

Formulation of Robusta Coffee Bean (*Coffea Robusta* Lindl. Ex De Will) Ethanol Extract Peel-off Mask Preparation

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ABSTRACT

Ultraviolet rays and other factors that form free radicals can cause accelerated skin aging. Robusta coffee (*Coffea robusta* Lindl. Ex De Will) contains xanthin alkaloids and chlorogenic acid that has antioxidant activity. This is an experimental study that aims to formulate ethanol from robusta coffee beans into a peel-off mask preparation and test various quality parameters. Robusta coffee bean ethanol extracted by maceration and made into 1%, 3%, and 5% concentration peel-off mask respectively. Peel-off mask preparations then undergo several tests including homogeneity, viscosity, pH, stability, and irritation tests on facial skin. The results showed that robusta coffee bean extract can be formulated as a peel-off mask with homogeneous, non-irritating results, with a pH range of 5.2 - 5.9, and is stable in storage for 12 weeks. The peel-off mask preparation was able to show that it did not cause an irritation reaction when tested on respondents.

Keywords: *Coffea robusta* Lindl. Ex De Will, Peel-Off Mask, Robusta Coffee Beans.

INTRODUCTION

Robusta coffee (*Coffea canephora*) is widely distributed in several countries in Africa and Asia such as India and Indonesia. Robusta coffee is more widely cultivated in Indonesia compared to Arabica coffee (Saehu et al., 2022). Robusta coffee extract is high in antioxidants which can be a valuable nutraceutical source that can be used to prevent/treat neurodegeneration (Angeloni et al., 2021).

Ultraviolet rays from sunlight, air pollution, stress, and factors that cause free radicals can cause skin aging. Damage caused by free radicals can cause collagen protein molecules to not form or separate from their compounds and reunite with other molecules that are not suitable, causing skin tissue to become hard and inflexible, resulting in fine lines as the hallmark of wrinkles (Gallagher & Lee, 2006). According to the survey, the main cause of premature aging experienced by Indonesians is excessive activity under the sun, which in a tropical climate with abundant sunlight can cause a high risk of skin damage that leads to premature aging. Therefore, anti-aging preparations are considered essential for skin care (Setiati, 2008). Clay-type face masks have been widely used for their ability to rejuvenate the skin. The skin changes when the mask starts to have a pulling effect on the skin as it dries. This sensation stimulates a refreshing sensation where the paste-type clay can remove impurities from the face. Dirt and blackheads are removed when the preparation is washed off the skin. The after-effect of the mask is bright and clear skin (Desi Waliasih et al., 2022).

Chemical compounds that have antioxidant activity can serve to rejuvenate the skin, and various natural ingredients from plants contain many chemical compounds that have antioxidant activity and have been used for skin care, for example, coffee beans. Chemical compounds in coffee beans consist of volatile and non-volatile compounds that affect the aroma and quality of coffee beans. Coffee beans contain various compounds, including caffeine, which is a xanthin alkaloid, and chlorogenic acid, including a class of polyphenolic compounds that have antioxidant activity. The polyphenol content of Robusta coffee beans is higher than Arabica coffee beans or other types of coffee beans (Yasir et al., 2022).

The use of coffee beans for facial skin care directly on the facial skin is less practical, so it needs to be made in

a dosage form, and the most preferred preparation for facial care is the form of a mask. Based on the description above, this study aims to make ethanol extract from robusta coffee beans and formulate it into a peel-off mask preparation and tested various quality parameters of the master preparation and as anti-aging on the skin of the respondents.

METHOD

This is an experimental study that aim to formulate ethanol from robusta coffee beans into a peel-off mask preparation and test various quality parameters. The formulation of anti-aging peel-off mask preparations using robusta coffee bean extract (*Coffea robusta* Lindl. Ex De Will) with concentrations of 1, 3, and 5% will undergo several tests, including homogeneity test, organoleptic, pH measurement, drying time test, stability check, viscosity determination, and irritation test on respondents. Respondents who were recruited for the irritation test and the ability of the preparation amounted to 12 people with the following criteria:

1. Healthy woman
2. Age between 20-35 years old
3. No documented allergy-related diseases
4. Agree to participate

The research was conducted from April to August 2023 at the Pharmacy Laboratory of STIKes Muhammadiyah Lhokseumawe.

