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Mangosteen (*Garcinia mangostana* L.) Rind Extract Lip Balm Formulation and Evaluation

Formulasi dan Evaluasi Sediaan Lip Balm dari Ekstrak Kulit Manggis (*Garcinia mangostana* L.)

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A R T I K E L I N F O

Keyword:
Xanthone, lip balm, stability test

Kata Kunci:
Xanton, *lip balm*, uji stabilitas

A B S T R A K

Research on lip balm was conducted to determine how the formulation affected its characterization and stability when containing mangosteen rind extract. Thin-layer chromatography, microscopy, and phytochemical screening procedures were used to evaluate mangosteen rind extract. The freeze-thaw technique was used to create the lip balm formulation and to test its stability. The yield of coccal crystals made from mangosteen rind extract, which had an Rf value of 0.39, was 11.37% w/w. The concentration of the extract in the form of F0 (blank), F1 (2.5%), F2 (5%), F3 (7.5%), and F4 (10%) was varied to create lip balm compositions. The ingredients are combined into a base that has already been adjusted to create lip balm. Evaluations of lip balm preparations were conducted using organoleptic tests, homogeneity tests, pH tests, melting point tests, spreadability tests, centrifugation tests, and freeze-and-thaw tests. The results of the physical characteristics of the lip balm preparation were good and met the requirements based on the organoleptic test, homogeneity test, pH test, melting point test, and spreadability test. The stability test showed that the lip balm preparation was physically stable, with no change after 6 cycles. The normality test shows a p-value > 0.05, indicating that the formulation is normally distributed. Homogeneity test >0.05, which means there is no significant difference between the formulas. The one-way ANOVA test shows p < 0.05, indicating an effect on the pH of all formulas.

A B S T R A C T

Penelitian lip balm telah dilakukan untuk mengetahui pengaruh formulasi terhadap karakterisasi dan stabilitas lip balm ekstrak kulit manggis. Ekstrak kulit manggis dikarakterisasi berupa uji skrining fitokimia, kromatografi lapis tipis (KLT), dan mikroskop. Formulasi lip balm dibuat dan uji stabilitas menggunakan metode freeze and thaw. Rendemen ekstrak kulit manggis diperoleh 11,37% b/b dengan nilai Rf 0,39 berbentuk kristal kokus. Sediaan lip balm dibuat dengan memvariasikan konsentrasi ekstrak berupa F0 (blanko), F1 (2,5%), F2 (5%), F3 (7,5%), dan F4 (10%). Proses pembuatan lip balm yaitu pencampuran semua bahan yang digunakan ke basis yang telah dioptimasi sebelumnya. Evaluasi yang dilakukan terhadap sediaan lip balm berupa uji organoleptis, uji homogenitas, uji pH, uji titik leleh, uji daya sebar, uji sentrifugasi, dan uji freeze and thaw. Hasil karakteristik fisik sediaan lip balm baik dan memenuhi persyaratan berdasarkan parameter pemeriksaan fisik pada uji organoleptis, uji homogenitas, uji pH, uji titik leleh, uji daya sebar. Uji kestabilan menunjukkan sediaan lip balm stabil secara fisik dan tidak adanya perubahan setelah pengujian menggunakan 6 siklus. Uji normalitas menunjukkan hasil >0,05 yang berarti formulasi berdistribusi normal. Uji homogenitas >0,05 yang berarti tidak terdapat perbedaan yang signifikan antara formula, Uji anava satu arah menunjukkan hasil p<0,05 menyatakan adanya pengaruh antara pH semua formula.

1. Introduction

The word "cosmetics" comes from the Greek word "kosmein," which means "decorated". Since the beginning of time, people have been aware of cosmetics. Cosmetics are defined as ingredients or products used on the skin, hair, nails, and lips (especially for cleansing, perfuming, adjusting appearance, and improving body odor), as well as for safeguarding or maintaining the body in good condition. This concept is found in Regulation of the Head of the Republic of Indonesia Food and Drug Agency Number 19 of 2015. One cosmetic of concern to the public today is lip cosmetics (Hanum et al., 2021).

The appearance of the lips is one facial feature that affects how the face is perceived as attractive. The lips' epidermis contains sweat glands and hair follicles, which help protect the lips from the environment (Anugrah *et al*., 2021). The lips are extremely sensitive to environmental factors and various cosmetics and lip care products, which may damage the skin and cause discomfort when the lips become dry, chapped, and lack color. To address lip skin problems, lip moisturizing cosmetics, such as lip balm, can be used (Yusuf, 2019).

