SKIN EXTRACT OF GRAPE (Vitis vinifera L) AS DYES IN LIPSTICK FORMULATION

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ABSTRACT
Grapes (Vitis vinifera L.) belong to the family Vitaceae. The color of the fruit varies. There are red, green and purple. The color of this wine contains anthocyanin dyes that can be used as natural dyes to replace synthetic dyes, the colors they contain are pretty intensive, so the researchers aimed to make lipstick formulations using natural dyes from grape skins. The lipstick formulation consisted of Cera alba, lanolin, vaseline alba, cetyl alcohol, oleum ricini, carnauba wax, propylene glycol, titanium dioxide, oleum rosae, butylhydroxytoluene, tween 80, and nipagin, as well as the addition of grape skin extract with a concentration of 15%, 20%, 25%, 30%, and 35%. Tests on the preparations made include inspection of homogeneity, melting point, lipstick strength, stability test against changes in shape, color, and odor during 30 days of storage at room temperature, smear test, pH examination, and irritation test preference test (Hedonic test). From the research results, grape skin extract can be formulated into lipstick preparations with pink to dark red colors. Lipstick preparations with grape skin extract as a dye are pretty stable, homogeneous, melting point 59°C, have good lipstick strength, pH ranges from 3.7 to 3.9 (close to the pH of the lip skin) are easy to apply with an even color, and do not irritate. Irritation the preparation preferred by 30 panelists was preparation with a concentration of 25%.

Keywords: grape skin, Vitis vinifera L., lipstick, lipstick components

INTRODUCTION
Cosmetics has a long history of human life and is based on archaeological excavations. It is known that cosmetics have been used by humans who lived during the stone age. Currently, cosmetics are an essential part of everyday life. The number of cosmetics used continues to increase and the increase in population every year (Mitsui, 1997). Cosmetics are needed by humans, both men, and women, from birth to when they leave this world. Various cosmetic products are used every day throughout the body, from hair to toes, and are generally used repeatedly, so safety requirements are needed for use (Tranggono and Latifah, 2007).

Synthetic materials contained in cosmetics often cause side effects that are detrimental to health, for example, the emergence of irritating effects and carcinogenic risks, especially in the use of dyes, so the effort to return to nature (back to nature) also affects the world of cosmetics with efforts to re-popularize and dig back. Cosmetics made from natural ingredients have long been forgotten. However, based on economic and technical considerations, some producers only use traditional elements in their cosmetic production (Wasitaatmadja, 1997).

Lipstick is a cosmetic preparation used to color the lips with an artistic touch to improve the aesthetics of makeup. Its function is to give the lips
a red color, as red as a pomegranate, which gives a healthy and attractive facial expression. Nevertheless, other colors became popular with people in reality, so lip paints varied from light colors to very dark colors ranging from pink, red-orange, to red-blue, and even purple (Ditjen POM, 1985). Lipstick is a facial cosmetic product that has become an identity for women in this modern era. Without this lip color message, many women feel less confident in public. The need for lipstick continues to increase along with the emergence of new lipstick products, both domestic and global brands, that continue to follow the needs of consumers.

Grapes are fruit plants in the form of vines belonging to the Vitaceae family, and they taste sweet, half sweet, half-sour, delicious, and fresh. The shape of the fruit is round or oval. This fruit is also known for containing many polyphenolic compounds and resveratrol, which play an active role in various body metabolisms and prevent the formation of cancer cells and various other diseases. This activity is also related to secondary metabolite compounds in grapes that act as antioxidant compounds that can ward off free radicals (Anonymous, 2012).

Based on the description above; The author wishes to make lipstick preparations using natural dyes on the skin of red-purple grapes as a coloring agent, namely by extracting dyes from grape skins, followed by the formulation of lipstick preparations using dye extracts obtained from grape skins.

### MATERIALS AND METHODS

This research method is experimental. The research includes sample preparation, manufacture of extracts of grape skin coloring agents, manufacture of dosage formulations, examination of physical quality of preparations, irritation test of preparations, anhedonic test on variations of preparations made.

