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The Effect of HPMC Concentration as a Gelling Agent on Color Stability of Copigmented Blush Gel Extract of Purple Sweet (*Ipomoea Batatas* (L.) Lam.)

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Purple sweet potato contains anthocyanin pigments that can be used as natural dyes. However, the use of anthocyanins as dyes has less stable properties. Anthocyanin stability can be increased by the addition of copigments. Copigment that can be used is apple extract at pH 4.6. In this study, a blush was made. One of the blush dosage forms that can be made is gel. Blush gel can provide a cool feeling, easy to wash, soft, and not sticky on the skin. In the creation of gels, the gelling agent is one of the components that affect the characteristics of gel preparations. HPMC is a gelling agent used in the manufacture of blush gel preparations. This study aims to determine the effect of increasing the concentration of HPMC as a gelling agent on the color stability of the copigmented purple sweet potato extract blush gel. The preparations were made in 3 formulas with varying concentrations of gelling agent (F1) 1%, (F2) 1.5%, and (F3) 2%. The evaluation carried out included testing the physical characteristics of the gel, namely organoleptic test, pH test, dispersion test, phase separation test, color stability test, and preference test. The results showed that the evaluation of blush gel preparations met the requirements of pharmaceutical standards with the pH test value of blush gel preparations being 4.72 to 5.84 and the spreadability test value being 5.26 cm to 6.48 cm. The results of the color stability test on the three formulas showed that there were significant differences. The increase in the concentration of HPMC as a gelling agent on the color stability of the copigmented purple sweet potato extract blush gel was unstable during 28 days of storage.

1. Introduction

Blush is a cosmetic preparation that is used to color the cheeks with an artistic touch so that it can improve aesthetics in facial makeup [1]. Blush is available in various color choices, but blush products on the market use synthetic dyes as their dyes [2]. Concerns about the safety of using synthetic dyes encourage the development of natural dyes. Natural dyes were chosen because they are safe if used in the long term. One of the natural dyes that have the potential to replace synthetic dyes is anthocyanins found in purple sweet potato [3].

Purple sweet potato is used as a natural dye because it contains 11.051 mg/100 gram of anthocyanin which is higher than other varieties [4]. In addition, the anthocyanins in purple sweet potatoes can be used as antioxidant compounds that are very useful for the body. The use of anthocyanins as dyes is less stable to oxygen, light, pH, sugar, and temperature [5]. Hence, it is necessary to make efforts to improve its stability. To increase the stability of anthocyanins, it is necessary to add copigmentation [6].

Copigmentation can naturally improve the color of anthocyanins, where the stability and color strength of anthocyanins can be increased by the addition of extracts from different plants that are rich in copigments [7]. One of the compounds that can be used as a copigment is apple extract at pH 4.6 [5]. Apple extract was chosen as a copigment because apples contain the highest amount and activity of antioxidants, as well as the largest amount of flavonoids (quercetin) in apples [8]. With the addition of apple extract at pH 4.6, it is expected to get the desired color, namely a purplish red color [5]. Copigmentation can increase the stability of anthocyanins by way of copigment compounds forming complex bonds with anthocyanins, which then reduces the interaction of anthocyanins with water molecules. The interaction of anthocyanins with water molecules causes anthocyanins to be degraded [9].

One of the blush dosage forms is gel. The gel was chosen because it has the advantage of providing a cool feeling on the skin, easy to wash, soft, and not sticky to the skin [10]. In the gel, there is a gelling agent which is a very important component in the manufacture of gel preparations. HPMC gel base is a gelling agent that is often used in the production of cosmetics and drugs as it can produce a clear gel, easily soluble in water, and has low toxicity [11]. HPMC is a semi-synthetic gelling agent derived from cellulose which is resistant to phenol, can form a clear gel, is neutral, and has a stable viscosity on long-term storage [12]. In addition, HPMC is stable at acidic pH and alkaline pH, which makes it very suitable for anthocyanin compounds which are stable at acidic pH. In this context, it aims to examine the effect of increasing the concentration of HPMC as a gelling agent on the color stability of the pigmented purple sweet potato extract blush gel.

