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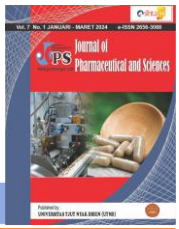

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Formulation and Evaluation of Lipstick Preparations with Hibiscus Flower Extract (*Hibiscus rosa-sinensis* L.) as a Natural Colourant

Formulasi dan Evaluasi Sediaan Lipstik Ekstrak Bunga Kembang Sepatu (*Hibiscus rosa-sinensis* L.) Sebagai Pewarna Alami

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Abstract
Synthetic dyes in decorative cosmetics remain a safety concern when prohibited colourants are used in products that are applied repeatedly to the lips. Hibiscus flower (Hibiscus rosa-sinensis L.) contains anthocyanins that can provide red to purple hues. This preliminary laboratory study prepared solid lipsticks containing 0%, 3%, 6%, 9%, and 12% hibiscus flower extract and evaluated organoleptic characteristics, approximate pH, short-term room-temperature stability, acute irritation response, and descriptive consumer preference. Dried flower powder underwent two-stage maceration with citric-acidified 96% ethanol, and the concentrated extract was incorporated into a wax-lipid base. The sticks weighed 3- 3.5 g and were easy to apply. The extract-free formula was colourless, while extract-containing formulas produced bright pink to dark red shades. No visible changes in shape, colour, or odour were observed during 30 days of room-temperature storage. All formulas showed an approximate pH of 5 by indicator paper and caused no redness, itching, or oedema in 10 volunteers during a 30-minute preliminary acute irritation screening. The 12% formula had the highest aggregate hedonic score, but statistical superiority could not be determined because panellist-level raw data were unavailable. These findings provide preliminary formulation evidence. Interpretation remains limited by non-identical base compositions, visual colour assessment, non-instrumental pH measurement, and the absence of anthocyanin quantification and accelerated stability testing.

Keywords*: *Hibiscus rosa-sinensis, anthocyanins, lipstick, natural colourant, physical stability

Abstrak
Pewarna sintetis pada kosmetik dekoratif masih menjadi isu keamanan apabila bahan pewarna terlarang digunakan pada produk yang diaplikasikan berulang kali pada bibir. Bunga kembang sepatu (Hibiscus rosa-sinensis L.) mengandung antosianin yang dapat menghasilkan warna merah hingga ungu. Penelitian laboratorium pendahuluan ini membuat lipstik batang yang mengandung ekstrak bunga kembang sepatu sebesar 0%, 3%, 6%, 9%, dan 12%, kemudian mengevaluasi karakteristik organoleptik, pH perkiraan, stabilitas jangka pendek pada suhu kamar, respons iritasi akut, serta tingkat kesukaan secara deskriptif. Serbuk bunga dimaserasi dalam dua tahap menggunakan etanol 96% yang diasamkan dengan asam sitrat, lalu ekstrak kental dimasukkan ke dalam basis lilin-lemak. Lipstik yang dihasilkan berbobot 3-3,5 g dan mudah dioleskan. Formula tanpa ekstrak tidak berwarna, sedangkan formula yang mengandung ekstrak menghasilkan warna merah muda terang hingga merah gelap. Tidak ditemukan perubahan visual pada bentuk, warna, atau bau selama penyimpanan selama 30 hari pada suhu kamar. Seluruh formula menunjukkan pH perkiraan 5 berdasarkan kertas indikator dan tidak menimbulkan kemerahan, gatal, atau edema pada 10 sukarelawan selama skrining iritasi akut awal selama 30 menit. Formula 12% memperoleh skor hedonik agregat tertinggi, tetapi keunggulan statistik tidak dapat ditentukan karena data mentah dari setiap panelis tidak tersedia. Hasil ini memberikan bukti atas formulasi awal. Interpretasi hasil dibatasi oleh komposisi basis yang tidak identik, penilaian warna secara visual, pengukuran pH noninstrumental, serta belum adanya pengukuran kadar antosianin dan uji stabilitas yang dipercepat.

Kata Kunci: Hibiscus rosa-sinensis, antosianin, lipstik, pewarna alami, stabilitas fisik.

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Introduction

Cosmetics are preparations intended for external body parts, including the epidermis, hair, nails, lips, external genital organs, teeth, and oral mucosa, mainly to clean, perfume, change appearance, improve body odour, protect, or maintain the body in good condition [1]. Lipstick is one of the most widely used decorative cosmetics because it adds colour, enhances appearance, and complements the aesthetics of facial makeup. Since lipstick is applied directly to the lips, colourant selection is important for both product performance and consumer safety.

