

Research Article

## Development of Liquid Lipstick Containing Natural Dyes from Kepok Banana Flower Peel Extract (*Musa Paradisiaca* L.) and Study on Its Stability

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### Abstract (in English)

Liquid lipstick is a decorative cosmetic preparation used to add color to the lips. This study aims to determine alternative colorants derived from natural ingredients. The peel of kepok banana flower (*Musa paradisiaca* L.) which contains anthocyanins can serve as a natural dye, as evidenced by the total anthocyanin content test using the differential pH method, yielding 35.958 mg/L. Three formulations were developed using varying concentrations of kepok banana flower peel extract: F1 (5%), F2 (7.5%), and F3 (10%). The formulations underwent six *freeze-thaw* (-21°C and 25°C) and *cycling* (4°C and 40°C) stability cycles, with evaluations of organoleptic properties, viscosity, pH, and spreadability after each cycle. All formulations showed good physical stability which are semi-solid texture, rose scent, homogeneity, brown color, and acceptable spreadability, viscosity, pH, and non-irritant effects. Preference testing with 30 panelists identified F2 as the most favored.

**Keywords:** Liquid Lipstick, Kepok Banana Flower Peel, Anthocyanin

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## 1 Introduction

Cosmetics are preparations intended to be applied to the body, particularly the external parts or other areas, with the purpose of supporting hygiene, enhancing beauty, and maintaining the skin, thereby increasing self-confidence. Cosmetics used to enhance appearance include nail cosmetics, eye cosmetics, hair cosmetics, facial cosmetics, and lip cosmetics especially lip colorants, as the lips are a part of the face that significantly influence the perception of one's facial appearance. Lip colorants are preparations designed to add color to the lips, thereby providing aesthetic value to one's appearance through the use of attractive color pigments [1]. Currently, lip colorants are available in the form of creams, crayons, and liquids such as lipstick, lip gloss, lip liner, and liquid lipstick [2].

Liquid lipstick is a type of lip colorant in a liquid form that is glossy and highly pigmented. Liquid lipstick is the liquid version of traditional lipstick, containing a higher amount of oil compared to solid lipstick formulations, which results in a more fluid consistency and facilitates easier application. Liquid lipstick is also generally more comfortable to use, as it contains less wax than solid lipsticks, thereby reducing the sensation of heaviness on the lips. One of the advantages of liquid lipstick is its durability, as it is less prone to breaking compared to solid lipstick formulations [3]. Liquid lipstick is widely used in daily human activities, and there is a possibility that the product may enter the digestive tract or be ingested along with food or beverages. Therefore, the colorants used in lip colorant formulations must be safe and non-toxic [4].

Colorants that tend to cause fewer side effects are natural dyes. Natural colorants are derived from natural sources such as minerals, plants, or animals. An example of a natural colorant source that can be used in lip colorant formulations is the bract (outer petal) of the banana flower from *Musa paradisiaca* L. The bract of the kepok banana flower (*Musa paradisiaca* L.) is known to contain a total anthocyanin content of 4.67 mg/100 g fresh weight, which is visually indicated by its reddish-purple coloration. Anthocyanins belong to the flavonoid group and typically impart purple, red, or blue hues to plants. The anthocyanin content in banana blossoms makes them a potential source of natural dye through extraction processes [5].

This study aims to identify and evaluate the physical quality parameters of liquid lipstick formulated with varying concentrations of kepok banana flower peel (*Musa paradisiaca* L.) extract as a natural colorant, as well as to assess the stability and safety of the prepared formulations.

## 2 Method

The equipment used in this study includes a rotary evaporator, blender, hot plate, pH meter, Brookfield viscometer, UV-Visible spectrophotometer, incubator, freezer, refrigerator, and the necessary glassware.

The materials used in this study include kapok banana flower peel (*Musa paradisiaca* L.) obtained from Kampung Meraang, Berau City; distilled water (aquadest); 96% ethanol; analytical grade ethanol; 1% HCl; 2N HCl; 2N NaOH; Dragendorff's, Mayer's, and Wagner's reagents; magnesium powder; 1% FeCl<sub>3</sub>; citric acid (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>); sodium phosphate (Na<sub>3</sub>PO<sub>4</sub>); potassium chloride (KCl); castor oil; beeswax; dimethicone; tocopherol; BHT; cetyl alcohol; titanium dioxide (TiO<sub>2</sub>); methylparaben; and oleum rosae.