2. Material and Methods

2.1 Preparation Extract

Purple sweet potato (*Ipomoea batatas* (L.) Lam.) powder was obtained from Institut Pertanian Bogor, West Java. 500 grams of purple sweet potato powder was extracted with 3.5 liters of 96% of ethanol. Then, HCl solution was added little by little which makes it to have a pH in the range of 1 to 3. Extraction was carried out by maceration for 3 days, which was followed by another maceration for 2 days while stirring occasionally. The liquid extract obtained was thickened with a rotary evaporator at a temperature of 40 ºC to obtain a thick extract [13].

2.2 Phytochemical Screening of Ethanolic Extract

The phytochemical compounds of kencur (aromatic ginger, *Kaempferia galanga*) ethanolic extract, such as anthocyanins were qualitatively identified following standard procedures described in the Harborne and Indonesian Herb Pharmacopoeia [3].

2.3 The Creation of Blush Gel of Purple Sweet Potato (Ipomoea batatas (L.) Lam.)

The ingredients used were weighed. The pigment was prepared by means of purple sweet potato extract, apple extract was added to a pH of 4.6 and then mixed until homogeneous (mixture 1). Gel base was made by dispersing HPMC in Aqua distillata which was then developed for 24 hours. Methyl paraben and propyl paraben were dissolved into propylene glycol, which was then mixed into the gel preparation that had been developed, then grounded until homogeneous [12]. Mixture 1 which was grounded, was then mixed until it was homogeneous.

Table 1. Purple Sweet Potato (*Ipomoea batatas* (L.) Lam.) Blush Gel Formula

Material Name	Formula (% w/v)			Function
	F1	F2	F3	
Purple sweet potato extract	12	12	12	Dye
Apple Extract	0,63	0,63	0,63	Copigment
HPMC	1	1,5	2	Gelling agent
Propylene glycol	15	15	15	Humectants
Methyl paraben	0,18	0,18	0,18	Preservative
Propylparaben	0,015	0,015	0,015	Preservative
Aquadest ad	100	100	100	Solvent

2.4 The Evaluation of Purple Sweet Potato (Ipomea batatas L.) Blush Gel Preparation

Organoleptic test was carried out by observing the physical appearance of the purple sweet potato blush gel visually such as smell, shape, and color [14]. The pH test of the gel preparation was measured using a pH measuring device by dipping it into the gel sample. The pH value of a good gel preparation was pH 4.5 to 6.5 and did not irritate the skin [10]. The homogeneity test was carried out by weighing the gel as much as 0.1 grams and was then smeared on a piece of transparent glass and was then observed. Homogeneity was indicated by the absence of coarse grains [14]. Spreadability test of a total of 1 gram of gel preparation was placed on a transparent glass, and was given a weight of 125 grams on top. Afterwards, the diameter formed after 1 minute was measured [15].

2.5 Separation Test Phase

Freeze-Thaw by means of preparations was stored at two different temperatures, namely at a temperature of 4 ºC for 48 hours and followed by a temperature of 45 ºC for 48 hours (1 cycle). The test was carried out in 6 cycles, whereby each cycle was observed whether there was a phase separation or not in the gel [16].

Centrifugation by weighing as much as 10 grams of each preparation, was put into a centrifugation tube, and then centrifuged at 3750 rpm for 5 hours. Thereafter, an observation was made whether there was separation [17].

2.6 Color Stability Test

A 5 ppm pure anthocyanin solution with 2 solvents was made, namely 90% of ethanol solvent and water. We then measured the maximum wavelength between 400 and 800 nm at t05, t10, and t15, to see if the value was fixed at that time (storing the measuring flask in a cupboard to avoid direct light). It was important to make sure the measured absorbance is between 0.2 and 0.8. After obtaining the data, the measurement conditions (type of solvent and measurement time lag) were determined which were suitable for the color stability test [4].

The reading of the color stability in the blush gel preparation was carried out by weighing the preparation as much as 1 gram and then putting it into 250 ml volumetric flask dissolved in 90% ethanol to the limit mark. The absorbance was then identified using UV-Vis spectrophotometry at a wavelength of 544 nm with absorbance between 0.2 to 0.8. Observation of color stability was carried out during 28 days of storage [5].

2.7 Data Analysis

The observed data from the dispersion test and color stability test were then analyzed using one-way ANOVA. If there was a significant difference between the formulas, then it was continued with the Tukey HSD test with a 95% confidence level to see if the formula has a significant difference or not.

