Research Article

Lip color formulation using mangosteen rind extract (*Garcinia mangostana* L.)

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ABSTRACT

Objective: This study aimed to extract dyes from the skin of the mangosteen fruit (*Garcinia mangostana* Linn) and utilize them in the manufacture of cosmetics, especially lip color preparations. **Materials and Methods:** The research included material collection and plant determination, dye extraction and phytochemical screening, physical stability test of mangosteen peel extract, lipstick formulation with extracts of mangosteen peel various concentrations, physical stability test of lipstick preparations made, safety tests, hedonic tests for preparations made, and statistical data analysis. **Results:** The separation of mangosteen rind pigment produced a thick extract of dyes of 15.52% w/w. Phytochemical screening resulted that *G. mangostana* peel extract showed the absence of quinone and steroid compounds. Mangosteen rind extract was quite stable at temperature of 10–50°C, but not in the temperature range between 60°C and 80°C. Storage evaluation for 56 days, pH, and color (by thin-layer chromatography) was stable, observation of fungal growth, the exit of rough gains, or liquid during storage time did not occur. Lip color formula with various concentrations of mangosteen rind (10, 15, and 20%) was safe to be used. **Conclusion:** Based on the hedonic test, lip color formula containing various concentrations of mangosteen rind color (10, 15, and 20%) was quite preferred by consumers. And among the three, the most preferred formula was formula 3, which was lip color with the addition of a dye concentration of 20%.

KEY WORDS: Dye extraction, Formulation, *Garcinia mangostana*, Lip color, Mangosteen

INTRODUCTION

Since ancient Egypt, cosmetics have been available. At that time, the use of cosmetic preparations was always associated with events that were mystical, astrological, religious, or other events that were artistic and sacred. Cosmetics preparations are often used as skincare to slow down the aging process, as well as to increase attractiveness so that a person can look healthy and attractive.[1,2] One of the cosmetic preparations that are widely used to increase traction, change the shape of the lips, so it looks more beautiful is lip color. In general, lipstick dosage forms are stems, but there are also other dosage forms such as ointments, liquid, pencil, transparent, and others. Lip color is one of the most popular decorative cosmetics by women ranging from teenagers, adults, to the elderly. A lip coloring formula in the form of bars, ointments, or liquid generally consists of oil, wax, wax, and dyes.[3-5] According to other literature, what is meant by lip coloring is a lip cosmetic preparations in the form of a dispersion of dyes in a mixture of oil, fat, and wax.[6] In the manufacture of lip color, a dye is an important element and is very determining whether or not the product is in the market. Dyes used can be natural dyes, synthetic dyes, or inorganic dyes. In general, the use of natural dyes is more preferred than synthetic or inorganic dyes, because both of these dyes can cause undesirable side effects, such as itching, irritation, or red spots. The natural dyes can be obtained from plants, animals, or minerals.[7]

Indonesia has the number two collection of plants in the world after the Amazon (America), but only a few species have been used as medicine or cosmetics. The genus *Garcinia* is a tropical plant that grows in the forests of Southeast Asia. Unfortunately, this genus has not been well studied. Some literature says there are about 100–400 species of *Garcinia* in Southeast Asia, of which 30–35 are recorded as native Indonesian medicinal plants. From the number of *Garcinia* plants registered as native Indonesian

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medicinal plants, there are only a few which have been studied in terms of chemical content and benefits or bioactivity.[8,9] *Garcinia mangostana*, also known as mangosteen; manggu (Java, Sumatra), mangustang (Nusa Tenggara), and kirasu (Sulawesi), up to 25 m high, growing at an altitude of 1500 m above sea level, especially in the lowlands. Fruit skin also contains pectin, tannins, catechins, resins, triterpenoids, mangostin, and dyes which are often used as tanners and anti-corrosion paint makers.[10,11] The use of mangosteen peel (*G. mangostana*) as a medicine runs parallel to the bark, namely, as a chewing agent (adstringent), chronic dysentery, chronic urinary tract inflammation, intestinal bleeding, intestinal bleeding, ulcers, ulcers, swelling of the tonsils, tumors in the oral cavity and esophagus, excess saliva, and vaginal discharge, while the bark is used as an antidiarrheal drug, anti-inflammatory drug, allergic inhibitor in the treatment of asthma, and anticancer.[11-16] So far, the mangosteen color has not been formulated for lipstick. Gupta, however, repented skin firming anti-aging cosmetic mask compositions in 2004.[17] The use of mangosteen as this study reports on a lip dye formulation and its evaluation studies with mangosteen skin dye (*G. mangostana* Linn).

