Lip Balm Formulation Based on Balinese Grape seed Oil

(Vitis vinifera l. Var Alphonso Lavallee)

Pande Ayu Naya Kasih Permatananda¹, Desak Putu Citra Udiyani², I Gede Suranaya Pandit³

¹²Faculty of Medicine and Health Science, Universitas Warmadewa, Indonesia
³Faculty of Agriculture, Universitas Warmadewa, Indonesia

ABSTRACT: Lip balm is defined as a cosmetic formulation that is applied to the lips to prevent dryness of the lips and protect the lips from foreign bodies, which makes lip balms different from lipsticks. There are many plant oils that can be used in making moisturizers, including lip balm, one of which is grapeseed oil. Although less popular with the public, Balinese grape has a higher flavonoid content than other grapes. The purpose of this study was to create a lip balm product derived from Balinese grape seed oil and find the best concentration of Balinese grape seed oil to create a lip balm product. The research method used in this research is experimental. Making lip balm preparations based on Balinese grape seed oil with various concentrations of 20%, 30%, 40%, 50%, 60%, and 70%. The ingredients used are Balinese grapeseed oil, lanolin, beeswax, propylene glycol and essential oils (perfume). Evaluation of lip balm preparations, namely homogeneity test, stability test carried out for three months at room temperature by observing changes in color, odor and dosage form, pH test, smear test, irritation test, and testing the ability of lip balm preparations to moisturize lips on the tested volunteers by using the preparation every day before going to bed and then measuring the moisture content of the lips every week until the fourth week using a skin analyzer. The result of this study is that grapeseed oil has the potential to be developed into cosmetic products, especially lip balm with the best concentration is 70%.

KEYWORDS: Balinese grape, Bali, Grapeseed oil, Herbal, Lip balm

INTRODUCTION

Lips are a sensitive part of the face. Unlike skin which has melanin to protect it from the sun, lips have no protection. Therefore, when the air is too hot or too cold, the lips can become dry and cracked. Apart from being unsightly, chapped lips also cause pain and discomfort [1]. One of the effective ways used by the public, especially women, to treat dry lips is by using lip balm.

Lip balm is defined as a cosmetic formulation that is applied to the lips to prevent dryness of the lips and protect the lips from foreign bodies, which makes lip balms different from lipsticks. To formulate a lip balm, it is very important to maintain a balanced concentration of the main ingredients such as butter, oil, and wax, so that the final product can be a harmonious combination achieved at a point between 65° to 75°. The resulting formulation may differ depending on the proportion of wax, oil, and pigment. Long-lasting products can be produced from a high proportion of waxes and pigments, otherwise a smoother lipstick or lip balm would be produced, which in the end the contact between these products and the skin will not produce a cracked or dry sensation, and lipstick or lip balm products should be able to form homogeneous layer on the lips to protect the lip mucosa from environmental factors such as radiation, dryness, and pollution [2,3].

Oil plays an important role in the manufacture of moisturizing cosmetics because it can form a thin layer on the surface of the skin so that it can prevent evaporation of water from the skin surface caused by the heat of the sun. Cosmetics that are added to a mixture of oils, such as plant oils that are easier to mix with skin fat, are better able to penetrate the stratum corneum cells, and have stronger adhesion [4]. There are many plant oils that can be used in making moisturizers, including lip balm, one of which is grapeseed oil. From research conducted by Hasan FA et al., in 2018 it was found that grapeseed oil can produce homogeneous lip balm preparations and can increase the water content of the lips [5].