3. Results and Discussion

Purple sweet potato was macerated using ethanol as solvent. Ethanol solvent was chosen because ethanol is a universal solvent. This solvent can dissolve almost all organic compounds in the sample, both polar compounds and non-polar compounds [2]. Therefore, it is very suitable to find anthocyanins contained in purple sweet potatoes which are polar. In the extraction process, HCl was added so that it became an acidic atmosphere. With an acidic environment, it can denature plant cell membranes and it can dissolve anthocyanin pigments, making them able to leave the cells and prevent flavonoid oxidation [4]. The calculation of the extract yield was then carried out. Yield is the ratio of the weight of the extract obtained with the weight of the simplicia powder. The yield of purple sweet potato extract obtained was 10.5375%.

Anthocyanin qualitative test aims to determine the truth that purple sweet potato extract contains anthocyanins. In the anthocyanin test, purple sweet potato extract was added with HCl. The results of the observations obtained a red color. The red color was formed by flavylium cations, where the number of methoxy groups in the anthocyanin structure was more dominant than the hydroxyl groups. Then, the NaOH solution was added little by little. Observations showed that the green color was slowly fading. At alkaline pH, it turned bluish green due to the carbinol structure of pseudobase [3]. When the pH was higher, the chalcone structure was formed which caused anthocyanins to lose their red color due to the formation of quinonoidal anions [5]. From the observations obtained when compared with the requirements that have been set, it can be concluded that the purple sweet potato extract contained anthocyanin compounds.

The organoleptic test was carried out by observing the physical appearance of the purple sweet potato extract blush gel preparation with the addition of a copigment, namely apple extract at pH 4.6 by visually observing the shape, color, and smell of the blush gel preparation. The results showed a semisolid form, a purplish red color was obtained due to the addition of a copigment, namely apple extract at pH 4.6 and the distinctive odor of the preparation.

In observing the pH of blush gel preparations with the addition of copigment, namely apple extract at pH 4.6, observations were made for 28 days. From these observations, the preparation experienced instability due to the presence of CO2 that was attracted to the preparation, which then reacted with water to release H+. Another aspect that caused changes in the pH value can occur because it was affected by the medium that was decomposed by high temperatures during manufacture or storage time which produces acids or bases. In addition, changes in pH are also caused by environmental factors such as temperature, poor storage, and others [14]. The preparation of purple sweet potato blush gel with the addition of copigment, namely apple extract at pH 4.6, experienced instability during 28 days of storage. However, these preparations still meet the requirements of a good skin physiological pH of 4.5 to 6.5 [10].

In the observation of the homogeneity test of all these formulas, there were no coarse grains placed on a pair of glass plates which were visually observed. When compared with the requirements for good homogeneity, a preparation must show a homogeneous preparation and there are no coarse grains so that it can be concluded that all preparations are homogeneous [18].

In the observation of blush preparations, purple sweet potato extract with the addition of copigment, namely apple extract at pH 4.6 has good dispersion. The requirements for good dispersion for gel preparations are 5 to 7 [14]. It can be concluded that the blush gel preparation of purple sweet potato extract with the addition of copigment, namely apple extract at pH 4.6, met the pharmaceutical requirements, although the dispersion values were different due to differences in the concentration of HPMC. The higher the HPMC concentration, the smaller the dispersion obtained.

Table 2. Spreadability Test Results

Formula	Yield Spread (cm)
F1	6, 48 ± 0,18
F2	6,18 ± 0,08
F3	5,26 + 0,08

Based on the results of statistical analysis data, the normality test using the Kolmogrov Smirnov test obtained a significant value > 0.05 which indicated that the data was normally distributed. Thereafter, the homogeneity test was carried out with a significant value > 0.05 which indicated that the data was homogeneously distributed. It was followed by the one-way ANOVA test with a significant value < 0.05 which demonstrated that the data of the dispersion test value of the three formulas had significant differences among the formulas. It was then continued with the Tukey HSD test to see if the formula had a significant difference or not. The Tukey HSD test obtained significant values from the three formulas, namely in F1 there was a significant difference in F3. In F2, there was a significant difference in F3. Furthermore, in F3 there was a significant difference both in F1 and F2.