A cosmetic product used on the lips is lip balm. Its major ingredients are wax, fat, and oil, and it works by producing an immiscible oil coating on the lips' surface to increase moisture and prevent dryness. Lip balm forms a layer that shields the lips from the surface layer (Ambari *et al*., 2020). Mangosteen rind is included in the lip balm formulation because it contains the active chemical, a compound xanthone, that has antioxidant effects. Free radical damage in skin cells is able to be repaired with antioxidants (Sofyan, 2016).

Based on research (Putri, 2015), Mangosteen rind (*Garcinia mangostana* L.) contains xanthones with anti-ageing, anti-hypertension, immunomodulating, osteoporosis-prevention, digestive system

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support, anti-inflammatory, antidiabetic, and antioxidant properties.

The purpose of this study is to produce Mangosteen rind extract and to evaluate the lip balm's stability and physical characteristics. Lip balm formulations were tested for organoleptic properties, homogeneity, pH, melting point, spreadability, centrifugation, and freeze-thaw stability.

2. Research Method

Materials

The materials used in this research was mangosteen rind from the Spice and Medicinal Plants Research Institute (BALITTRO), 96% ethanol (Solvent ethanol TK), beeswax, almond oil, VCO (Virgin Coconut Oil), honey, HCL 2N, Dragendorff reagent (Brataco), Mayer reagent (Brataco), Liebermann-Burchard reagent (Brataco), acetone (Merck), boric acid (Merck), oxalic acid (Merck), ether (Merck), 10% iron III chloride solution (Merck), acetic acid (Merck), sulfuric acid (Merck), chloroform (Merck), distilled water, aluminum foil, and plastic wrap.

The equipment utilized includes standard laboratory glassware (Iwaki), hot plate (Thermo Scientific), digital pH meter (Ezdo), analytical digital balance (Biobase), refrigerator (Aqua), oven (Memmert), rotary evaporator (Buchi), water bath (B-One), UV cabinet (Camag), Silica Gel 60 GF254 TLC KLT plate (Merck), vacuum desiccator (Normax), centrifuge, thermometer, and lip balm packaging.

Methods

Extract preparation

Mangosteen rind extraction was performed using the maceration method with 96% ethanol solvent. The extraction was carried out at a 1:10 ratio. The first extraction was performed by soaking the simplisia for 5 days at a 1:7 ratio, followed by remaceration for 2 days with the addition of the remaining solvent. The filtrate was concentrated using a rotary evaporator at 50 rpm and 40℃ (Rizikiyan et al., 2021). The extract was then added to lip balm formulations in a variety of concentrations, including 2.5%, 5%, 7.5%, 10%, and blank

Phytochemical screening

Test solutions for phytochemical screening were prepared by dissolving 500 mg of mangosteen peel extract in 50 mL of 96% ethanol (Table 1) (Puspitasari et al., 2013).

Microscopic identification

Identification using a microscope is done by preparing a sample, then dripping a liquid such as chloral hydrate or aquadest-glycerin onto a glass slide, then slowly covering it with a cover glass. Observe under a microscope at the lowest magnification first to see specific identifying fragments, then compare with the reference (Rahayu et al., 2020).

Lip balm formulation

Lip balm was made at room temperature. Beeswax was divided into equally small slices and shreds of 10 mesh size. VCO and almond oil were combined in a beaker. The mixture was then topped off with honey and distilled water, and everything was thoroughly blended. Once the mixture was homogeneous, beeswax was gradually added and allowed to melt and dissolve. After the final step of adding mangosteen rind extract and stirring until homogeneous, the lip balm is allowed to sit for 10 minutes before being formed in a plastic container. This method was applied to temperature optimization at 60°C and 100°C. The lip balm is examined and preserved in a cool, dry setting (Table 2) (Kusrini et al., 2020).

Organoleptic test

The preparation's exterior qualities, such as its color, scent, texture, and other potential changes throughout development, are observed during organoleptic testing (Suleman et al., 2022).

Homogeneity test

Preparation is tested by spreading the mixture onto one glass, covering it with another glass, and assessing its homogeneity (Putridhika et al., 2022).

The pH test

This test uses a calibrated pH meter. The sample was weighed at 1 g, dispersed in 10 mL of distilled water, and heated on a hot plate. When the preparation's pH is between 4.5 to 7.0, the lip pH range, it qualifies (Imani & Shoviantari, 2022).