Grapeseed oil is obtained from the seeds of grapes. Grapeseed oil generally contains 75% linoleic acid, 15% oleic acid, 6% palmitic acid, 3% stearic acid and 1% linolenic acid. The antioxidat properties of compounds contained in grapeseed oil and high unsaturated fatty acids from grapeseed oil make grapeseed oil more resistant and not easily oxidized, so it is often used in the cosmetic industry, culinary pharmaceuticals and other health purposes [6]. Balinese grapes that have the scientific name of Vitis vinifera L. var. Alphonso Lavallee is one of the superior fruits of the island of Bali. This grape is cultivated in Buleleng Regency in three sub-
districts, namely Seririt, Gerokgak and Banjar. This type of grape has fruit with purplish black color and belongs to the black variety (black grape variety). This Balinese grape is also known as 'Ribier' and is often used as fresh fruit (table grape) or wine (wine grape) [7]. A previous survey study found that Balinese grapes are less desirable than imported wines even though they are sold at lower prices. The lack of sweetness, the small size of the fruit and the presence of seeds are some of the weaknesses in the quality of Balinese grapes and the cause of their lack of marketability. However, the results of research by Nile et al in 2013 found that grapes of the Alphonso Lavallee variety (Balinese grapes) contained more flavonoids than grapes from other black varieties such as Flouxa, Black Pegaru, Concord, Campbell Early, and Spherper [7,8], which makes Balinese grapes have great potential to be used in the health sector as herbal alternative [9]. Seeing this also, researchers are interested in developing grapeseed oil from Balinese grapes to make lip formulations that can be used not only to maintain healthy lips, but also to support women's beauty [10].

RESEARCH METHOD

This research method is experimental. The research includes sample identification, physical quality inspection of the preparation including homogeneity test, stability test, pH measurement, smear test, skin irritation test, and testing the ability of the preparation to moisturize lips using a skin analyzer.

A. Time and Place of Research

Research will be conducted at the Biomedical Laboratory, Faculty of Medicine, Warmadewa University and took time about 6 months from the time this proposal is approved.

B. Tools and Materials

B.1 Tools

The tools used include: oil pressor machine, digital balance, water bath, stirring rod, spatula, tissue, object glass, evaporating dish, dropper pipette, small plastic pot, pH meter, mortar, pestle and necessary glassware, and portable skin analyzer

B.2 Materials

The materials used in this study were beeswax, aquadest, lanolin/shea butter, propylene glycol, and balinese grapeseed oil

B.3 Participants/Volunteers

Criteria for volunteers for the panel are as follows:
1. Healthy woman
2. There is no history of disease related to allergies.
3. Willing to volunteer.

B.4 Working Procedure

B.4.1 Making the Balinese Grape Oil

500 grams of dried Balinese grapeseed is put into an oil press machine to get 50 cc of grapeseed oil.

B.4.2 Lip Balm Formulation

Based on the literature review, a basic formulation for making the Balinese grapeseed oil moisturizer was made which consists of 3 main components, namely emollient, occlusive, and humectant ingredients.

B.4.3 Procedure for Making Lip Balm

Method of preparation:
1. Grapeseed oil was weighed in a cup.
2. Beeswax, lanolin, propylene glycol had been weighed and put in a separate dish, then melted in a water bath.
3. Grapeseed oil was slowly mixed in a cup while stirring and heated.
4. The cup was lifted from the top of the bath.
5. Perfume was added, stirred until homogeneous.
6. The preparation was then left until it was not too hot and then put it in a small pot and allowed to freeze.

Based on the above formula, modification of lip balm based on grapeseed oil formula was carried out in various comparisons. This was done to get a good lip moisturizer base in texture, consistency, and ability to moisturize the lips, which can be seen in table I.
Table 1. Lip Balm Dosage Forms with Various Concentration of Grapeseed Oil

<table>
<thead>
<tr>
<th>Formulation</th>
<th>Grapeseed extract (gr)</th>
<th>Lanolin (gr)</th>
<th>Beeswax (gr)</th>
<th>Propylene Glycol (gr)</th>
<th>Base (gr)</th>
<th>Total Formulation (gr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>20</td>
<td>21,02</td>
<td>57,03</td>
<td>1,95</td>
<td>80</td>
<td>100</td>
</tr>
<tr>
<td>F2</td>
<td>30</td>
<td>18,39</td>
<td>49,90</td>
<td>1,70</td>
<td>70</td>
<td>100</td>
</tr>
<tr>
<td>F3</td>
<td>40</td>
<td>15,76</td>
<td>42,77</td>
<td>1,45</td>
<td>60</td>
<td>100</td>
</tr>
<tr>
<td>F4</td>
<td>50</td>
<td>13,14</td>
<td>35,64</td>
<td>1,22</td>
<td>50</td>
<td>100</td>
</tr>
<tr>
<td>F5</td>
<td>60</td>
<td>10,51</td>
<td>28,52</td>
<td>0,97</td>
<td>40</td>
<td>100</td>
</tr>
<tr>
<td>F6</td>
<td>70</td>
<td>7,88</td>
<td>21,35</td>
<td>0,73</td>
<td>30</td>
<td>100</td>
</tr>
</tbody>
</table>