The samples used were purplish-red grapes that were still freshly obtained from the Supermarket Brastagi, Jl Gatot Subroto, No. 288, Medan-North Sumatra. The comparison preparation used was Wardah®.

**Extract**

A total of 127.05 grams of grape skins were macerated with 1 liter of 96% ethanol, mixed with 0.5% citric acid, covered, and left for 12 hours protected from light while frequently stirring, filtered with filter paper, the filtrate was collected (first filtrate). Then the dregs were macerated again with 1 liter of 96% ethanol filtered through filter paper. The results obtained were mixed with the first filtrate and then evaporated with the help of a rotary evaporator at a temperature of ± 50°C, then dried with the help of a freeze dryer at a temperature of -40°C for two days (Hidayat, 2006), in order to obtain the following grape skin extract called EKBA.

**Lipstick Making Lipstick**

Making is done by using grape skin extract as a coloring agent in various concentrations, namely 15%, 20%, 25%, 30%, and 35%.

**Formula**

The grape skin extract dissolve nipagin in the extract; it is necessary to add propylene glycol. Propylene glycol used as a solvent is 5-80% (Rowe et al., 2009).

Titanium dioxide is used as much as 0.5% to concentrate the color of the lipstick. Butyl hydroxytoluene is used as an antioxidant as much as 0.0075-0.5% (Rowe et al., 2009). The coloring matter from grape skin extract cannot mix with fat and wax, so it is necessary to add tween 80 as an emulsifier. Oleum rosae is used as much as 0.1% as perfume.

Modification of the formula made is as follows:

| R/ Cera alba | 36.0 |
| Vaseline alba | 36.0 |
| Lanolin | 8.0 |
| Cetyl alcohol | 6.0 |
| Oleum ricini | 8.0 |
| Carnauba wax | 5.0 |
| Nipagin | 0.1% |
| Propylene glycol | 7% |
| Titanium dioxide | 0.5% |
| Butyl Hydroxy Toluene | 0.5% |
| Tween 80 | 2% |
| Oleum Rosae | 0.1% |

The weight of one lipstick made is 3.5g. Each formula is made up of 5 lipsticks. The total number of lipsticks made = five formulas, and each formula is made of 5 lipsticks, so the total number of lipsticks made = five formulas x 5 lipsticks = 25 lipsticks.

The total amount of lipstick base ingredients used for 25 lipsticks is = 25 lipsticks x 3.5g = 87.5g, rounded to 100 g, so the required lipstick
ingredients can be calculated as follows:

A. Additives

- Nipagin 0.1% = \( \frac{0.1}{100} \times 100g = 0.1g \)
- Propylene glycol 7% = \( \frac{7}{100} \times 100g = 7g \)
- Titanium dioxide 0.5% = \( \frac{0.5}{100} \times 100g = 0.5g \)
- Butyl hydroxy toluene 0.5% = \( \frac{0.5}{100} \times 100g = 0.5g \)
- Tween 80 2% = \( \frac{2}{100} \times 100g = 2g \)
- Oleum Rosae 0.1% = \( \frac{0.1}{100} \times 100g = 0.1g \)

B. Main Ingredients = 100g - \((0.1 + 7 + 0.5 + 0.5 + 2 + 0.1)g\)

- Cera alba = \( \frac{36}{99} \times 89.8g = 32.65g \)
- Lanolin = \( \frac{3}{99} \times 89.8g = 7.25g \)
- Vaseline alba = \( \frac{36}{99} \times 89.8g = 32.65g \)
- Cetyl alcohol = \( \frac{5}{99} \times 89.8g = 5.44g \)
- Oleum ricini = \( \frac{8}{99} \times 89.8g = 7.25g \)
- Carnauba wax = \( \frac{5}{99} \times 89.8g = 4.53g \)

