic stirrer at a speed of ±500 rpm for 30 minutes.

4. Preparation of Lip Cream

Lipcream preparations are made by weighing the ingredients first. The oil phase ingredients were melted at a temperature of 68°C in a cup, then transferred to a hot mortar, kaolin was added and crushed, then the secang wood extract was added with TiO2 and stirred until homogeneous. Then, after the mixture has all been cooled, methyl paraben and propyl paraben are added to the mixture, then crushed until everything is evenly distributed, then transferred to a lip cream container.

Table 1: Formulation of Secang Wood Nanoemulsion

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Concentration (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Base</td>
</tr>
<tr>
<td>Secang wood extract</td>
<td>-</td>
</tr>
<tr>
<td>Tween 80</td>
<td>15</td>
</tr>
<tr>
<td>VCO</td>
<td>4</td>
</tr>
<tr>
<td>PEG 400</td>
<td>10</td>
</tr>
<tr>
<td>Aquadest</td>
<td>ad 100</td>
</tr>
</tbody>
</table>

The reduction of the globule size of the mixture was carried out using a bath type sonicator for approximately 60 minutes. After the nanoemulsion is formed, the nanoemulsion is diluted with distilled water, stirred little by little using a magnetic stirrer for ±15 minutes until a nanoemulsion is formed.

Table 2: Formulation of Lip cream preparations

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>F0</th>
<th>F1</th>
<th>F2</th>
<th>F3</th>
<th>F4</th>
</tr>
</thead>
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<tr>
<td>Secang Wood Extract</td>
<td>-</td>
<td>1</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Secang Wood Nanoemulsion</td>
<td>-</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>White wax</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>Bees wax</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Cethyl alcohol</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Dimethicone</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Titanium dioxide</td>
<td>0,5</td>
<td>0,5</td>
<td>0,5</td>
<td>0,5</td>
<td>0,5</td>
</tr>
<tr>
<td>Caolin</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Propyl Paraben</td>
<td>0,3</td>
<td>0,3</td>
<td>0,3</td>
<td>0,3</td>
<td>0,3</td>
</tr>
<tr>
<td>Methyl Paraben</td>
<td>0,1</td>
<td>0,1</td>
<td>0,1</td>
<td>0,1</td>
<td>0,1</td>
</tr>
<tr>
<td>Castor Oil</td>
<td>ad 100</td>
<td>ad 100</td>
<td>ad 100</td>
<td>ad 100</td>
<td>ad 100</td>
</tr>
</tbody>
</table>

5. Evaluation of Lip cream Preparations

- Particle Size and Polidispersity Index

In this study, evaluation of the physical quality of cinnamon extract nanoemulsions was carried out, including the size of the nanoemulsion globules using the Particle Size Analyzer (PSA). The sample is put into the sample cuvette up to the specified mark and it is ensured that there are no bubbles. Samples were read 3 times to ensure consistent values.

- Organoleptic test

Organoleptic observation is an objective observation of the texture, color and aroma of lip cream products. Organoleptic tests are carried out by observing the texture, color and aroma of the product.

- pH test

The pH test is carried out to determine the additivity and alkalinity of a preparation. In human lip skin, the physiological pH range is 4.0-6.0. The pH test was carried out with a pH meter. First, the pH meter was calibrated using a neutral buffer solution (pH 7.0), an acid buffer solution (pH 4.0), and a basic buffer solution (pH 9.0). The test sample is made in a concentration of 1%, then the electrode is dipped in the sample and let the tool show the pH number until it is constant. The test was carried out with three replicas for each formula.
 Homogeneity Test

Homogeneity test is a method used to find out whether the mixing of each component is good. This test is carried out using transparent glass by smearing the preparation on it, then covering it with another glass deck. The preparation is said to be homogeneous if there are no rough spots on the transparent glass6.

Spreadability Test

Spreadability testing was carried out to evaluate the speed at which lipcream spreads when used so that its application to the lips becomes easier. The spreadability test was carried out by preparing a sample of 0.5 g of lipcream on a glass that had been coated with graph paper, then placing a petri dish on it for 60 seconds. The results of his observations by calculating the area of the area given the preparation. Then, the formula is placed on top of another glass, then given a load weighing 200 g and left for 60 seconds and the diameter of the spread is calculated11.