The freeze-thaw test was carried out to see if there was a phase separation in the blush gel preparation of purple sweet potato extract with the addition of a copigment, namely apple extract at pH 4.6 with two different temperatures. From the observations for 6 cycles, all formulas did not experience phase separation. At 4°C it crystallized so that the consistency of the preparation became thicker. This was because the preparation tended to shrink at low temperatures, making the particles combine to form tighter bonds between themselves. As a result, the viscosity increased and the water rate decreased [12]. However, after being placed at a temperature of 45°C, the preparation returned to its original shape and there was no phase separation. It can thus be concluded that the blush gel preparation of purple sweet potato extract was stable.

Centrifugation is a phase separation test with very fast shaking under the influence of gravity. The preparation was centrifuged at 3750 rpm for 5 hours and the result was equivalent to the effect of gravity for one year [15]. The results of the observations showed that in all the blush gel formulas of purple sweet potato extract with the addition of copigment, namely apple extract at pH 4.6, there was no phase separation. This indicates that the preparation was stable and was expected to be stable equivalent to the effects of gravity for one year. This showcased that the blush gel preparation of purple sweet potato extract with the addition of apple extract copigment at pH 4.6 was stable.

The color stability test aims to measure or see the color stability of each formula. Testing of blush gel preparations of purple sweet potato extract with the addition of copigment, namely apple extract at pH 4.6, was carried out using a UV-Vis spectrophotometer with a wavelength of 544 nm. Anthocyanins could specifically absorb light in the ultraviolet (UV) to violet absorption region, but were stronger in visible light in the spectrum. The presence of conjugated double bonds in the chromophore group contained in the anthocyanin structure makes anthocyanins able to absorb light in visible light. Thus, this is the reason why the color stability test readings were used in a UV-Vis spectrophotometer with a wavelength of 544 nm located in visible light [19].

Anthocyanins as dyes are less stable to oxygen, light, pH, sugar, and temperature [5]. With the addition of apple extract copigment at pH 4.6 in the blush gel preparation, it was expected to increase the color stability of anthocyanins, with the desired color being purplish red. However, based on observations for 28 days, the three formulas experienced changes in absorbance when reading the UV-Vis spectrophotometry. This could be caused by factors that affect color stability, namely pH, light, low temperature, copigment, metal ions, oxygen, enzymes, concentration, and storage time so that anthocyanins were degraded. The instability of anthocyanins caused the compound to easily undergo hydrolysis at the glycosidic bond and the aglycone ring became open, thus forming a labile aglycone, as well as a colorless carbinol and chalcone groups [19].

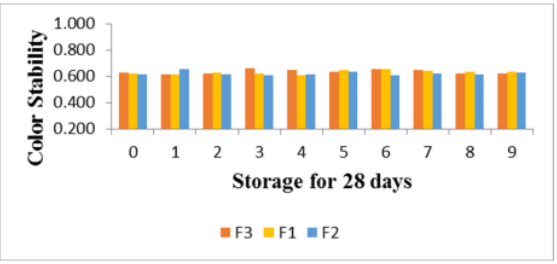

Figure 1. Observation Results of Color Stability Test

Based on the results of statistical analysis data, each formula was tested for normality using the Kolmogrov Smirnov test. A significant value was obtained > 0.05 which indicated that the data was normally distributed. It was then continued with the homogeneity test. The significant value obtained > 0.05 indicated that the data was homogeneously distributed. We then proceeded with testing with the one-way ANOVA parameter which obtained a significant value < 0.05, indicating the color stability value data of the three formulas. There were significant differences among the formulas. We continued with the Tukey HSD test, which obtained a significant value from the three formulas, namely > 0.05, demonstrating that there is no significant difference in the formula during 28 days of observation. It can be concluded that the increase in the concentration of HPMC as a gelling agent on the color stability of the copigmented purple sweet potato extract blush gel did not experience stability during 28 days of storage.

4. Conclusion

Based on the results of the study, the results obtained from the statistical analysis of color stability found that there were significant differences in each formula. It can be concluded that the increase in the concentration of HPMC as a gelling agent on the color stability of the copigmented purple sweet potato (*Ipomoea batatas* (L.) Lam.) blush gel extract did not experience color stability during 28 days of storage.

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