Based on the orientation of the lipstick base using the above formula, it was found that a good lipstick base was obtained. The results of the orientation towards the concentration of grape skin extract in the lipstick preparation used in this study gave a clear color of 15%, so based on this 15% concentration, variations in the concentrations of 15%, 20%, 25%, 30%, and 35% were made. Obtained lipstick formulations with varying concentrations of grape skin extract can be seen in table 1 below:

| EKBA   | Formula | Weight (grams) | Base Weight (grams) | Total Weight (grams) |
|--------|---------|----------------|---------------------|----------------------|
| Formula 1 | EKBA 15% | 3              | 17                  | 20                   |
| Formula 2 | EKBA 20% | 4              | 16                  | 20                   |
| Formula 3 | EKBA 25% | 5              | 15                  | 20                   |
| Formula 4 | EKBA 30% | 6              | 14                  | 20                   |
| Formula 5 | EKBA 35% | 7              | 13                  | 20                   |
| Blank   | EKBA 0%  | 0              | 20                  | 20                   |

Procedure for making lipstick

Weighed Cera alba, cetyl alcohol, carnauba wax, vaseline alba, and lanolin were put in a vaporizer cup, then dissolved on a water bath so that Mass I was obtained. Butyl hydroxytoluene was dissolved in oleum ricini, then added titanium dioxide, nipagin, and Tween 80, stirred until homogeneous, then obtained Mass II. Then Mass I and Mass II were mixed while stirring slowly until homogeneous in a still-hot evaporating dish, so that a lipstick base was obtained, which was used to make lipsticks of various formulas based on variations in EKBA concentrations and made one formula without the addition of EKBA which was used as a blank formula.

The grape skin extract was dissolved in propylene glycol, stirred and then mixed with the lipstick base in a liquid state, stirred until homogeneous, finally added oleum rosae while the liquid was put into the mold and allowed to freeze. After freezing, the lipstick is removed from the mold and put into a container (roll-up lipstick). The results of the manufacture can be seen in appendix 6.

Physical Quality Examination of Preparations Physical

Quality inspection of preparations is carried...
out on each lipstick preparation, including inspection of homogeneity, melting point, lipstick strength, and stability of the preparation, which includes observations of changes in shape, color, and odor of the preparation smear test and pH check.

**Homogeneity examination**

Each lipstick preparation made from grape skin extract was checked for homogeneity by applying a certain amount of the preparation on a transparent piece of glass. The preparation must show a homogeneous arrangement, and there are no coarse grains (Directorate General of POM, 1979).

**Examination of the melting point of the lipstick**

Observations were made on the melting point of lipstick by melting the lipstick. The ideal Lipstick melting temperature is a temperature that is close to the lip temperature between 36-38°C. However, due to the need to pay attention to the factor of resistance to environmental weather temperatures, especially temperatures in the tropics, the melting temperature of lipstick is made higher, which is in the range of 55-75°C (Directorate General of POM, 1985).

The method of observing the melting point of lipstick used in this study was to put the lipstick in an oven with an initial temperature of 50°C, after which the temperature was increased by one °C every 15 minutes. It is observed at what temperature the lipstick begins to melt.

**Examination of lipstick strength**

Observation of the strength of the lipstick is done by placing the lipstick horizontally at a distance of approximately 1/2 inch push or pull-down weighing 3.5g. Every 30 seconds, the weight of the load is increased by 10 grams. The addition of the weight of the load as a suppressor is carried out continuously until the lipstick breaks when the lipstick breaks are the value of the strength of the Lipstick (Vishwakarma, et al., 2011).

**Examination of the stability of the preparation**

Observation of the lipstick stability was carried out by observing the changes in the shape, color, and odor of each formula during storage at room temperature on the 1st, 5th, 10th day and after that every five days until the 30th day. Changes were observed from the beginning of printing to storage for 30 days.

The smear test was carried out visually by applying lipstick on the back of the hand and then observing the thickness of the color attached to the skin by applying five times of application at a certain pressure, as is usually the case with lipstick. Lipstick preparations have good greasing power if the color that sticks to the skin on the back of the hand is thick and evenly distributed with several smears at a specific pressure. Meanwhile, the preparation is said to have poor greasing power if the color is slightly and unevenly attached. Observations were made on each smear, and an examination was carried out on each formula made (Keithler, 1956).

**Determination of the pH of the preparation**

The determination of the pH of the preparation is carried out 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. Then the electrodes were washed with distilled water then dried with a tissue.