MATERIALS AND METHODS

Materials

The plant material used in this study was mangosteen (*G. mangostana* Linn.) rind obtained from Bandung Caringin Market, West Java. This plant material was determined in the herbarium section of the Plant Taxonomy Laboratory, Department of Biology, Faculty of Mathematics and Natural Sciences, Padjadjaran University.

Methods

Mangosteen fruit skin color separation

Fresh mangosteen skin was washed, after being separated from the fruit, which was white to clean. Then crushed using a blender with 95% ethanol solvent, and macerated for 3 × 24 h. Then filtered and ethanol extract obtained was evaporated using a rotary evaporator at 40°C until a thick extract was formed.

Phytochemical screening

Phytochemical screening of mangosteen skin extract was carried out using standard method[18,19] to determine its alkaloid, polyphenols, tannins, flavonoids, monoterpenoid and sesquiterpenoids, steroid, quinone, and saponin compounds.

Test of physical stability of mangosteen fruit skin color

Examination of the physical stability of the mangosteen rind pigment results of separation carried out on temperature by heating the extract of the dyes from the separation to a temperature of 80°C in the oven and cooling it to a temperature of 10°C in the refrigerator. Color changes that occur (darker colors) throughout 5, 15, and 60 min were observed. If a dye that was heated and cooled at a certain temperature and time frame changes color to darker, it meant that the dye is unstable at that temperature. Dyestuff extract was checked for pH stability using a universal pH indicator during storage for days 1, 7, and 14, and every week for 56 days.

Formulation of making lip color with mangosteen skin color substances of various concentrations

Based on the orientation that has been carried out, the basic formula chosen in the manufacture of lip color is the basic formula with the following composition.[20]

- R/Carnauba wax 10%
- White wax 15%
- Lanolin 5%
- Alcohol 5%
- Castor oil 65%
- Pigment 10%.

With this basic, lip color with 10%, 15%, and 20% mangosteen peel coloring agent was formulated, as shown in Table 1.

Ways of making

Carnauba wax, white wax, lanolin, cetyl alcohol, and some castor oil were melted on a water bath at 70–80°C. Then, the dye and titanium dioxide were crushed together with the remaining castor oil. After that, in a hot mortar mix, the melted substance added to the dye mixture, stirring until it was homogeneous and cold. After it cooled down, put it in a container, as a comparison using a formula without dyes.

Table 1: Lip color formula with variations in the concentration of mangosteen rind

Formula	Composition (%)						
	Carnauba wax	White wax	Lanolin	Cetyl alcohol	Castor oil	Titan dioxide	Pigment
F0	9.09	13.64	4.54	4.54	66.37	1.82	-
F1	9.09	13.64	4.54	4.54	57.28	1.82	9.09
F2	9.09	13.64	4.54	4.54	52.73	1.82	13.64
F3	9.09	13.64	4.54	4.54	48.19	1.82	18.18

F0: Lip color formula without dye, F1: Lip color formula with 10% mangosteen rind color, F2: Lip dye formula with 15% mangosteen rind color, F3: Lip dye formula with 20% mangosteen rind color

The Formula Evaluation

Physical stability test: The test of the physical stability of lip color during storage includes

Observation of changes in shape, color, and odor

Observation of changes in the shape, color, and odor of the preparations was made on three preparations of each formula during storage day 1, 7, and 14, and then every week until the 56th day. The same observation was also carried out on comparative lip color preparations obtained from the market.

Observation of the growth of fungi, and the release of crystals or liquid

Observation of fungal growth, crystal, or liquid discharge was carried out visually on three preparations from each formula during storage of days 1, 7, and 14, and then every week until the 56th day. The same observation was also carried out on comparative lip color obtained from the market.

Homogeneity check

Homogeneity checks were carried out visually by pressing the preparation using the thumb and forefinger. The homogeneity of the preparation as indicated by the absence of lumps such as sand. Tests were carried out on three preparations of each formula during storage days 1, 7, and 14, and subsequently every week until the 56th day. The same test was also carried out on comparable lip colorings.