Melting point test

The melting temperature test uses an oven with an initial temperature of 50℃, increased by 5℃ every 5 minutes. The temperature at which the preparation started to melt was observed. The preparation is said to be good if the preparation melts at 50-70℃ (Putridhika et al., 2022).

Spreadability test

A few grams of each lip balm ingredient were weighed for the test and placed in the middle of two flat glasses. Following the addition of 125 g of load, the diameter of the lip balm that spread was measured after it had stood for 1 minute. Good spreadability for semi-solid preparations is 5-7 cm (Jessica et al., 2018).

Centrifugation test

Each preparation was mixed to 4 g, then centrifuged for 10 minutes at 5000 rpm. The presence of separation was then verified (Widayanti et al., 2014).

The freeze-thaw method

The preparation must first be kept at 4 °C, then heated to 45 °C to complete the phase-separation cycle using the freeze-thaw method. The solution was placed in a vial, sealed, and kept at 4°C for 24 hours (1 cycle) before being transported to 45°C for 24 hours. Six cycles of the test were run while tracking any organoleptic changes (Widayanti et al., 2014).

Data analysis

One-way ANOVA was used to statistically evaluate the effects of modifications to lip balm formulations, including the addition of mangosteen rind extract.

3. Result and Discussion

The mangosteen rind sample used in this study was obtained from the Spice and Medicinal Plants Research Agency (BALITRO). The mangosteen rind has a dark color, a unique mangosteen rind smell, and a bitter taste, according to the macroscopic test. There are fragments of sclereids, endocardium, parenchyma, and mesocardium in the mangosteen rind (Kementerian Kesehatan RI, 2017). Microscopic identification revealed that the mangosteen rind powder contains fragments of sclereids (Figure 1). The difference in fragment shape between the powdered simplicia and the mangosteen rind extract is reflected in a greater number and density, which is due to the mangosteen peel extract undergoing an extraction process that makes the fragments appear clearer and brighter in color.

The maceration procedure using 96% ethanol was used to extract the active ingredients from the mangosteen rind. The extraction results were then concentrated using a rotary evaporator at 40°C, yielding 11.37%. Mangosteen rind extract was identified using Thin Layer Chromatography (TLC). To obtain an Rf value of 0.39, stains on the plate were examined using a 254 nm UV lamp (Figure 1)

Table 1. Phytochemical test (Puspitasari et al., 2013)

Phytochemical test	Test performed
Alkaloids	Dragendorff test
Flavonoids	Acetone + boric acid + oxalic acid test
Saponins	HCL 2N test
Tannins and polyphenols	$FeCl_3$ test
Glycosides	$CH_3COOH + H_2SO_4$ test
Steroids and triterpenoids	Lieberman-Burchard test

Table 2. Lip balm formulation (Kusrini et al., 2020)

Material	F0	F1	F2	F3	F4
Mangosteen rind extract	-	0.025	0.05	0.075	0.1
Beeswax	2.5	2.5	2.5	2.5	2.5
Almond oil	5	5	5	5	5
VCO	5	5	5	5	5
Honey	2.5	2.5	2.5	2.5	2.5
Distilled water ad	1	1	1	1	1

The phytochemical screening results showed that mangosteen peel extract contains alkaloids, flavonoids, saponins, tannins, polyphenols, and glycosides (Table 3).

The mangosteen rind contains xanthone compounds with strong antioxidant, anticancer, anti-inflammatory, and antibacterial properties. Xanthone is a tricyclic compound that is effective in fighting free radicals, inhibiting the growth of cancer cells and bacteria, and has therapeutic potential for various health conditions. This study developed a lip balm formulation containing mangosteen rind extract, formulated on a natural basis without preservatives. Different amounts of mangosteen rind extract can be added to lip balm: 2.5% (F1), 5% (F2), 7.5% (F3), and 10% (F4). The properties are then put through a variety of physical tests, including ones for homogeneity, pH, melting point, spreadability, and stability, which was tested using the freeze-thaw method.

The organoleptic test results for the lip balm indicate that at F0, it is white and odorless because it does not contain mangosteen rind extract. Meanwhile, F1, F2, F3, and F4 produced cream-colored flowers with a characteristic odor. When applied to the lips, each formulation has the same soft, semi-solid shape. The lip balm formulation process, which aims to reduce particle size to ensure even dispersion, affects homogeneity.

The purpose of the melting point test is to determine the melting point of the lip balm preparation, which is the temperature at which all ingredients completely melt. Considering the findings of the melting point test on lip balm performed both before and after the freeze-and-thaw test, there was no significant change in the formulas F0, F1, F2, F3, and F4, which melted completely at 50°C within 5 minutes. The spreadability test was carried out to determine the extent of the lip balm preparation's ability to spread on the lips (Table 4).