Materials and Equipment

The materials used in this study include Robusta coffee beans, 80% ethanol, polyvinyl alcohol (PVA), carbomer 940, sodium lauryl sulfate, glycerin, methylparaben, distilled water, and buffer solution at pH 4.01 and 7.01. The equipment includes glassware, porcelain mortar, stamper, porcelain cup, parchment paper, spatula, spoon, glass pot, stirring rod, aluminum foil, dark glass container, funnel, filter paper, rotary evaporator, skin analyzer and moisture checker, pH meter, analytical balance, and Brookfield viscometer.

Formulation Preparation

Coffee bean sampling

Sampling of coffee beans was carried out purposively without comparing samples with the same plants from other regions. The samples used were robusta coffee beans (*Coffea robusta* Lindl. Ex De Will) from Takengon District, Aceh, Indonesia.

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Processing of coffee bean samples

Coffee beans that can already be harvested are separated from the stem and the skin is separated from the seeds. Then the coffee beans are washed and dried in a drying cabinet at a temperature of about 40°C. Next, it is pulverized using a blender and then stored in a dry and tightly closed container at room temperature.

Preparation of robusta coffee bean extract (*Coffea robusta* Lindl. Ex De Will) was carried out by maceration using 80% ethanol solvent. A total of 600 grams of robusta coffee powder was put into a dark glass container, added with 4.5 liters of 80% ethanol until submerged, closed, and kept at room temperature for 5 days

protected from light while occasionally stirring, then filtered to obtain the macerate. Next, the pulp was macerated again (re-maceration) with 80% ethanol as much as 1.5 liters for 2 days using the same procedure. This re-maceration was repeated until it was fully extracted, that is, the last macerate was clear (colorless). All the macerates were combined, blotted, and poured. Then, the collection of mass obtained was distilled using a rotary evaporator at a temperature of 40°C until a thick extract was obtained. Then the thick extract is evaporated on a water bath with the lowest possible temperature of about 60°C until a dry extract is obtained. Then, it is stored in a well-closed container, then called ethanol extract of robusta coffee beans (EERCB).

Peel-Off mask base formula

The peel-off mask base formulation was made based on the formula from (Birck et al., 2014). In this study, the peel-off mask base formula is a modified formula that does not use perfume and coloring. The preservative used is nipagin 0.02% and 96% alcohol used as much as 20%. The modified formula is as follows:

- | | |
|--------------------------|-----------|
| 1. Polyvinyl Alcohol | 10 g |
| 2. Carbomer 940 | 0.5 g |
| 3. Glycerin | 10 g |
| 4. Sodium Lauryl Sulfate | 2 g |
| 5. Ethanol 96% | 20 g |
| 6. Nipagin | 0.2 g |
| 7. Distilled water | ad 100 ml |

All ingredients were weighed, a beaker glass was poured, then polyvinyl alcohol was added to distilled water, heated in a water bath until it expanded completely, then stirred homogeneously. Then carbomer 940 was expanded in hot distilled water as much as 20 times the weight of carbomer 940, allowed to stand for 15 minutes to fully expand, and stirred homogeneously to form mucilago. Then added to the polyvinyl alcohol phase and stirred until homogeneous. Followed by nipagin, sodium lauryl sulfate which has been dissolved in hot distilled water. Next, glycerin was added, stirred until homogeneous, and then allowed to cool. After that, 96% ethanol was added and stirred homogeneously to form a peel-off mask base.

Formulation of peel-off mask preparation

Peel-off masks were made in 4 formulas, namely formula 0 (blank), formula 1 (1% EERCB concentration), formula 2 (3% EERCB concentration), and formula 3 (5% EERCB concentration). Each mask preparation formula was made as much as 200 g.

Table 1. EERCB peel-off mask dosage formula

Material	Concentration (%)			
	F0 (Blank)	F1 (EERCB 1%)	F2 (EERCB 3%)	F3 (EERCB 5%)
EERCB	-	1 g	3 g	5 g
Peel-off mask base	ad 100	ad 100	ad 100	ad 100

Quality Testing

Determination of the physical quality of the preparation is carried out on the peel-off mask preparation

including homogeneity test, organoleptic observation, pH measurement, testing the drying time of the preparation, checking the stability of the preparation, and determining the viscosity of the peel-off mask preparation.