Colourant safety remains a major issue in decorative cosmetics. Indonesian cosmetic regulations restrict the use of prohibited ingredients, including hazardous synthetic colourants, and the national regulatory authority continues to report the circulation of cosmetics containing banned or dangerous substances [2,3]. This situation underscores the need for safer colourant sources, particularly plant-derived pigments. However, replacing synthetic dyes with natural dyes must not rely only on the claim that plant materials are natural. A natural pigment must also demonstrate acceptable organoleptic quality, stability, pH compatibility, skin tolerance, and user acceptance [4].

Anthocyanins are water-soluble flavonoid pigments responsible for red, purple, and blue colours in many plant tissues. Their colour expression depends on chemical structure, pH, temperature, oxygen, light, metal ions, and interaction with other matrix components [5,6]. These characteristics make anthocyanins attractive for the development of natural colourants, but they also pose formulation challenges. In anhydrous or semi-solid systems such as lipstick, anthocyanin-rich extracts must be incorporated into a compatible wax-lipid base while preserving colour intensity and physical integrity. Acidification during extraction can help maintain the red flavylium form of anthocyanins, and recent extraction reviews emphasise solvent composition, pH, and process conditions as critical variables [7–9].

Hibiscus flower (Hibiscus rosa-sinensis L.) is widely available in tropical regions, including Indonesia. It has long been reported to contain anthocyanins and other flavonoids [10]. A recent study of 16 Hibiscus rosa-sinensis cultivars showed that flower colour is associated with flavonoid composition and antioxidant capacity, supporting the relevance of this species as a pigment source [11]. Hibiscus rosa-sinensis red flower extract also contains phenolic constituents with reported biological activity, including antibacterial activity against antibiotic-resistant Helicobacter pylori strains [12]. These findings support its selection as a candidate colourant for topical cosmetic formulation.

Recent studies have explored the use of natural dyes in lip products. Hibiscus extract has been tested in herbal lipstick and lip balm formulations, but previous results indicate that colour intensity, homogeneity, and dosage require optimisation [13,14]. Hibiscus extract has also been formulated into lip cream as a natural colourant [15]. Other Indonesian and international studies have used beetroot, roselle, secang wood, harimonting, senggani flower, kepok banana flower peel, jati leaf, and henna leaf as natural colourant sources for lipstick, lip cream, or lip balm [16-23]. These studies show growing interest in plant-based lip cosmetics, but clearer comparative evidence is still needed regarding the use of hibiscus flower extract in solid lipstick sticks.

This preliminary study aimed to formulate and evaluate solid lipstick preparations containing hibiscus flower extract as a natural colourant. The objectives were to describe the visual colour produced by different extract concentrations; assess organoleptic characteristics, short-term room-temperature stability, approximate pH, immediate irritation response, and descriptive consumer preference; and identify the formula with the highest aggregate performance under the tested conditions. The study was not designed to

establish statistical superiority, quantitative correlations between anthocyanin colour and quantitative measures, long-term safety, or product shelf life.

Methods

Materials and apparatus

The apparatus consisted of standard laboratory glassware, a blender, an evaporating dish, lipstick roll-up moulds, an analytical balance, an oven, a water bath, and pH indicator paper. The materials included fresh hibiscus flowers (Hibiscus rosa-sinensis L.), distilled water, 96% ethanol, citric acid, cera alba, vaseline album, cetyl alcohol, anhydrous lanolin, carnauba wax, butylated hydroxytoluene (BHT), Tween 80, rose oil, propylene glycol, and nipagin. A colourimeter, a spectrophotometer, a calibrated pH meter, and a controlled-temperature stability chamber were not used in the original experiment.

Sample collection and processing

Fresh hibiscus flowers were obtained purposively from Simpang Limun Market, Medan, Indonesia. A total of 2 kg of fresh flowers were wet-sorted, washed under running water, drained, dried in a drying cabinet, dry-sorted, ground into a powder, and stored in a tightly closed container until extraction. The plant material was assessed macroscopically, and its observed characteristics were consistent with Hibiscus rosa-sinensis L. as described in the literature [10].