Stickiness Test

The adhesion of the preparation is evaluated to see the duration required for the preparation to adhere to the lips. The higher the stickiness response value, the longer the time needed for the lipcream to stick. The spreadability test was carried out by weighing 0.5 g of the formula and then smearing it over an object glass whose area was known, then placing another glass object on top of it and pressing it with a 50 g load for 5 minutes. The object glass was installed on the test equipment and given a load of 20 g and then the time was recorded until the two object glasses separated13.

Irritation Test

The irritation test was carried out on healthy rabbits weighing 2-2.5 kg. Before testing, rabbits were acclimatized in cages for 5-7 days. Rabbits had their back fur shaved 24 hours before testing to an area of approximately 10x15 cm. Then the rabbit’s back is divided into 4 areas measuring 2x3 cm. The test was carried out on 3 rabbits. Before treatment, the test area was cleaned with NaCl. The lipcream preparation is given by smearing it on the test area. After applying the preparation, the rabbit’s back is covered with a non-reactive bandage. After 24 hours, the bandage was removed and the test area was cleaned with water to remove any lip cream residue. The test area was examined and observed at 24, 48, and 72 hours. Each test preparation was calculated for the irritation index9.

RESULT AND DISCUSSION

In this research, the process of making 500 g of extract was carried out using the maceration method with 5 liters of ethanol (1:10) for 3 cycles. The result of the thick extract obtained was 70 g and the % yield was 14%. The use of ethanol as a solvent was chosen because of its ability to filter from non-polar to polar polarities. The brazилиn compound is a compound that gives secang wood a red color with solubility that is easily dissolved in ethanol. The brazilin compound will oxidize to produce a braziliin compound which is brownish red in color, so the cold extraction method is chosen, namely the maceration method12.

The preparation of 10% secang wood extract nanoemulsion preparation was carried out using a mixture of emulsifier, co-emulgator, and water phase. Based on the results of testing the globule size and polydispersity index, the secang wood extract nanoemulsion produced a globule size of 127.54 ± 0.45 nm. (P<0.3±0.013). The globule size results obtained were less than 100 nm. This is due to the correct composition of the emulsifier and co-emulgator used and the process of making the nanoemulsion which begins with low speed homogenization using a magnetic stirrer and then the globule size reduction process is carried out using a bath sonicator10.

The use of VCO can disperse secang wood extract which can reduce the surface tension between the oil phase and the water phase. The use of PEG 400 as an emulsifier and co-emulgator can reduce the amount of energy needed to destroy globules by filling the surface gaps of the particles to make them denser, which will help the tween 80 emulgator reduce interfacial tension16. The polydispersity index value indicates the uniformity of globule distribution in the preparation. The polydispersity index results obtained were 0.3 ± 0.013, indicating a high level of globule distribution uniformity (<0.5) and it was assumed that it would be physically stable in long-term storage. This polydispersity index test correlates with globule size, the smaller the globule size will provide high globule distribution uniformity. The use of a bath type sonicator that utilizes mechanical vibrations resulting from ultrasonic waves causes cavities which will later generate high energy which will make the globule size become nanometer sized8. In this research, a 10% secang wood extract nanoemulsion preparation was incorporated into a lip cream preparation.

Lipcream preparations were made using nanoemulsion of secang wood extract (NE-KS) and conventional secang wood extract without making nanoemulsion (Eks-KS) with varying concentrations of 1% and 3% respectively. The evaluation of lip cream preparations carried out included organoleptic tests, spreadability, adhesive power, spreadability, pH test, homogeneity test, irritation test and stability test.

Based on the results of organoleptic tests on nanoemulsion and conventional lip cream preparations, the difference in the concentration of the added extract makes the color also different13. The lip cream preparation made has a semi-solid texture and a distinctive aroma of secang wood. In conventional lip cream preparations (F1 and F2), the lip cream color looks paler compared to nanoemulsion lip cream preparations (F3 and F4) which produce a purplish pink color. The higher the concentration of the extract, the more purplish
the lip cream color produced. The purplish color produced in the nanoemulsion lip cream preparation is due to the high homogeneity of the results of making the sappan wood extract nanoemulsion, so that the color distribution is homogeneous and the resulting color is more even, compared to conventional lip cream preparations which are paler in color and have an uneven color from the cup tree extract.