The homogeneity test is carried out using a glass object. A certain amount of the preparation is applied to a piece of glass and scratched with another piece of glass, the preparation should show a homogeneous arrangement, and no coarse grains are visible. Organoleptical observations include the shape, color change, and odor of the peel-off mask preparation which are observed visually. Determination of the pH of the preparation was carried out using a pH meter. The device was first calibrated using a neutral pH standard buffer solution (pH 7.01) and an acidic pH buffer solution (pH 4.01) until the device showed the pH value. Then the electrode is washed with distilled water, then dried with a tissue. Samples were made in a concentration of 1%, namely weighing 1 g of the preparation and dissolving it in distilled water up to 100 ml. Then the electrode is dipped in the solution and the pH value is observed.

Dry time testing was carried out by applying a peel-off mask to a portion of the face with a cheek area of approximately 1 mm thick and observing the time required for the preparation to dry, namely the time from when the peel-off mask was applied until a dry layer was completely formed. Observation of the stability of the preparation was carried out at room temperature storage for 12 weeks with an observation time interval of every week which included changes in color, odor, pH, drying time, and viscosity. Viscosity testing was carried out using a Brookfield viscometer.

The irritation test was carried out on the robusta coffee bean extract peel-off mask preparation to know whether the peel-off mask can irritate the skin or not. Irritation can be divided into 2 categories, namely primary irritation which will occur immediately after contact or contact with the skin, and secondary irritation whose reaction only occurs several hours after contact or contact with the skin. The irritation test was conducted 3 times a day (morning, afternoon, evening) for 3 consecutive days. A positive irritation reaction is characterized by redness, itching, or swelling of the skin behind the treated ear.

RESULT AND DISCUSSION

Homogeneity, organoleptic, and pH test results

The results of the homogeneity test of the robusta coffee bean extract peel-off mask preparation showed that all preparations applied to transparent glass showed no granule, indicating that the preparations made have a homogeneous composition. The results of organoleptic observations of robusta coffee bean peel-off mask preparations showed a brownish-yellow color and a distinctive smell of coffee beans.

Table 2. Results of pH Measurement for 12 weeks

Formula	Time (Week)											
	1	2	3	4	5	6	7	8	9	10	11	12
F0	6,2	6,2	6,2	6,2	6,2	6,2	6,2	6,2	6,2	6,2	6,2	6,2
F1	5,9	5,9	5,9	5,9	5,9	5,9	5,9	5,8	5,8	5,8	5,8	5,8
F2	5,4	5,4	5,4	5,4	5,4	5,4	5,4	5,4	5,4	5,3	5,3	5,3
F3	5,3	5,3	5,3	5,3	5,3	5,3	5,3	5,3	5,2	5,2	5,2	5,2

Table 2 shows that the results of the pH test of peel-off mask preparations containing ethanol extract of robusta coffee beans obtained a pH ranging from 5.2 to 5.9. As the amount of robusta coffee bean extract increased, the pH of the preparation became more acidic. This may be caused by the acidic pH of robusta coffee bean extract, which is 4.0.

Dry time, stability of preparation, and viscosity test results

Based on Table 3, the results obtained show that the longer the storage, the time required for the peel-off mask preparation containing robusta coffee bean ethanol extract to dry increases.

Table 3. Results of dry time test for 12 weeks

Formula	Dry time (Minutes)											
	1	2	3	4	5	6	7	8	9	10	11	12
F0	17	17	17	17	17	17	17	17	17	18	18	18
F1	17	17	17	17	17	17	17	18	18	18	19	19
F2	17	17	17	17	17	17	18	18	18	19	19	19
F3	17	17	17	17	18	18	18	19	19	19	20	20

Evaluation of the stability of the preparation was carried out during 12 weeks of storage with weekly observation intervals. The peel-off mask preparation was stored at room temperature and observed for changes in odor and color. The test results showed that the peel-off mask preparation containing ethanol extract from robusta coffee beans did not change during 12 weeks of storage. The results of observations of the stability of the preparation for 12 weeks can be seen in Table 4.

Table 4. Results of stability of preparation test for 12 weeks

10	Parameter	Weeks											
		1	2	3	4	5	6	7	8	9	10	11	12
F0	Color	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
	Odor	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
F1	Color	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
	Odor	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
F2	Color	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
	Odor	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
F3	Color	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
	Odor	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)

(+) changes present; (-) no changes.