Extraction procedure

The extract was prepared by two-stage maceration. One kilogram of dried hibiscus flower powder was macerated in 1.5 L of 96% ethanol containing 30 g of citric acid for 5 days in a closed, light-protected container, with occasional stirring. The residue was then remacerated with 0.5 L of 96% ethanol containing 10 g of citric acid for 2 days. Thus, the cumulative powder-to-solvent ratio was 1:2 (w/v), and the citric acid concentration was 20 g/L (2% w/v) at each maceration stage. The filtrates were combined and evaporated on a water bath until a thick extract was obtained. These conditions adhered to the original laboratory protocol and were not optimised. The final extract pH and total anthocyanin content were not measured. Acidification was intended to favour anthocyanin colour retention, but future work should optimise the solvent ratio, acid concentration, extraction yield, and extract pH [5,8,9].

Lipstick formulation and preparation

Table 1. Formula of lipstick preparations containing hibiscus flower extract

Composition (g)	F0	F1	F2	F3	F4
Cera alba	17.91	16.41	15.87	15.32	14.78
Anhydrous lanolin	3.77	3.64	3.52	3.40	3.28
Vaseline album	16.02	16.41	15.87	15.32	14.78
Cetyl alcohol	2.83	2.73	2.64	2.55	2.46
Oleum ricini	3.71	3.64	3.52	3.40	3.28
Carnauba wax	2.36	2.28	2.20	2.12	2.05
Hibiscus flower extract	0.00	1.50	3.00	4.50	6.00
Propylene glycol	2.50	2.50	2.50	2.50	2.50
Tween 80	0.50	0.50	0.50	0.50	0.50
Rose oil	0.25	0.25	0.25	0.25	0.25
BHT	0.05	0.05	0.05	0.05	0.05
Nipagin	0.05	0.05	0.05	0.05	0.05

Note. F0 = 0% extract; F1 = 3%; F2 = 6%; F3 = 9%; F4 = 12%. Several wax-lipid base components were adjusted to maintain an approximately 50 g batch; therefore, differences among formulas cannot be attributed exclusively to extract concentration.

Five formulas were prepared: F0 as the extract-free comparator and F1, F2, F3, and F4 containing hibiscus flower extract at 3%, 6%, 9%, and 12%, respectively. Nipagin was dissolved in propylene glycol, and the hibiscus extract was added and mixed until homogeneous to form mass A. BHT was dissolved in oleum ricini to form mass B. Masses A and B were combined. The wax-lipid phase containing cera alba, carnauba wax, cetyl alcohol, anhydrous lanolin, and vaseline album was melted on a water bath, after which the extract phase was incorporated. Rose oil and Tween 80 were added while stirring during cooling. The molten mass

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was poured into roll-up moulds, cooled, removed, and packed. To maintain an approximately 50 g batch, the quantities of several base components varied among formulas. Therefore, the formulas were not compositionally matched except for extract concentration, and F0 should not be interpreted as a strictly matched control.

Quality evaluation and data analysis

The evaluation included organoleptic observation, short-term room-temperature stability, approximate pH screening, preliminary acute irritation screening, and descriptive hedonic testing. Organoleptic evaluation covered shape, colour, odour, and ease of application. Short-term stability was assessed visually at room temperature on days 0 and 30 by observing shape, colour, and odour. The original protocol did not include instrumental L*, a*, and b* colour measurements, total colour difference (ΔE), accelerated stability testing, or temperature cycling. Approximate pH was screened by inserting pH indicator paper into the molten lipstick mass; no calibrated pH meter or standardised aqueous dispersion was used. The irritation screening involved 10 healthy adult volunteers. Each preparation was applied to the inner lower arm, and redness, itching, and oedema were observed for 30 minutes, with observations at 5-minute intervals. This procedure was intended only to detect immediate visible reactions and was not a repeated-insult patch test. The hedonic test involved 10 panellists who applied each formula to the back of the hand and scored appearance, odour, spreadability, colour, and texture on a 3-point scale: 3 = very like, 2 = like, and 1 = dislike [24]. Because panellist-level raw scores were unavailable, only aggregate total and mean scores were analysed. Kruskal-Wallis and post hoc tests could not be performed validly on the available aggregate data. Adult volunteers received information about the procedure and provided consent before participation. No invasive procedure was performed.

Results and Discussion

Five lipstick formulas were prepared. The results should be interpreted as preliminary descriptive findings because several requested instrumental and inferential analyses were not part of the original dataset. The results are presented in four tables and one figure in accordance with the journal format.