### 2.1 Sample Preparation and Extraction

Sample preparation began with the collection of kapok banana flower peel (*Musa paradisiaca* L.), which were then cleaned and subjected to wet sorting. The bracts were subsequently chopped into smaller pieces and air-dried to prevent degradation of anthocyanin compounds due to sunlight exposure. The dried samples were then dry-sorted and ground into a fine powder. The extraction method used was maceration with a solvent mixture of 96% ethanol and 1% HCl in a ratio of 85:15, conducted over 72

hours with occasional stirring to maintain an acidic environment, thereby enhancing the extraction of anthocyanins. The extract was then filtered using filter paper to obtain the macerate. The macerate was concentrated using a rotary evaporator at approximately 50°C to remove the solvent, yielding a thick extract [4].

## 2.2 Qualitative Analysis of Anthocyanins in Kepok Banana Flower Peel Extract (*Musa paradisiaca* L.)

Qualitative test for anthocyanin compounds was conducted to confirm the presence of anthocyanins in the extract of kepok banana flower peel (*Musa paradisiaca* L.). The qualitative test was carried out using 2N HCl and 2N NaOH reagents. The procedure involved reacting the extract with each reagent separately. The appearance of a red color in the presence of HCl and a greenish-blue color with NaOH indicates the presence of anthocyanin compounds in the extract [4].

## 2.3 Quantitative Analysis of Anthocyanins in Kepok Banana Flower Peel Extract (*Musa paradisiaca* L.)

### 2.3.1 Preparation of Buffer Solutions at pH 1.0 and 4.5

The buffer solution at pH 1.0 was prepared by dissolving 1.49 grams of potassium chloride (KCl) in distilled water in a 100 mL volumetric flask up to the mark. Hydrochloric acid (HCl) was then added until the pH reached 1.0. The buffer solution at pH 4.5 was prepared by dissolving 1.815 grams of sodium phosphate and 0.96 grams of citric acid in distilled water in a 100 mL volumetric flask up to the mark. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were subsequently added to adjust the pH to 4.5 [6].

### 2.3.2 Penentuan Kadar Total Antosianin

The total anthocyanin content was determined using the differential pH method, which involves measuring the absorbance of the extract incubated at pH 1.0 and pH 4.5. Absorbance measurements were performed using a UV-Vis spectrophotometer at wavelengths of 510 nm and 700 nm. The obtained data were then calculated to determine the total anthocyanin content using the following formula [7].

$$A = (A_{510} - A_{700})_{\text{pH } 1.0} - (A_{510} - A_{700})_{\text{pH } 4.5}$$

The total anthocyanin content was then calculated using the formula:

$$\text{Total Anthocyanin (mg/L)} = \frac{A \times \text{MW} \times \text{DF} \times 1000}{\epsilon \times l}$$

Explanation:

|    |                                                                  |
|----|------------------------------------------------------------------|
| A  | = Sample absorbance                                              |
| MW | = Molecular Weight of cyanidin-3-glucoside (449,2 g/mol)         |
| DF | = Dilution Factor                                                |
| l  | = Cuvette Width (1 cm)                                           |
| ε  | = Molar absorptivity of cyanidin-3-glucoside (26.900 L (cm.mol)) |

## 2.4 Formulation of Liquid Lipstick with Kepok Banana Flower Peel Extract

Liquid lipstick formulations containing banana blossom bract (*Musa paradisiaca* L.) extract were prepared with varying extract concentrations of 5%, 7.5%, and 10%. The formulation details are presented in Table 1.