Lipstick is made in a concentration of 1%, that is, 1 gram of the preparation is melted and dissolved in 100 ml of distilled water over a water bath, then the electrode is immersed in the solution. Let the tool show the pH value until it is constant. The number shown by the pH meter is the pH of the preparation (Rawlins, 2003).

**Irritation Test and Hedonic Test**

After testing the physical stability of the preparation, then proceed with the irritation test and the hedonic test on the preparation made.

1. **Irritation**

   a test Irritation test was carried out on lipstick preparations made from grape skin extract to ensure that the lipstick made did not irritate the skin. Irritation can be divided into primary irritation that will appear immediately after attachment or contact with the skin and secondary irritation, whose reaction occurs only a few hours after touching or sticking to the skin. The signs caused by the two irritations are marked by redness, itching, or swelling of the skin to which the preparation is attached (Directorate General of POM 1985).

   The technique used in this irritation test is
an open test (Open Test) done by smearing the formula with the highest concentration, namely 35% grape skin extract behind the earlobe on ten volunteers. Observed for 24-48 hours after basting (Wastatiatmadja, 1997).

Panelist for irritation test (Directorate General of Drug and Food Control, 1985):
1. Female
2. Age between 20-30 years of
3. good physical and mental health
4. No history of allergic disease
5. Expresses willingness to be a panelist for irritation

2. test Preferred test (Hedonic Test)
A preference test is carried out to determine the panelist's level of preference for lipstick preparations that are made. This preference test was carried out visually on 30 panelists by applying an lipstick made from various concentrations of grape skin extract on the skin on the back of his hand. Parameters observed in this preference test are the ease of applying lipstick the homogeneity and intensity of the lipstick color when applied to the back of the hand. The criteria for panelists are as follows (Soekarto, 1981):
1. The panelists used were untrained panelists who were randomly selected as many as 30 panelists.
2. good health
3. Not under stress
4. Have knowledge and experience of organoleptic assessment methods.
Panelists write down their preferences for a lipstick with the following criteria:

| Description     | Code | Value |
|-----------------|------|-------|
| Strongly Like   | SS   | 5     |
| Like            | S    | 4     |
| Dislike         | KS   | 3     |
| Dislike         | TS   | 2     |
| Very Dislike    | STS  | 1     |

RESULT AND DISCUSSION

Quality Inspection Results Physical Preparation

Homogeneity of the preparation homogeneity of the preparation is done by applying the lipstick preparation on a transparent piece of glass. The results of the homogeneity examination showed that all lipstick preparations did not show any coarse grains when the preparation was applied to transparent glass. This shows that the preparations are homogeneous (Directorate General of POM, 1979).

The melting point of the lipstick The melting point of lipstick is made by melting it in an oven with an initial temperature of 50°C and increasing it by one °C every 15 minutes.

| Formula | for EKBA Concentration | Temperature °C |
|---------|------------------------|----------------|
| F-1     | EKBA 15%               | 59             |
| F-2     | EKBA 20%               | 59             |
| F-3     | EKBA 25%               | 59             |
| F-4     | EKBA 30%               | 59             |
| F-5     | EKBA 35%               | 59             |
| Blank   | EKBA 0%                | 59             |
Table 3 above shows the melting point results of all lipsticks melting at 59°C. This indicates that the preparation of lipstick made meets the requirements for the melting point of lipstick, which is in the range of 55-75°C (Directorate General of POM, 1985).

**Lipstick**

Examination of lipstick strength was carried out by hanging weight of 3.5g, which was added 10g every 10 seconds % inch from the edge of the lipstick tip, and observing the weight of the load when the lipstick was broken, the results were as follows:

| Formulation | Concentration | Weight (grams) |
|-------------|---------------|----------------|
| F-1         | EKBA 15%      | 113.5          |
| F-2         | EKBA 20%      | 113.5          |
| F-3         | EKBA 25%      | 103.5          |
| F-4         | EKBA 30%      | 103.5          |
| F-5         | EKBA 35%      | 93.5           |
| BLANKO      | EKBA 0%       | 143.5          |
| Wardah® (Comparative Lipstick) |               | 93.5 |

Table 4 above shows that the results of the electrical strength examination show differences in the ability of lipstick preparations to withstand loads. Differences in the concentration of EKBA cause this difference used. The higher the concentration of grape skin extracts in the lipstick preparation, the less lipstick base is used. This causes lipstick with a concentration of 35% more easily broken than other lipstick preparations that use lower grape skin extract.