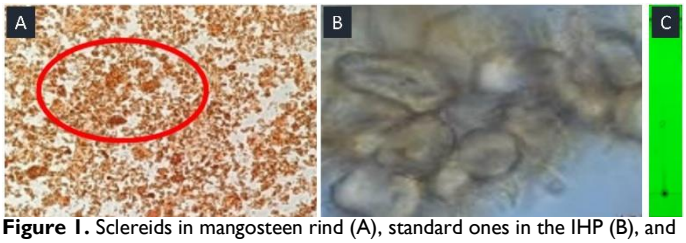

Figure 1. Sclereids in mangosteen rind (A), standard ones in the IHP (B), and the TLC profile of the extract (C)

Table 3. Phytochemical screening of mangosteen rind extract

Phytochemical test	Results
Alkaloids	+
Flavonoids	+
Saponins	+
Tannins	+
Polyphenols	+
Glycosides	+

The centrifugation test was carried out to observe phase separation or, indirectly, to assess it. Based on the centrifugation test results, there was no phase separation or change in the lip balm before or after the freeze-thaw test for the formulae F0, F1, F2, F3, and F4 (Table 5). This indicates that all lip balm formulations are stable. Based on the investigated methods' phase separation, honey helps maintain the lip balm's stability.

The freeze-thaw method is used to determine stability under the effect of temperature changes throughout the course of a specific storage period. All formulations were kept at 4°C for 24 hours, then at 40°C for 24 hours. This process' one cycle was identified. It took six cycles (12 days) to complete this research. The freeze and thaw stability test was done since the stability test at room temperature for 30 days was adequate to completely clarify the stability of lip balm formulated with mangosteen rind extract. Storage under extreme conditions can induce instability more quickly than at room temperature. In this study, it is evident that all formulas are stable during freeze-thaw testing, as determined by pH test parameters.

The pH test results for lip balm before freezing and thawing in Figure 2 show that the difference in pH was due to the higher extract concentration in each lip balm formula. Considering the possibility that this might affect the pH measurements, each formula still has a pH within the weak acid range, 4.5-7.0. The statistical test results show a p-value of 0.463 (<0.05), indicating that there is no effect of mangosteen rind extract variation on lip balm formulations.

The test continued by conducting a homogeneity test, used to determine the results of the variance equality test. The homogeneity test was performed using the Levene test. Based on the homogeneity test, the p-value of 0.822 (> 0.05) indicates that there is no significant difference between the formulas, so the data are homogeneous. The test indicates that the data are normally distributed and homogeneous, so the test proceeds with the One-way ANOVA. Based on the One-way ANOVA test, a significance value of 0.000 <0.05 was obtained, so it can be concluded that there is an influence between the pH of all formulas during freeze and thaw testing on days 1, 4, and 7

Table 4. Melting point test result

Formulation	Before freeze-thaw	After freeze-thaw
F0	Melting	Melting
F1	Melting	Melting
F2	Melting	Melting
F3	Melting	Melting
F4	Melting	Melting

Table 5. Centrifugation testing results

Formulation	Before freeze-thaw	After freeze-thaw
F0	Not Separate	Not Separate
F1	Not Separate	Not Separate
F2	Not Separate	Not Separate
F3	Not Separate	Not Separate
F4	Not Separate	Not Separate

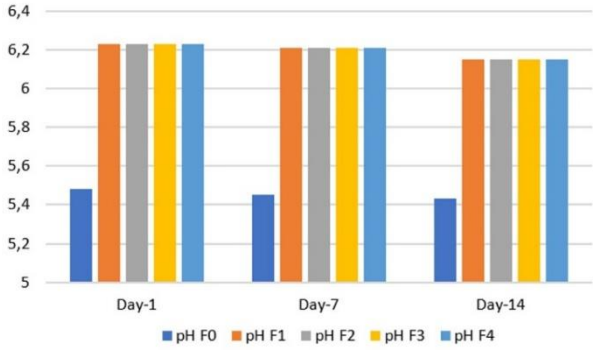

Figure 2. The pH of the lip balms

4. Conclusion

Different lip balm formulations showed acceptable physical properties and complied with standards based on organoleptic test, homogeneity test, pH test, melting point test, and power test spread. The physical properties of the material, a pH test, and a centrifugation test can be used to determine whether the lip balm preparation is stable during the freeze-thaw stability test.

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