Table 1. Formulation of Liquid Lipstick Preparation with Extract of Kepok Banana Flower Peel

| Ingredients                | Formulas (%) |             |             |             | Function       |
|----------------------------|--------------|-------------|-------------|-------------|----------------|
|                            | F0           | F1          | F2          | F3          |                |
| Banana flower peel extract | -            | 5%          | 7,5%        | 10%         | Coloring agent |
| Castor oil                 | 60           | 60          | 60          | 60          | Emollient      |
| Beeswax                    | 6            | 6           | 6           | 6           | Base           |
| Dimethicon                 | 10           | 10          | 10          | 10          | Emollient      |
| Kaolin                     | 2            | 2           | 2           | 2           | Texturizer     |
| Tocopherol                 | 0,05         | 0,05        | 0,05        | 0,05        | Antioxidant    |
| BHT                        | 0,5          | 0,5         | 0,5         | 0,5         | Antioxidant    |
| Cetyl Alcohol              | 2            | 2           | 2           | 2           | Emulsifier     |
| Titanium dioxide           | 0,5          | 0,5         | 0,5         | 0,5         | White Pigment  |
| Methyl Paraben             | 0,3          | 0,3         | 0,3         | 0,3         | Preservative   |
| <i>Oleum Rosae</i>         | <i>q.s.</i>  | <i>q.s.</i> | <i>q.s.</i> | <i>q.s.</i> | Fragrance      |

## 2.5 Evaluation of Physical Characteristics of Liquid Lipstick Preparation with Kepok Banana Flower Peel Extract (*Musa paradisiaca* L.)

### 2.5.1 Organoleptic Test

Organoleptic test was conducted by visually observing the preparation using the five senses, including its shape, texture, color, and aroma [8].

### 2.5.2 Homogeneity Test

Homogeneity test was performed by applying a sample of the preparation onto a transparent glass slide and observing whether any coarse particles or impurities were present. The preparation is considered homogeneous if no coarse particles are visible on the glass slide [8].

### 2.5.3 Viscosity Test

Viscosity test was conducted by placing the preparation into the viscometer container. The spindle was then attached to the device, and the viscometer was operated. The viscosity reading displayed on the viscometer screen was recorded. An acceptable viscosity value for liquid lipstick preparations ranges from 10,000 to 20,000 cPs [2].

### 2.5.4 pH Test

pH test was performed by placing the sample to be tested into a beaker. The electrode was then immersed into the beaker and left until the device displayed a stable pH reading. The pH measurement was conducted in triplicate for each different extract concentration. The expected pH range for lip colorant preparations should correspond to the physiological pH of the lips, which is 4.5–6.5 [2].

### 2.5.5 Spreadability Test

Spreadability test was conducted by placing 500 mg of the preparation on a transparent glass plate measuring 13 x 25 cm. The preparation was then covered with another glass plate and subjected to weights of 0 g, 50 g, 100 g, and 150 g, each applied for 1 minute. The diameter of the spread preparation was subsequently measured. The preparation is considered to have good spreading ability if the increase in diameter is approximately 5–7 cm [2].

## 2.6 Stability of Liquid Lipstick Preparation with Kepok Banana Flower Peel Extract (*Musa paradisiaca* L.)

### 2.6.1 Cycling Test

The cycling test is a stability test conducted by exposing the preparation to cold (approximately 4°C) and hot (approximately 40°C) temperatures. Each temperature condition is maintained for 24 hours, and the process is repeated for a total of 6 cycles [9].

### 2.6.2 Freeze Thaw

The freeze-thaw test is a stability test conducted by exposing the preparation to cold temperature (-21°C) and room temperature (approximately 25°C). Each temperature condition is maintained for 24 hours, and the process is repeated for a total of 6 cycles [9].

## 2.7 Safety and Acceptability Testing of Liquid Lipstick Preparation with Kepok Banana Flower Peel Extract (*Musa paradisiaca* L.)

### 2.7.1 Irritation Test

Irritation test was conducted to observe any irritation effects on the skin following application. This test was performed topically on volunteers who consented to participate as panelists. The preparation to be tested was applied to the inner upper arm skin and covered with gauze and adhesive tape. The area was then monitored for signs of irritation, such as edema or erythema, at 0, 24, 48, and 72 hours after application [10].

### 2.7.2 Hedonic Test

Hedonic test was conducted to determine the formulation most preferred by the respondents. This test involved collecting questionnaires from panelists regarding the prepared formulations. Panelists were provided with a scale of parameters to interpret their evaluations. The parameters observed in the hedonic test included aroma, texture, and color of each formulation tested. Panelists' responses were recorded using a numerical scale, where 1 corresponds to "Strongly dislike," 2 to "Dislike," 3 to "Neutral," 4 to "Like," and 5 to "Strongly like" [11].