The results of the examination of the strength of the lipstick showed that the lipstick preparation was broken at a weight increase of 93.5-143.5 grams. This hat indicates that the preparation made has good strength. This conclusion is drawn by comparing the weight of the load used in testing lipsticks on the market, namely Wardah® lipstick weighs 93.5 grams. So Lipstick preparations made using grape skin extract have good lipstick strength.

**Stability preparations**

checks were carried out by observing changes in the shape, color, and odor of the preparations stored at room temperature for 30 days.

| Observation | Formula | (Days) |
|-------------|---------|--------|
| Forms       | of ECBA 15% | b b b b b b |
|             | EKBA 20%   | b b b b b b |
|             | EKBA 25%   | b b b b b b |
|             | EKBA 30%   | b b b b b b |
|             | EKBA 35%   | b b b b b b |
|             | EKBA 0%    | b b b b b b |
| Color       | 15%       | p p p p p p |
|             | EKBA 20%   | mm mm mm mm mm mm mm |
|             | EKBA 25%   | mm mm mm mm mm mm mm |
|             | EKBA 30%   | m m m m m m m m |
|             | 35%       | m m m m EKBA EKBA |
|             | %         | mt mt mt mt mt mt Odor |
| 15          | 0%        | bk bk bk bk bk bk |
|             | EKBA 20%   | bk bk bk bk bk bk |

Table 5. Observation Data on Changes in Shape, Color, and Odor Observation Preparations
Table 5 above shows that the stability test results for all preparations made are constant. Stable in storage at room temperature for 30 days of observation. From the observation of the shape, it was found that all the lipstick preparations made did not change either from the initial form of printing to 30 days at room temperature storage. The increased concentration of grape skin extract used, the more concentrated the lipstick color produced. Lipstick with a concentration of 15% and 20% grape skin extract gave a pink color, a concentration of 25% and 30% gave a red color, while a concentration of 35% gave a dark red color. In comparison, the smell produced from all lipstick preparations is the distinctive smell of the perfume used, namely oleum rosae. The odor of the preparation remained stable in storage for 30 days of observation at room temperature.

A smear test A

lipstick preparation is said to have an excellent greasing power if it provides an intensive, even, and homogeneous color when applied to the skin on the back of the hand. Based on the smear test, it was found that variations in the concentration of grape skin extract used resulted in different color intensities of different lipstick preparations, so the application required different application repetitions to get an even and intensive color. The results of smearing can be seen as follows.

| Concentration of EKBA• | The amount of application that gives an even and intensive color |
|------------------------|---------------------------------------------------------------|
| F-1 EKBA 15%           | fifth                                                          |
| F-2 EKBA 20% The       | application second                                             |
| F-3 EKBA 25% The       | application second                                             |
| F- 4 EKBA 30%          | First                                                          |
| F-5 EKBA 35%           | First                                                          |

The data above shows that preparations with 30% and 35% concentrations have high color density and have the best greasing power. One application of the preparation that has given intensive color indicates this. In contrast, the first application still produces a color that is too light for other formulas.

pH

Examine pH examination was conducted by making a 1% solution of lipstick preparation in distilled water and testing the pH using a pH meter. The results are as follows:
Table 7. Data Examination of pH

| Formula | Concentration of EKBA | pH |
|---------|-----------------------|----|
| F-1     | EKBA 15%              | 3.9|
| F-2     | EKBA 20%              | 3.9|
| F-3     | EKBA 25%              | 3.8|
| F-4     | EKBA 30%              | 3.7|
| F-5     | EKBA 35%              | 3.7|
| Blank   | EKBA 0%               | 6.1|

Table 7 shows the results of pH examination for formulas without grape skin extract having a pH of 6.1, while those made using grape skin extract have a pH ranging from 3.7 - 3.9. This pH is close to the physiological pH of the skin, which is ± 4. Thus, the formula can be used for lipstick preparations (Balsam, 1972).