The spreadability test aims to determine the ease with which a preparation is evenly distributed and spread over the skin. The spreadability test results of nanoemulsion lip cream preparations F3 and F4 had a greater spreadability ranging from 5.9 and 6.8 cm compared to conventional lip cream preparations F1 and F2 ranging from 5.3 and 6.4. The higher the concentration of the extract, the lower its ability to spread because the consistency of the preparation is thicker. The lower the viscosity of a preparation, the greater the distribution will be so that the contact between the active substance and the skin will be wider and the absorption of color into the skin will be even. Overall, the lip cream preparations show that nanoemulsion and conventional lip cream preparations have a value of more than 1 second, so it can be said that the preparations are good.

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Type of Testing (average)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>pH</td>
</tr>
<tr>
<td>F1 1% Convensional Lipcream</td>
<td>5.2</td>
</tr>
<tr>
<td>F2 3% Convensional Lipcream</td>
<td>6.1</td>
</tr>
<tr>
<td>F3 1% Nanoemulsion Lipcream</td>
<td>6</td>
</tr>
<tr>
<td>F4 3% Nanoemulsion Lipcream</td>
<td>6.4</td>
</tr>
</tbody>
</table>

Homogeneity testing aims to see the shape of the lip cream which is homogeneous between the base and extract. The homogeneity test results showed that the nanoemulsion lip cream preparation had no coarse grains when applied to the glass object and the color was even. The conventional lip cream preparations F1 and F2 showed that there were coarse grains that were not homogeneous when applied to a glass object and the color of the sappan wood extract was uneven. Nanoemulsion lip cream preparations are said to be more homogeneous because the process of making secang wood extract with a homogeneous and stable nanoemulsion base, whereas conventional lip cream preparations of sappan wood extract are directly included in the lip cream base. The pH check of the lip cream preparation is carried out to see the stability of the preparation. The test results showed that the nanoemulsion lip cream preparations F3 and F4 had pH values of 6 and 6.4 respectively, while the conventional lip cream preparations F1 and F2 had pH values of 5.2 and 6.1 respectively, where the secang wood extract had a pH of 4.8. The difference in pH value is due to differences in the concentration of sappan wood extract in the preparation which increases and the addition of ingredients in the base which apparently affects the pH value. The lip cream preparations made have a pH value that is still within safe limits for the pH of topical preparations, namely 4.5-7.

Testing for the irritating properties of the preparation was carried out to determine whether the preparation irritated the skin or not with acute irritating properties within 24-72 hours in albino rabbits. The results of the irritation test showed an irritation index value of 0 for the nanoemulsion lip cream preparation, which means it does not irritate the skin, while the conventional F2 lip cream preparation has an irritation index value of 0.33, which means very mild irritation. This shows that the use of extract concentrations of 1% and 3% in making secang wood extract nanoemulsion does not irritate the skin, and the composition of the additional ingredients is still within the permitted range for application to the skin. It can also be seen in testing the pH of the preparation which shows that the pH value still enters the skin's pH range.

In this research, it can be said that the manufacture of secang wood extract nanoemulsion which is incorporated into lip cream preparations has homogeneity and even color, as well as preparation evaluations that meet the requirements when compared with conventional lip cream preparations.

**CONCLUSIONS**

In this research, secang wood extract can be formulated in a lip cream preparation with the size of the secang wood extract nanoemulsion globules being 127.54 ± 0.45 nm (PI: 0.3 ± 0.013). The F3 and F4 nanoemulsion lip cream preparations have a purplish color, semi-solid texture, typical secang wood aroma, good spreadability and adhesion, a pH that is within the pH range of topical preparations, the preparation is homogeneous, and does not irritate the skin. Meanwhile, conventional lip cream preparations have a paler color, good adhesion and spreadability, but are not homogeneous, and the preparation is very mildly irritating.

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**Conflict of Interest**

The authors declare no conflict of interest.
REFERENCES