## 3 Result and Discussion

### 3.1 Sample Preparation and Extraction

Maceration is a simple extraction process involving the soaking of powdered plant material in a suitable solvent. This process is carried out at room temperature without any heating. The principle is based on the solvent's ability to penetrate the cell walls and enter the cell cavities containing various active compounds. This method was chosen because it avoids heat application, preventing the degradation or decomposition of natural compounds, such as anthocyanins, which are sensitive to high temperatures. Additionally, this method offers the advantage of simple equipment and procedures [12]. Extraction was performed using a solvent mixture of 96% ethanol and 1% HCl in an 85:15 ratio. Ethanol was selected due to its polarity, availability, and non-toxicity. The addition of 1% HCl was intended to create an acidic environment, as anthocyanin compounds are more stable under acidic conditions [13].

The obtained extract was then used to calculate the percentage yield. Yield is the ratio of the dry weight of the extract to the weight of the starting plant material. The purpose of determining the yield is to indicate the amount of bioactive components contained in the resulting extract. A higher extract yield suggests a greater quantity of active compounds successfully extracted [14]. The yield obtained from the extraction of kapok banana flower peel (*Musa paradisiaca* L.) was 12.87%. This result is higher compared to the yield reported by Sudirman et al. (2024), which was 9.46% [4], Hanum et al. (2023), which was 2.84% [15], and Sundari et al. (2022), which was 11% [16].

### 3.2 Qualitative Test of Anthocyanin

The presence of anthocyanin compounds in the kepok banana flower peel extract was confirmed through qualitative testing. The qualitative anthocyanin test was performed by adding 2N HCl and 2N NaOH. Kepok banana flower peel extract heated with 2N HCl exhibited a stable red color, while the extract treated with 2N NaOH showed a color change from red to green, consistent with the standard qualitative test for anthocyanins [4]. Anthocyanin compounds exhibit color variations depending on the pH level. In acidic conditions at pH 1–2, anthocyanins appear red due to the dominance of the flavylium ion, which is known as the most colored and stable form. When the pH rises above 4 and approaches neutral, anthocyanins undergo structural changes resulting in various colors such as yellow (Chalcone

form), blue (Quinoidal form), or even colorless (Carbinol base), which also leads to a reduction in red color intensity. These color changes indicate the presence of anthocyanins in kepok banana flower peel extract [17].

### 3.3 Determination of Total Anthocyanin Content

Determination of anthocyanin content in the banana blossom bract extract was carried out using the pH-differential method, which utilizes the structural changes of anthocyanins caused by pH variations. In this method, buffer solutions at pH 1.0 and 4.5 were used. At pH 1.0, anthocyanins exist in the red-colored flavylium cation form, whereas at pH 4.5, they convert to the colorless carbinol or hemiketal form. The difference in absorbance between these two pH conditions reflects the anthocyanin concentration, with higher absorbance at pH 1.0 indicating the stability of the compound in strongly acidic environments. Measurements were performed at a wavelength of 510 nm, which is the maximum absorbance wavelength for cyanidin-3-glucoside compounds. A wavelength of 700 nm was used to correct for the presence of turbidity or particulate matter in the samples [13].

The analysis results showed that the extract of banana flower peel (*Musa paradisiaca* L.) contained a total anthocyanin content of 35.958 mg/L. This value is higher than the anthocyanin content reported in several other types of banana flower peel, such as *Musa raja* flower, which contains 30.22 mg/L [18], *Musa batu* flower with 32.29 mg/L, and *Musa ambon* flower with 33.0808 mg/L [19]. Based on the results obtained from the qualitative test and total anthocyanin content analysis, it can be concluded that the extract of kapok banana flower peel (*Musa paradisiaca* L.) contains anthocyanin compounds, with a total anthocyanin content of 35.958 mg/L. Therefore, it can be formulated as a natural coloring agent.