Organoleptic characteristics of lipstick preparations

All formulas produced solid lipstick sticks that were easy to apply and weighed 3- 3.5 g. The extract-free comparator (F0) was colourless. F1 produced a bright pink shade, F2 and F3 produced pink shades, and F4 produced a dark red shade. A darker shade was visually observed as the amount of extract increased. However, total anthocyanin content was not quantified, and the base composition varied among formulas. The pattern must therefore be interpreted as a descriptive formulation trend rather than a quantified concentration-colour relationship [11].

Visual differences among formulas are relevant to immediate consumer perception. F4 showed the darkest visible pigmentation and had the highest aggregate colour score. Similar formulation studies have reported greater visual pigmentation at higher levels of natural colourants [17,20]. Nevertheless, the non-identical wax-lipid composition may also have influenced texture, spreadability, pigment dispersion, and appearance. The observed differences cannot be attributed solely to hibiscus extract.

Table 2. Organoleptic characteristics and approximate pH of lipstick preparations

Formula	Extract (%)	Form	Color	Odor	Application	pH*
F0	0	Solid stick	Colorless	Rose oil odour	Easy to apply	5
F1	3	Solid stick	Bright pink	Rose oil odour	Easy to apply	5
F2	6	Solid stick	Pink	Rose oil odour	Easy to apply	5
F3	9	Solid stick	Pink	Rose oil odour	Easy to apply	5
F4	12	Solid stick	Dark red	Rose oil odour	Easy to apply	5

A central formulation challenge is the polarity mismatch between hydrophilic anthocyanins and the predominantly lipophilic wax-oil base. Propylene glycol and Tween 80 were used to wet and disperse the polar extract within the molten base. However, the study did not quantify dispersion efficiency, particle or droplet distribution, phase separation, or colour bleeding during storage. BHT may reduce the oxidation of lipid components, but its presence does not confer protection of anthocyanins from degradation caused by

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light, oxygen, temperature, and unfavourable pH. These degradation pathways are well documented for anthocyanins [5-9]. Future studies should combine microstructural assessment with instrumental colour measurement to evaluate pigment distribution and migration.

Figure 1. Lipstick preparations containing hibiscus flower extract at 0%, 3%, 6%, 9%, and 12%.

Plant material identification

The macroscopic characteristics of the collected floral material were consistent with those of Hibiscus rosa-sinensis L., as described in the literature [10].

Physical stability, pH compatibility, and irritation response

After 30 days of room-temperature storage, no visible changes in shape, colour, or odour were observed. This result demonstrates only short-term organoleptic consistency under the tested conditions. It does not establish quantitative colour stability or predict shelf life. Cera alba and carnauba wax likely contributed to stick rigidity, while vaseline album, lanolin, and oleum ricini supported softness and application. Because the proportions of these base components varied among formulas, the observed physical characteristics cannot be attributed exclusively to the extract.

All formulas produced an approximate pH reading of 5 on indicator paper. This value should be treated as a screening estimate because indicator paper has limited resolution and a lipid-rich solid matrix is not suitable for precise direct measurement. Topical formulation pH remains relevant to skin tolerance [25], while anthocyanin colour is strongly influenced by pH [5,6]. A future validated procedure should disperse 1 g of lipstick in 10 mL of distilled water heated to approximately 60°C, cool the dispersion to room temperature, and measure its pH with a calibrated pH meter.

The 30-minute preliminary acute irritation screening showed no immediate visible redness, itching, or oedema in the 10 volunteers. This finding supports only the absence of an acute visible reaction under the specific test conditions. It does not establish safety after repeated application to the lips or rule out delayed irritation and sensitisation. Future studies should use a larger panel, documented ethical approval, longer observation periods (e.g., 24, 48, and 72 hours), and an appropriate repeated patch-testing protocol in accordance with applicable cosmetic safety guidance.

Table 3. Short-term visual observations and preliminary acute irritation response

Formula	Colour on day 0	Visual colour observation, day 30	Visual shape and odour observation, day 30	Redness	Itching	Edema
F0	Colorless	No visible change	No visible change	-	-	-
F1	Bright pink	No visible change	No visible change	-	-	-
F2	Pink	No visible change	No visible change	-	-	-
F3	Pink	No visible change	No visible change	-	-	-
F4	Dark red	No visible change	No visible change	-	-	-

Note. "No visible change" indicates unaided visual observation only; no instrumental L, a*, b*, or ΔE measurement was performed. A dash (-) indicates no immediate visible irritation reaction during the 30-minute preliminary acute screening.*

The 30-day room-temperature observation was not an accelerated stability study. Samples were not stored at 40 ± 2°C and 75 ± 5% relative humidity, and no 4°C/40°C cycling test was conducted. Consequently, the present data cannot predict shelf life or stability during distribution. Future work should assess

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organoleptic properties, pH, hardness, spreadability, bleeding, and instrumental colour values at scheduled intervals for at least 12 weeks under accelerated and cycling conditions.