B.4.4 Physical Quality Examination of the Preparations

Physical quality examination was carried out on each lip balm preparation. Physical quality checks include: homogeneity test, stability test, pH measurement, smear test, irritation test and the ability of the preparation to moisturize lips using a skin analyzer.

a. Homogeneity test
Homogeneity test was carried out using a glass object. A certain amount of the preparation when applied to a piece of glass or other suitable transparent materials, the preparation must then show a homogeneous arrangement and no coarse grains should be seen [11].

b. Observation of the stability of the preparation
The stability check of the preparation was carried out for changes in the shape, color, and odor of the preparation, carried out on each preparation during storage at room temperature 1, 4, 8 and 12 weeks [11].

c. Measurement of pH of preparations
Determination of pH of preparations was carried out using a pH meter. How it works: 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. Then the electrode was washed with distilled water, then dried with a tissue. Samples were made with a concentration of 1%, that weighed 1 g of the preparation dissolved in distilled water which had been heated to 100 ml and allowed to cool. Then the electrode was immersed in the solution. The tool was left until it showed a constant pH value. The number shown by the pH meter is the pH of the preparation [12].

d. Smear test
The smear test was done visually by applying lip balm on the back of the hand and then observing the amount of preparation that sticks to a certain pressure as we usually use lip moisturizer. Examination was carried out on each preparation that was made and applied to the back of the hand with 5 times of application [13,14].

e. Irritation test
Irritation test was carried out by applying the test preparation to the normal skin of a human panel to determine whether the preparation could cause irritation to the skin or not. The technique used in this irritation test was an open patch test (open test) on the inner forearm of 12 panelists. The open patch test was carried out by applying the preparation made at the location of the attachment with a certain area (2.5 x 2.5 cm), leaving it open and observing what happened. This test was carried out 3 times a day for 3 consecutive days. The adhesive test location is the part of the panel skin that is used as the location area for the patch test. Usually the most appropriate areas for patch testing are the back, the inside of the upper arm, the folds of the elbow, and the skin behind the ear [11,14].

f. The ability of the preparation to moisturize the lips
Testing the effectiveness of the lip balm was carried out on 12 volunteers and divided into groups, namely:

a) Group I : 2 volunteers for lip balm formula 1 (20%)
b) Group II : 2 volunteers for lip balm formula 2 (30 %)
c) Group III : 2 volunteer for lip balm formula 3 (40%)
d) Group IV : 2 volunteer for lip balm formula 4 (50%)
e) Group V : 2 volunteer for lip balm formula 5 (60%)
f) Group VI : 2 volunteers for lip balm formula 6 (70%)
All volunteers measured the initial condition of the lip skin in the test area to be applied lip balm. The test carried out was a moisture test, using a skin analyzer. Treatment began by applying lip balm to evenly coat the lips. Lip balm was applied according to the groups that have been set above. Changes in lip skin condition were measured before application and after application (1 week, 2 weeks, 3 weeks, and 4 weeks).

C. Data Analysis
Research data will be described step by step according to work procedures. Reporting on the formulation of lip balm preparations will be explained in a narrative descriptive manner. Examination of the quality of the preparation, namely homogeneity test, stability test, pH, smear test, and irritation test will be presented in the form of tables and narratives. The data on the results of the effectiveness test, namely the water content, will be presented in the average and standard deviation for each formula and presented in the form of tables and graphs. The data were then analyzed for distribution using the Shapiro-Wilk test. If the data is normally distributed, the paired t-test will be used, but if the data is not normally distributed, the Wilcoxon test will be used to determine the effectiveness of lip balm between formulas. The difference is declared significant if the P value <0.05.