### 3.4 Evaluation of the Physical Characteristics of Liquid Lipstick Formulation with Kepok Banana Flower Peel (*Musa paradisiaca* L.) Extract

Table 2. Results of the Physical Characteristics Evaluation of Liquid Lipstick Formulation with Banana Blossom Bract (*Musa paradisiaca* L.) Extract

| Parameters    | Formulas          |                   |                   |                   |
|---------------|-------------------|-------------------|-------------------|-------------------|
|               | F0                | F1                | F2                | F3                |
| Texture       | Semi-solid        | Semi- solid       | Semi- solid       | Semi- solid       |
| Color         | White             | Very light brown  | Light brown       | Brown             |
| Scent         | Rose              | Rose              | Rose              | Rose              |
| Homogeneity   | Homo-genous       | Homo-genous       | Homo-genous       | Homo-genous       |
| pH            | 5.40 ± 0.011      | 5.31 ± 0.011      | 5.25 ± 0.018      | 5.22 ± 0.020      |
| Viscosity     | 18.919 ± 0.19 cPs | 18.708 ± 0.16 cPs | 17.947 ± 0.10 cPs | 17.059 ± 0.20 cPs |
| Spreadability | 5.26 ± 0.027 cm   | 5.30 ± 0.019 cm   | 5.37 ± 0.020 cm   | 5.48 ± 0.038 cm   |

Organoleptic test is a method used to evaluate the quality of a material or product through the five senses. Parameters assessed in this test include color, aroma, and texture. The results showed that all three formulas had a semi-solid form, a rose-like fragrance, and were homogeneous, as indicated by even distribution and the absence of fine particles. The colors obtained were very light brown for formula F1, light brown for F2, and a darker brown for F3.

Homogeneity test was conducted to ensure that the active compounds and ingredients in the formulation were evenly mixed. The observation was performed visually by applying the formulation onto a transparent glass slide with the aid of light, allowing clearer evaluation of the component distribution within the preparation. The results of the homogeneity test showed that the formulation did not contain coarse particles and was therefore considered homogeneous [8].

Viscosity test aims to determine the thickness level of the formulation. The ideal viscosity range for liquid lipstick is between 10,000 and 20,000 cPs. The viscosity test results showed variations among the formulas, with F1 measuring 18,708 cPs, F2 at 17,947 cPs, and F3 at 17,059 cPs. This difference is related to the concentration of extract dissolved in glycerin before being incorporated into the formulation. The higher the extract concentration, the more the viscosity tends to decrease.



Nevertheless, all formulas remain within the acceptable viscosity range for liquid lipstick preparations, which is 10,000–20,000 cPs [2].

pH test was conducted to ensure that the formulation produced is within the physiological pH range of the lips, which is between 4.5 and 6.5. An inappropriate pH can cause side effects. An excessively acidic condition may lead to inflammation, while an overly alkaline condition can cause dryness due to disruption of the moisture balance in the lip skin [20]. The pH test results showed variation among the formulas, with F1 having a pH of 5.56, F2 at 5.25, and F3 at 5.22. This difference is likely due to the acidic nature of the anthocyanin content in the extract, where an increase in extract concentration tends to lower the formulation's pH. Nevertheless, all formulas remain within the pH range compatible with the physiological pH of the skin, which is 4.5–6.5, thus safe for use as liquid lipstick formulations [2].

Spreadability test aims to describe how the product disperses when applied to the lips. An ideal formulation should spread easily without requiring excessive pressure. Spreadability is considered good if the resulting area ranges between 5 and 7 cm [21]. The test results showed variations, with F1 having a spreadability of 5.30 cm, F2 at 5.37 cm, and F3 at 5.48 cm. The spreadability values were inversely proportional to viscosity, where F1, having a thicker consistency, tended to spread less compared to formulations F2 and F3. Despite these differences, all formulas remained within the acceptable spreadability range for liquid lipstick, which is 5–7 cm [8].

### 3.5 Stability of Liquid Lipstick Preparation from Kepok Banana Flower Peel Extract

Stability testing is an important parameter in quality assessment, aimed at determining the extent to which a preparation maintains its characteristics during the storage and usage period. The evaluation is carried out by monitoring changes in the physical properties of the preparation throughout the storage period. Stability testing using the cycling test method is an approach to simulate temperature fluctuations that may occur during storage from daily to annual periods. This procedure involves alternately storing the preparation at low temperature (4°C in a refrigerator) and high temperature (40°C in an oven), each for 24 hours. One cycle lasts 48 hours, and the test is conducted for a total of 6 cycles or 12 days. During this process, the preparation experiences various thermal stress conditions that may affect its stability. At the end of each cycle, observations are made on the physical characteristics of the preparation, including organoleptic testing, homogeneity, pH, viscosity, and spreadability [22].