Descriptive hedonic evaluation

The aggregate hedonic results showed that F4 had the highest total score and mean score. F4 reached a total score of 132 and a mean of 2.64 across five attributes, followed by F1 (111; 2.22), F0 (108; 2.16), F3 (104; 2.08), and F2 (88; 1.76). Descriptively, the high F4 score was associated with appearance, spreadability, colour, and texture. These values provide a ranking of aggregate scores only.

Natural lip-product studies often report that consumer preference is influenced by the combined effects of colour intensity and spreadability [13,16,18,19]. In the present dataset, F4 had the highest descriptive aggregate score. However, aggregate totals do not preserve the within-panellist ordinal observations required for a Kruskal-Wallis analysis. Therefore, the study cannot conclude that F4 was significantly preferred or statistically superior to the other formulas. Future studies should retain panellist-level data, randomise and blind sample presentation, use a larger panel, apply the Kruskal-Wallis test for each attribute, and conduct corrected post hoc pairwise tests when the omnibus result is significant.

Table 4. Aggregate descriptive hedonic scores of lipstick preparations

Formula	Appearance	Odor	Spreadability	Color	Texture	Total score	Mean
F0	23	18	23	19	25	108	2.16
F1	24	17	25	21	24	111	2.22
F2	15	17	23	17	16	88	1.76
F3	23	17	23	20	21	104	2.08
F4	29	20	28	29	26	132	2.64

Note. Scores were obtained from 10 panellists using a 3-point scale: 3 = very likely, 2 = likely, and 1 = unlikely. Only aggregate scores were available. Inferential statistical testing and pairwise significance comparisons were not performed.

Study limitations and recommendations

This study has several important limitations. First, total anthocyanin content was not measured in the concentrated extract or finished lipsticks, so a quantitative relationship between anthocyanin level and colour intensity cannot be established. Second, colour stability was assessed visually, without colourimetric L*, a*, b*, or ΔE data. Third, pH was estimated with indicator paper, and the pH of the concentrated extract was not measured. Fourth, only aggregate hedonic scores were available, preventing the use of valid Kruskal-Wallis and post hoc analyses. Fifth, the wax-lipid base composition was not held constant across formulas, so differences in physical and sensory characteristics may partly reflect changes in base proportions. Sixth, stability observation was limited to 30 days at room temperature without accelerated or cycling tests. Seventh, the irritation assessment involved only 10 volunteers and a 30-minute acute observation period, which cannot determine cumulative irritation or sensitisation. Eighth, the sample-to-solvent ratio and 2% citric acid concentration used during extraction were not optimised. Future research should use compositionally matched formulas or an inert balancing material, quantify anthocyanins by the pH differential or another validated spectrophotometric method, measure colour instrumentally, determine extract and product pH with a calibrated meter, retain panellist-level data for inferential analysis, conduct accelerated and cycling stability tests, and perform ethically approved repeated-use safety assessments.

Conclusions

Under the preliminary conditions of this study, hibiscus flower extract was incorporated into solid lipstick preparations, yielding shades ranging from bright pink to dark red. No visible changes in shape, colour, or odour were detected during 30 days of room-temperature storage. All formulas showed an approximate pH of 5 on indicator paper, and no immediate visible redness, itching, or oedema occurred during the 30-minute acute irritation screening in 10 volunteers. The 12% formula had the highest descriptive aggregate hedonic score, but statistical superiority was not established. Because the base composition varied among formulas, the observed differences cannot be attributed solely to extract concentration. The study therefore provides preliminary evidence for formulation rather than definitive proof of colour stability, shelf life, long-term safety, or an anthocyanin concentration-response relationship. Further work requires matched formulation design, total anthocyanin analysis, calibrated pH measurements, instrumental colour assessment,

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accelerated and cycling stability studies, panellist-level statistical analysis, and more comprehensive safety testing.

Conflict of Interest

The authors declare that there is no conflict of interest related to this manuscript.

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Supplementary Materials

The stability photographs on day 0 and day 30 can be provided as supplementary material upon request.

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