RESULT AND DISCUSSION
Analysis of the quality of grapeseed oil made from the mechanical extraction - expeller pressing method in this study was not carried out, so the quality of the raw material for grapeseed oil could not be ascertained.

A. Results of Homogeneity Test
The results of homogeneity examination of grapeseed oil as lip balm preparations with concentrations of 20%, 30%, 40%, 50%, 60%, and 70% showed that all preparations did not show any coarse grains when the preparation was applied to the transparent glass. This shows that the preparations made have a homogeneous composition. The homogeneity test was carried out with the aim of knowing the homogeneity aspect of the lip balm preparation that had been made. Homogeneous preparations will produce good quality because it shows the drug ingredients are evenly dispersed in the base material, so that in each part of the preparation contains the same amount of drug. If the drug substance is not evenly dispersed in the base material then the drug will not achieve the desired therapeutic effect [15].

B. Observation Results of Preparations Stability
Lip balm preparations were stored at room temperature away from sunlight from March-May 2021 and changes in texture, color, and odor were observed. The results of observations on the stability of the preparations showed that all preparations (formulation 1 – formulation 6) remained stable at room temperature storage for 1, 4, 8, and 12 weeks where there was no change in each parameter tested.

The storage room temperature for this lip balm based on grape seed oil for 12 weeks from March to May 2021 is known to be between 25°C – 30°C. Based on previous research, grapeseed oil was found to have a weakness in terms of stability. Grapeseed oil stored at room temperature 22°C and exposed to sunlight can show damage which is indicated by an increase in the peroxide value reaching 484 meq O₂/kg oil. Air and sunlight can accelerate the oxidation process of linoleic acid and affect the stability of linoleic acid, so grapeseed oil is expected to be packaged in good dosage forms or formulations [16].

C. Results of PH Examination
PH is one of the important parameters in topical dermatology preparations. The recommended pH for cosmetic preparations ranges from 4.5-6.5 according to the physiological pH of the skin. Preparations with a more acidic pH are recommended for areas such as the armpits and inguinal for the skin's defense function. During 12 weeks of observation, the average pH of the 6 formulations ranged from 5.5 to 6.2. Preparations for cosmetics are recommended to be weakly acidic or alkaline to avoid skin irritation [17].

D. Results of Smear Test
Based on the smear test conducted on the lip balm preparation of grapeseed oil with a variant of 6 concentrations on the back of the hands of the research subjects, it was found that concentrations of 20%, 30%, and 40% were difficult to smear evenly on the surface of the back of the hand due to the higher dose of the carrier in comparison to the oil. Meanwhile, at concentrations of 50%, 60%, and 70% the preparations can be smeared evenly.

E. Results of Irritation Test
The irritation test was carried out by observing the presence of erythema, papules, vesicles, and/or edema reactions on the volar part of the antebrachial region after applying lip balm with different concentrations. Based on the test for 3 days in a row, there were no research subjects who experienced irritation or itching reactions on the skin that was smeared with lip balm preparations. Irritation is one of adverse reactions that may be occurred to one of the primary constituents of cosmetic formulation or contamination or procedural misconduct. This testing procedure is aimed to prevent unwanted dangerous reaction before applying to lips [18,19].

F. Results of Measurement of Moisture Content

To evaluate the effectiveness or ability of lip balm preparations, measurements of water content on the lips of research subjects were carried out before using lip balm preparations (baseline) and after using lip balm which were measured at week I, week II, week III, and week IV using a portable skin analyzer. The research subjects involved in this study were 12 women aged 17-50 years with each group consisting of 2 research subjects who met the inclusion criteria and were willing to participate in this study from beginning to end. The results of the water content test can be seen in the table 2.