Stability testing using the freeze-thaw method aims to simulate extreme temperature conditions, such as storage in warehouses during winter or product shipping in summer. Significant temperature fluctuations can disrupt the formulation's stability. During storage in the freezer, the water in the preparation may crystallize, causing water droplets to come closer together. The formed ice crystals have the potential to penetrate the oil droplets and damage the interfacial membrane, which can trigger emulsion separation, either reversible or irreversible. The test is conducted by alternately storing the preparation at -21°C (freezer) and 25°C (room temperature), each for 24 hours. One cycle lasts 48 hours, and the test is carried out over six cycles (12 days). Evaluation of physical characteristics, including organoleptic testing, homogeneity, pH, viscosity, and spreadability, is performed at the end of each cycle [23].

Table 3. Orgaoleptic and Homogeneity Test Result of the Formulation in Stability Testing

| Cycle | Cycling Test    |        |        |        | Freeze Thaw |        |        |        |
|-------|-----------------|--------|--------|--------|-------------|--------|--------|--------|
|       | F0              | F1     | F2     | F3     | F0          | F1     | F2     | F3     |
|       | Viscosity (cPs) |        |        |        |             |        |        |        |
| 0     | 19.609          | 19.342 | 18.844 | 17.849 | 19.609      | 19.342 | 18.844 | 17.849 |
| 1     | 19.413          | 19.236 | 18.560 | 17.636 | 19.449      | 19.271 | 18.614 | 17.671 |
| 2     | 19.027          | 18.702 | 17.849 | 17.067 | 19.200      | 18.987 | 18.133 | 17.422 |
| 3     | 18.491          | 18.204 | 17.529 | 16.213 | 19.164      | 18.702 | 17.742 | 17.102 |
| 4     | 18.807          | 18.382 | 17.564 | 16.605 | 19.314      | 18.635 | 17.778 | 17.031 |
| 5     | 18.631          | 18.311 | 17.422 | 16.462 | 18.951      | 18.489 | 17.600 | 16.960 |
| 6     | 18.453          | 17.991 | 17.351 | 16.356 | 18.631      | 18.311 | 17.422 | 16.604 |
|       | pH              |        |        |        |             |        |        |        |
| 0     | 5,31            | 5,23   | 5,16   | 5,13   | 5,31        | 5,23   | 5,16   | 5,13   |

|                    |      |      |      |      |      |      |      |      |
|--------------------|------|------|------|------|------|------|------|------|
| 1                  | 5,25 | 5,21 | 5,17 | 5,14 | 5,27 | 5,27 | 5,2  | 5,16 |
| 2                  | 5,31 | 5,24 | 5,21 | 5,16 | 5,40 | 5,32 | 5,26 | 5,25 |
| 3                  | 5,37 | 5,32 | 5,28 | 5,27 | 5,48 | 5,31 | 5,29 | 5,26 |
| 4                  | 5,45 | 5,35 | 5,34 | 5,33 | 5,48 | 5,35 | 5,27 | 5,23 |
| 5                  | 5,52 | 5,44 | 5,35 | 5,32 | 5,45 | 5,33 | 5,26 | 5,23 |
| 6                  | 5,50 | 5,43 | 5,33 | 5,31 | 5,43 | 5,29 | 5,24 | 5,21 |
| Spreadability (cm) |      |      |      |      |      |      |      |      |
| 0                  | 5,18 | 5,23 | 5,28 | 5,38 | 5,18 | 5,23 | 5,25 | 5,38 |
| 1                  | 5,21 | 5,26 | 5,30 | 5,40 | 5,20 | 5,21 | 5,30 | 5,40 |
| 2                  | 5,25 | 5,28 | 5,33 | 5,45 | 5,21 | 5,25 | 5,36 | 5,41 |
| 3                  | 5,31 | 5,35 | 5,39 | 5,58 | 5,25 | 5,28 | 5,38 | 5,43 |
| 4                  | 5,28 | 5,31 | 5,45 | 5,55 | 5,23 | 5,30 | 5,35 | 5,45 |
| 5                  | 5,33 | 5,33 | 5,46 | 5,58 | 5,29 | 5,35 | 5,40 | 5,50 |
| 6                  | 5,38 | 5,43 | 5,49 | 5,60 | 5,31 | 5,38 | 5,45 | 5,59 |