Irritation

The irritation test was carried out by applying lipstick from the formula with the highest concentration of EKBA, which is 35% behind the earlobe, and observed for 24-48 hours.

Table 8. Irritation Test Data

| Observation    | 1  | 2  | 3  | 4  | 5  | 6  | 7  | 8  | 9  | 10 |
|----------------|----|----|----|----|----|----|----|----|----|----|
| Skin Redness   | (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (+) |
| Itchy skin     | (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (+) |
| Swollen skin   | (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (-)| (+++)

Remarks:  
(-) : no irritation  
(+) : skin redness  
(++) : itchy skin  
(++++) : swollen skin  

Table 8 The above shows the results of the irritation test carried out on ten volunteers by applying lipstick behind the earlobe and observed for 24-48 hours, indicating that all panelists gave negative results to the irritation reaction parameters observed, namely red skin, itching, or swelling. The irritation test results' data shows that the lipstick preparation made using grape skin extract as a coloring agent is relatively safe to use.

Results of the hedonic Test

The data obtained from the assessment sheet (questionnaire) were tabulated, and the preference value for each preparation was determined by finding the average result for each panelist at a 95% confidence level. benefits are as follows:

Table 9. Data Hedonic Test Assessment

| Panelist | Results on Various Preparations |
|----------|---------------------------------|
|          | FI : EKBA 15% | F2 : EKBA 20% | F3 : EKBA 25% | F4 : EKBA 30% | F5 : EKBA 35% |
| 1        | 1 STS | 2 TS | 5 SS | 3 KS | 4 S |
| 2        | 1 STS | 3 KS | 5 SS | 4 SS | 2 TS |
| 3        | 1 STS | 4 S  | 5 SS | 3 KS | 2 TS |
| 4        | 1 STS | 2 SS | 3 KS | 4 S  | 5 SS |
| 5        | 1 STS | 2 KS | 3 4  | 5 S  | SS SS|
| 6        | 1 STS | 2 TS | 3 KS | 4 S  | 5 SS |

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The calculation results obtained the interval of preference value for each preparation as follows:

**Table 10. Interval Test Result of Preferred-Value of Each Formula**

| Formula | Concentration EKBA | an interval of preference value obtained | The smallest | Conclusion |
|---------|--------------------|----------------------------------------|-------------|------------|
| F-1     | EKBA 15%           | 0.967-1.433                           | 0.967= 1    | Very dislike |
| F-2     | EKBA 20%           | 2.138-2.795                           | 2.138 = 2   | Dislike    |
| F-3     | EKBA 25%           | 3,576-4,290                           | 3,576 = 4   | Likes      |
| F-4     | EKBA 30%           | 3,292-4,041                           | 3,292 E. 3  | Dislikes   |
| F-5     | EKBA 35%           | 3,359-4,122                           | a 3         | Dislikes   |

Based on Table 10 above, it can be concluded that the panelists prefer lipstick preparations containing 25% EKBA. The panelists did not like the formulas containing 30% and 35% EKBA, because the results of the color applied were too dark, while the formula with 15% EKBA was very disliked because the color applied was too light. The panelists did not like the formula with 20% EKBA because of the color. Produced is still too young. This conclusion was drawn because the average Indonesian skin color is brown.

Overall, the test results for lipstick preparations were formulated using grape skin extract with various concentrations, including physical quality and irritation tests. All met the requirements of good lipstick. However, the preference test results using 30 panelists, it appears that the preferred formula is a formula with a concentration of grape skin extract by 25%.

**CONCLUSION**

Grape skin extract can be used as a colorant in the formulation of lipstick preparations. The resulting colors are pink at a 15% and 20% grape skin extract.
extract dye, red at a 25% and 30% grape skin extract dye concentration, and dark red at a 35% grape skin extract dye concentration. The physical quality of all preparations made is stable and does not show any changes in shape, color, and odor in storage for 30 days at room temperature. The lipstick preparations did not irritate, and the panelists preferred the preparations with a 25% grape skin extract concentration.

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