Based on the average measurement results, it was found that all preparations succeeded in increasing the moisture content of the lips of research subjects, preparations with a concentration of 70% succeeded in increasing the highest water content of 6.72 or 13.09% and statistically significant through the paired t-test (value P<0.05). To determine the difference in the increase in water content between concentration groups, a one-way anova test was carried out and the test obtained a P value of 0.002, which means the difference in the increase in water content between groups was significant. The increase in water content in each preparation with different concentrations can be seen in the graph 1.

**Table 2. Moisture Content of Lip After Lip Balm Application**

<table>
<thead>
<tr>
<th>Concentration Formulation</th>
<th>Moisture content after lip balm application</th>
<th>Increase of moisture content</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>baseline</td>
<td>1 week</td>
<td>2 weeks</td>
</tr>
<tr>
<td>20</td>
<td>50.25</td>
<td>51.25</td>
<td>50.85</td>
</tr>
<tr>
<td>20</td>
<td>48.21</td>
<td>48.90</td>
<td>49.21</td>
</tr>
<tr>
<td>Average</td>
<td>49.23</td>
<td>50.08</td>
<td>50.03</td>
</tr>
<tr>
<td>30</td>
<td>55.72</td>
<td>56.72</td>
<td>57.02</td>
</tr>
<tr>
<td>30</td>
<td>52.12</td>
<td>52.33</td>
<td>52.25</td>
</tr>
<tr>
<td>Average</td>
<td>53.92</td>
<td>54.53</td>
<td>54.64</td>
</tr>
<tr>
<td>40</td>
<td>48.57</td>
<td>48.87</td>
<td>49.02</td>
</tr>
<tr>
<td>40</td>
<td>45.51</td>
<td>46.21</td>
<td>46.34</td>
</tr>
<tr>
<td>Average</td>
<td>47.04</td>
<td>47.54</td>
<td>47.68</td>
</tr>
<tr>
<td>50</td>
<td>55.12</td>
<td>57.22</td>
<td>58.67</td>
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<tr>
<td>50</td>
<td>46.61</td>
<td>47.82</td>
<td>50.03</td>
</tr>
<tr>
<td>Average</td>
<td>50.87</td>
<td>52.52</td>
<td>54.35</td>
</tr>
<tr>
<td>60</td>
<td>47.92</td>
<td>49.01</td>
<td>50.87</td>
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<tr>
<td>60</td>
<td>46.83</td>
<td>48.21</td>
<td>50.17</td>
</tr>
<tr>
<td>Average</td>
<td>47.38</td>
<td>48.61</td>
<td>50.52</td>
</tr>
<tr>
<td>70</td>
<td>50.32</td>
<td>52.24</td>
<td>53.97</td>
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<tr>
<td>70</td>
<td>52.34</td>
<td>53.98</td>
<td>55.13</td>
</tr>
<tr>
<td>Average</td>
<td>51.33</td>
<td>53.11</td>
<td>54.55</td>
</tr>
</tbody>
</table>
The expected water content in the skin is at least 60%, with dry criteria if the water content in the skin is 0-39%, normal with 40-59%, and moist with 60-100% [20]. Having seen the initial value of the water content on the lips of the research subjects, 100% of the research subjects have normal water content on the lips. After using grapeseed-based lip balm, the water content increased but none reached 60%. Good moisture is necessary to maintain the integrity of the stratum corneum. Cosmetics such as lip balms are made to develop and maintain the skin barrier function and prevent dryness. Moisturizers do not work by adding water to the skin, but moisturizers work by preventing and reducing the evaporation of water from the skin. The skin is expected to rehydrate itself, so the use of moisturizers is recommended in high intensity and long duration [21].

CONCLUSION
Balinese grapeseed oil (*vitis vinifera l var alphonso lavalle*) has great potential and can be developed as a lip balm to maintain healthy skin, especially lips. Lip balm preparations made from Balinese grapeseed oil are homogeneous, stable at room temperature, have a PH that is safe for the lips, and do not irritate the skin. Lip balm formulation with a concentration of 70% is the best concentration from the Balinese grapeseed oil and the most effective in increasing the moisture content of the lips.

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REFERENCES