The results of the organoleptic evaluation, based on color, texture, and aroma parameters during stability testing using both the cycling test and freeze-thaw methods, showed that formulas F0, F1, and F2 did not experience any organoleptic changes after six cycles, in terms of color, aroma, or texture. However, formula F3 exhibited slight color fading at the final cycle of testing. This may be caused by the degradation of active compounds due to high storage temperatures. The homogeneity test from both stability methods showed that all formulas (F0, F1, F2, and F3) remained homogeneous after storage, as indicated by the absence of coarse particles in the preparations.

The results of the viscosity test using the cycling test and freeze-thaw methods are presented in Table 3. Both tests showed a decrease in viscosity over time, presumably due to minor degradation of the ingredients in the formulation. However, the viscosity values obtained for F0, F1, F2, and F3 remained within the acceptable range of 10,000–20,000 cPs [2]. The data from both cycling test and freeze-thaw cycles were analyzed using One Way ANOVA with the hypothesis  $H_0: S_0 = S_1 = S_2 = S_3 = S_4 = S_5 = S_6$ , which means that there is no difference in viscosity values across all cycles. The results showed a significance value of 0.240 ( $>0.05$ ) for the cycling test and 0.525 ( $>0.05$ ) for the freeze-thaw test, indicating that the  $H_0$  is accepted. In other words, there were no significant differences in the viscosity values across the cycles. These results support the hypothesis that the viscosity of the formulation remains stable over six storage cycles in both the cycling test and freeze-thaw methods.

The results of the pH test using the cycling test method, as shown in Table 3, indicate that in formula F1, an increase in pH begins to occur starting from the third cycle, with a significant spike observed in the fifth cycle. The pH values obtained during the freeze-thaw cycles in Table 3 tend to increase with each cycle. These pH changes are presumed to be influenced by external factors such as room temperature during testing and suboptimal storage conditions, which may lead to degradation of the formulation's components [24]. Despite the fluctuations, all pH values remain within the acceptable range for lip color formulations, which is between 4.5 and 6.5 [2]. The data for cycling test cycles were analyzed using the Kruskal-Wallis test, while the freeze-thaw cycles were analyzed using One-Way ANOVA with the hypothesis  $H_0: S_0 = S_1 = S_2 = S_3 = S_4 = S_5 = S_6$ , which means there is no difference in pH across all cycles. The result for the cycling test showed a significance value of 0.003 ( $<0.05$ ), indicating that the  $H_0$  was rejected and there is a significant difference in pH values across the cycles. This result does not support the hypothesis that the formulation's pH remains stable over six cycles of storage under the cycling test method. Meanwhile, a significance value of 0.297 ( $>0.05$ ) was obtained for the freeze-thaw method, indicating that the  $H_0$  was accepted, or there is no significant difference in pH values across the cycles. This analysis supports the hypothesis that the formulation's pH remains stable during six cycles of freeze-thaw storage.

Spreadability test using the cycling test method, as shown in Table 3, indicated that all formulas (F0, F1, F2, and F3) experienced an increase in spreadability during the third cycle. Meanwhile, the results from the freeze-thaw method, also presented in Table 3, showed a gradual increase in spreadability as the number of cycles progressed. This increase is likely related to the decrease in viscosity due to minor degradation of components within the formulation base, making the product easier to spread. Despite the



fluctuations, the spreadability values of all formulas remained within the acceptable standard range of 5–7 cm [2]. The data for both cycling test and freeze-thaw cycles were analyzed using One Way ANOVA with the null hypothesis  $H_0: S_0 = S_1 = S_2 = S_3 = S_4 = S_5 = S_6$ , which means that the spreadability results across all cycles are not significantly different. The significance values obtained were 0.090 ( $>0.05$ ) for the cycling test and 0.192 ( $>0.05$ ) for the freeze-thaw test, indicating that the  $H_0$  is accepted or that there is no significant difference in spreadability values across the cycles. These results support the hypothesis that the spreadability of the formulation remained stable over six storage cycles using both the cycling test and freeze-thaw methods.