Test color stability against temperature

This test was done by heating the preparation to a temperature of 80°C in the oven and cooling it to a temperature of 10°C in the refrigerator for a span of 5, 15, and 60 min. Color changes that occur during this period were observed. Tests were also carried out on comparative lip color preparations.

Test the pH stability of the preparation

Each lip color formula was checked for pH stability by melting the preparation in a vaporizer cup on a water bath while stirring, then allowed to wait for a bit to cool, and measured its pH using a universal pH. Tests were also carried out on comparative lip colors. This pH measurement was carried out during storage days 1, 7, and 14, and then every week until the 56th day.

Thin-layer chromatography (TLC)

TLC was carried out on the extract of mangosteen rind extract and the dyes in the preparation made. TLC was done to find out whether the dyes that had been formulated in lip dyes could still be identified as the same dyes as the extracted dyes. The eluent was a mixture of butanol-acetic acid-water (4:1:5) developer solution. TLC plate with Silica Gel GF 254 absorbent measuring 4 cm × 7 cm was applied. Its Rf value was calculated by measuring the spacing creepage distance, compared to the creepage distance of the developer solution.

Topical test

The topical test was done by applying lipstick preparations made on the skin of the back of the hand. Good preparations were lip color preparations that were easily applied and leave a color that sticks well to the skin of the back of the hand. While lip color formulations that were not well marked were not easily applied and do not leave a color that sticks to the lips. This test was carried out on three preparations from each formula and against the comparative lip color during the storage period of days 1, 7, and 14, and then every week until the 56th day.

Irritation test

The irritation test for lip color preparations was done by applying the preparations to the skin of the backs of the volunteers' hands, to find out if the lipstick preparations made could irritate the skin. If the irritation arose shortly after skin adhering or touching, it was called primary irritation, and the secondary irritation reaction occurred only a few hours after touching or sticking to the skin.[21]

Hedonic test

To find out the level of panelists' preference for the colors of the preparations made, a visual hedonic test was carried out on 30 panelists by filling out a questionnaire.[22] Each panelist was asked to apply the lip color to the skin of the back of the hand, then assess each preparation according to the preference scale found on the questionnaire. From the results of this favorite test will get data about the level of preference for the lip color formula made. The results obtained in the form of an ordinal scale must be processed first with the successive interval method so that the interval scale is obtained, then processed with ANOVA, and further tests with Newman-Keuls.[23,24]

RESULTS

Mangosteen Fruit Skin Color Separation

The results of the separation of dyes from the mangosteen rind obtained using ethanol 95% as the extracting liquid, dark red dark and shaped thick extract of fresh mangosteen rind as much as 15.52% W/W.

DISCUSSION

Phytochemical Screening

Phytochemical screening results of mangosteen rind extract are shown in Table 2. It is shown that mangosteen rind extract contains tannin compounds that were included in the polyphenol group. This was

following the literature which said mangosteen rind contains alkaloids, flavonoids, quinones, saponins, polyphenols, triterpenoids, steroids, and tannins that make mangosteen rind reddish-purple.[21] Qualitative analysis revealed that the presence of phenols, flavonoids, and triterpenoids in the mangosteen rind extracts was also reported by others.[25-28]

Physical Stability Test Results of Mangosteen Fruit Skin Color

Stability to temperature changes

The results of the test of the stability of dyes to temperature are shown in Table 3. Table 3 revealed that the extract of mangosteen rind was quite stable at temperatures of 10–50°C, but in the temperature range between 60°C and 80°C was unstable because in that temperature range showed a change in color from dark red to red-black. Since in the manufacture of lipstick the temperature used to melt the carrier material (base) was around 70°C–80°C, while the dyes from the mangosteen rind were not resistant to heating, the lipstick preparation made was in a liquid form.

PH stability

The results of the pH stability of mangosteen peel extract are shown in Table 4. It was shown that the extract of mangosteen peel was quite stable for 56 days of storage, which was between 4 and 5.5. This was still under the pH of the required lip color, which was around 4.00–7.00, according to the pH of the lip.[29] This means that judging from the pH of the mangosteen peel dye was quite safe to use.