Based on the stability test results, the liquid lipstick formulation containing kepok banana flower peel extract demonstrated stable physical characteristics, such as texture and aroma throughout six storage cycles. Although a gradual fading in color was observed at the end of the test in the formula of the highest concentration, all three formulas overall meet the quality standards for liquid lipstick preparations. In addition, the parameters of pH, spreadability, and viscosity did not show significant changes, despite being subjected to varying temperature treatments during the stability evaluation.

### 3.6 Safety and Acceptability Testing of Liquid Lipstick Preparation with Kepok Banana Flower Peel Extract

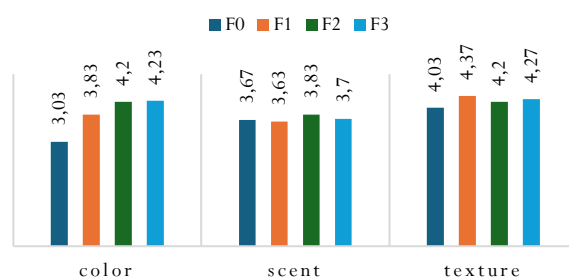
#### 3.6.1 Irritation Test

Irritation test was conducted to ensure that the formulation does not cause adverse side effects on the skin. Irritation refers to an inflammatory reaction on the skin or mucous membranes resulting from repeated or prolonged exposure to certain chemical substances [25]. The test was carried out using the skin patch test method by applying the formulation to the upper arm area and covering it with a patch. The formulation was left under occlusion for 4 hours before removal, with this duration intended to prevent irritation exceeding the mild category [10]. Observations of skin conditions were made at 0, 24, 48, and 72 hours after application, as reactions to irritants typically appear within 12 to 48 hours post-exposure. The reactions observed included erythema and edema, with erythema indicated by skin redness accompanied by mild lesions, and edema characterized by swelling or raised skin surfaces compared to normal conditions [26].

The irritation test conducted on 30 panelists showed no symptoms of erythema (redness) or edema (swelling) on the skin. All tested formulations (F0, F1, F2, and F3) did not cause any irritation reaction after 4 hours of application and based on observations at 0, 24, 48, and 72 hours post-application. Therefore, the irritation score obtained was 0, indicating that all formulations are classified as non-irritating.

#### 3.6.2 Hedonic Test

The hedonic test is an evaluation method aimed at determining the panelists' level of preference for the characteristics of the formulation. In this test, panelists are asked to provide subjective ratings based on their preferences for parameters such as aroma, color, and texture of each formulation using a numerical scale provided in the questionnaire. The panelists' responses are recorded on a numerical scale where 1 represents "Dislike very much," 2 is "Dislike," 3 is "Neutral," 4 is "Like," and 5 is "Like very much" [11].



Picture 1. Hedonic Test Result

#### 4. Conclusion

Liquid lipstick formulation can be developed using natural dye derived from the extract of Kepok banana heart peel (*Musa paradisiaca* L.), which contains anthocyanin compounds at a concentration of 35.958 mg/L. Formulations with extract concentrations of 0%, 5%, 7.5%, and 10% resulted in physical characteristics of white, very light brown, light brown, and brown colors, respectively, all with a rose scent and semi-solid texture. The viscosity values ranged between 10,000–20,000 cPs, pH values between 4.5–6.5, and spreadability between 5–7 cm, all meeting the required specifications. Stability studies on all formulations showed physical stability, marked by no changes in color, aroma, texture, and homogeneity. The viscosity, pH, and spreadability values indicated formulation stability, as all remained within the specified ranges after six cycles of two different stability tests. None of the formulations (F0, F1, F2, and F3) caused irritation reactions such as erythema or edema. The most preferred formula among 30 panelists was F2.

#### 5. Declarations

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##### 5.2 Author contributions

All authors were involved in the development and writing of this article.

##### 5.3 Ethics

Ethical clearance was obtained from the Health Research Ethics Committee of the Faculty of Pharmacy, Mulawarman University, with the ethical approval number No. 272/KEPK-FFUNMUL/EC/EXE/10/2024.

##### 5.4 Conflict of Interest

The authors declare no conflicts of interest during the research and the preparation of this article.

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