Table 2: Phytochemical screening results of mangosteen skin extract

Secondary metabolite	Result
Alkaloids	+
Flavonoids	+
Quinone	–
Saponins	+
Polyphenols	+
Tannin	+
Triterpenoids	–
Steroids	–

(+): Detected, (-): Not detected

Table 3: Physical stability of mangosteen fruit skin color changes to temperature changes

Temperature (C°)	Discoloration after (minutes)		
	5	15	60
10	dr	dr	dr
20	dr	dr	dr
30	dr	dr	dr
40	dr	dr	dr
50	dr	dr	dr
60	rb	rb	rb
70	rb	rb	rb
80	rb	rb	rb

dr: Dark red, rb: Red black

Lip coloring with mangosteen skin color substances of various concentrations

The results of observations of the shape, color, and odor of lip dyes made with variations in the concentration of the mangosteen rind dye are shown in Table 5. From Table 5, it could be seen that lip color made with variations in the concentration of mangosteen peel 10–20% had a good shape and had a characteristic odor of fat because it contained a base consisting of a mixture of fat, oil, and wax. The preparation had a color that varies from pink to dark red depending on the concentration of the dye added. Fp as a comparison formula from the market had the same shape, color, and odor as F3 (dye concentration of 20%).

Physical stability test results for lip coloring

In the temperature range of 10–50°C, there were no color changes of all lip color formulas. Color changes occur at a temperature of 60°C and so on. Unlike comparative lip color that remained stable at temperatures up to 80°C.

Lip stability pH test

The results of the pH stability test for lip color during storage are shown in Table 6. It showed that the pH of each formula during storage experiences a noticeable change. Based on the statistical calculation of pH changes concerning storage time, it found that H0 was rejected because F table ($\alpha = 0.05$) was smaller than F count, which meant there was a significant pH difference between lip color formulas for 56 days of storage time, i.e., between pH 4 and 5. This was due to the influence of various concentrations of dyes added, which were 10, 20, and 20%. However, despite the change in pH, the pH range still met the pH requirements for lip color, which was between pH 4 and 7.[29]

Lipstick viscosity measurement

The results of measurements of changes in viscosity (centipoise) of lip color with various concentrations of dyes during storage, it was found that the viscosity of lip color with various concentrations of mangosteen rind color decreased significantly. From the statistical

Table 4: pH stability test for mangosteen rind extract during storage time

Compound	The pH of the dye extract on day today								
	1	7	14	21	28	35	42	49	56
Dyestuff of mangosteen rind	5.5	5.0	5.0	4.5	4.5	4.5	4.5	4.0	4.0

calculations using the complete random block method and the ANOVA, it revealed that H0 was rejected because F table (α = 0.05) was smaller than F count, which meant that there was a real difference in viscosity between lip color formulas for 56 storage time day. It also noticed that in general, the viscosity of preparations, including formulas without dyes had decreased during storage. This decrease in viscosity was partly since there was no formula stabilizing agent in the base for lip colors such as antioxidants, preservatives, and others were not added or because of the interaction between the components contained in the formula, and because of the treatment of the ready-made formula. The addition of mangosteen peel dye with varying concentrations turned out to also affect the viscosity of lip color preparations, as evidenced by the greater concentration of the dyes added, the greater the viscosity produced. This was because most of the compounds contained in mangosteen peel coloring were tannins that were known to react with the protein contained in the formula so that the preparation became more viscous and viscosity would be higher.

Table 5: Observation results of a shape, color, and odor of lip color with variation concentration of mangosteen rind

Formula	Shape	Color	Odor
F0	good	white	Typical fat
F1	good	pink	Typical fat
F2	good	pink	Typical fat
F3	good	red	Typical fat
Fp	good	red	Typical fat

F0: Formula without dye, F1: Lip dye formula with 10% mangosteen rind color, F2: Lip dye formula with 15% mangosteen rind color, F3: Lip dye formula with 20% mangosteen rind color, Fp: Comparative lip coloring formula

Table 6: pH stability test results for lip color during storage

Formula	Lip color pH on the day								
	1	7	14	21	28	35	42	49	56
F0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
F1	5.0	4.5	5.0	5.0	5.0	4.5	4.0	5.0	4.0
F2	5.0	5.0	4.5	4.5	4.5	4.5	5.0	4.5	4.5
F3	0.5	5.0	5.0	4.5	4.5	4.0	4.5	5.0	4.0
Fp	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5

F0: Formula without dye, F1: Lip dye formula with 10% mangosteen rind color, F2: Lip dye formula with 15% mangosteen rind color, F3: Lip dye formula with 20% mangosteen rind color, Fp: Comparative lip coloring formula

TLC results

The value of Rf and the blotch color of the mangosteen rind extract extraction agent and the dyes resulting from the separation of the lip color are shown in Table 7. Table 7 showed that the dyes from the mangosteen rind and the dyes from the separation of lip color formulas 1, 2, and 3 on days 1 and 28 have the same Rf value of 0.73 indicating that the dyes that had been formulated into lip color F1, F2, and F3 had not changed, and could still be identified as the same dyes as the dyes derived from mangosteen rind. On day 56 Rf of the dyes in formulas 1, 2, and 3 experienced a slight change. This could be caused by a reaction between the components of lip color during storage, or the decomposition of the dye and other constituent components of lip color due to environmental influences.

Lip color test

It was found that when F1 was applied to the skin of the back of the hand gave less color, whereas F2 and F3 provided a pretty good color to the skin of the back of the hand, almost the same as the result of applying comparative lipstick. This was possibly due to the concentration of dyes added to F1 was very small so that when applied less left the color on the skin. It could be said that the higher the concentration of the dye added, the better the resultant smearing on the skin of the back of the hand.

Irritation test results

It was found that after irritation testing of each lip color formula for 20 volunteers, all of the formulas did not cause an irritation reaction. This was evidenced by the absence of symptoms of irritation caused by the onset of red skin, itching, bumps, or swelling. Hence, it could be said that the lip color made with variations in the concentration of the mangosteen rind color, was quite safe to use.

Hedonic test

It was found that the results of the questionnaire given to volunteers, had an ordinal scale, which was a scale to differentiate and state the ranking order, where arithmetic law did not apply. To see whether there was a difference between panelist preferences for the three lip color formulas, the data ought first

Table 7: TLC of mangosteen rind extract and color dyes separation from lip dyes during storage

Separating dyes from	Observation with			UV 254 nm		
	Rf mean on the day today			Spotting color on the day today		
	1	28	56	1	28	56
Mangosteen rind	0.73	0.73	0.76	brown	brown	Brown
Formula 1	0.73	0.73	0.76	brown	brown	brown
Formula 2	0.73	0.73	0.76	brown	brown	brown
Formula 3	0.73	0.73	0.76	brown	brown	brown

TLC: Thin-layer chromatography

be changed into an interval scale, which was a scale that showed that the greater the number, the higher the level of preference, using the successive interval method, it was known that the F count (13, 18) > F table (3, 11). This meant that the null hypothesis with the assumption that there was no significant difference between the level of panelists' preference for the various concentrations of mangosteen peel dye. From the results of the calculation of preference test, it found that lip color with the addition of mangosteen peel dye concentration of 10, 15, and 20% was quite favored by panelists. Based on the Newman-keuls test, it could be seen that the lip color F2 was significantly different from F1 as well as F3, while F1 lip color was not significantly different from F3. By looking at the average value of interval scale data obtained, it was known that the F3 lip color was more favored than the F1 and F2 lip color. This might be due to the lip color formula with 20% dyes producing a better, more attractive color and smearing result than panelists compared to other lip coloring formulas. However, the more dyes added the less it tastes.

It must be admitted that some lipstick formulations from herbal dyes that are different from our study have also been carried out by researchers. Color pigments of *Bixa orellana* (Bixaceae) seeds,[30] *Monascus purpureus* extract,[31] *Theobroma cacao* seeds,[32] *Daucus carota*,[33] water fraction of mangosteen,[34] and *Beta vulgaris* extract[35] among others dyes have been reported.

CONCLUSION

From the research that has been done, it concluded the dye from the mangosteen rind (*G. mangostana* Linn.) in the form of thick extract which was dark red, had a pH between 4 and 5.5 and was stable to temperatures of 10–50°C. The result of TLC showed that the dyes from the mangosteen peel could still be identified as the same dyes as the dyes produced by the lipstick made during storage. Lip color formula with various concentrations of mangosteen rind (10, 15, and 20%) was quite safe to use because it did not irritate its use. Among the three, the most preferred formula was formula 3, which was lip color with the addition of a dye concentration of 20%.

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