Table 5 shows that peel-off mask preparations made containing ethanol extract of robusta coffee beans have decreased viscosity over time, this can be caused by the length of storage of preparations affected by the environment such as air.

Table 5. Results of viscosity test for 12 weeks

Formula	Viscosity											
	1	2	3	4	5	6	7	8	9	10	11	12
F0	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 000	13. 000	13. 000

F1	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 225	13. 000	13. 000	13. 000	13. 000
F2	13. 000	13. 000	13. 000	13. 000	13. 000	13. 000	13. 000	12,275	12,275	12,275	11,950	11,950	11,950
F3	13. 000	13. 000	13. 000	13. 000	13. 000	12. 125	12. 125	12. 125	12. 125	11,555	11,555	11,555	11,555

Results of Irritation Test on Respondents' Skin

Table 6. Results of Irritation test

Observation	Respondents												
	1	2	3	4	5	6	7	8	9	10	11	12	
Erythema	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
Pruritus	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)
Edema	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)

(-) no reaction; (+) erythema; (++) pruritus; (++++) edema.

The results of the irritation test conducted on 12 respondents showed that all respondents gave negative results on the irritation reaction parameters (Table 6).

DISCUSSION

A peel-off gel mask was successfully formulated by utilizing the water fraction extract of robusta coffee beans (*Coffea canephora*). The peel-off gel mask formula contains PVA which plays a role in the peel-off effect due to its sticky nature so that it can form a film that is easily removed after drying (Birck et al., 2014). This standard formula was modified in that some ingredients were removed, namely without the use of perfume, and colorants. Robusta coffee bean extract used to make anti-aging peel-off mask preparations are concentrations of 1%, 3%, and 5%. The mask preparation obtained is a brown peel-off mask, with a distinctive odor.

The robusta coffee bean extract peel-off mask preparation proved to be homogeneous after the homogeneity test was carried out with transparent results when applied to glass and no granules. When a preparation shows the results of smearing or distribution that there are no coarse particles and the color of the preparation is evenly distributed, it is declared homogeneous (Pramushinta & Ajiningrum, 2018). The results of organoleptic observations of the robusta coffee bean peel-off mask preparation show a brownish-yellow color and a distinctive smell of coffee beans, the organoleptic test of preparations containing robusta coffee extract has test results, namely preparations that are brownish and smell typical of coffee (Wulandari & Agustin, 2022).

The results of the pH examination of peel-off mask preparations showed pH ranged from 5.2 to 5.9. The pH test results indicate that the more the preparation becomes more acidic as more robusta coffee bean extract is added. The suitability of the pH of the preparation with the pH of the skin will affect the skin's acceptance of the preparation. pH preparations that are too acidic will cause skin irritation and vice versa, pH that is too alkaline can cause dry and dull skin effects (Sulastri & Rahmiyati Jurusan Farmasi Fakultas, 2016).

After the irritation test was conducted for 12 days, monitoring was carried out for symptoms of erythema, pruritus, and edema. The test results showed that this peel-off mask did not cause significant side effects in all respondents. Previous research reported that cascara extract cream from Gayo Arabica coffee grounds was safe to use in Wistar rats and was able to reduce erythema and edema due to UV exposure. However, the use of cascara extract cream from Gayo Arabica coffee grounds did not provide a significant decrease in TNF- α

levels (Lestari & Hajar, 2023).

The results obtained show that the longer the storage, the time required for the peel-off mask preparation containing ethanol extract of robusta coffee beans to dry increases. Evaluation of dry time must be done to determine the time it takes for the mask preparation to dry and form a film layer when used, the dry time requirement for peel-off masks is 15-30 minutes (Setiyadi & Qonitah, 2020). Judging from the results of the robusta coffee bean mask evaluation in the range of 17-20 minutes, it shows that the dry time of this preparation is good.

There was no change observed in stability during 12 weeks of storage. A viscosity test is very important to determine the spreadability parameters of a preparation, the test results show that the peel-off mask preparation made containing ethanol extract of robusta coffee beans has decreased in viscosity, this can be caused by the length of storage of the preparation affected by the environment such as air. The increase in viscosity is caused by the hydroxyl group contained in polyvinyl alcohol that has not undergone a complete hydration process during manufacture (Santoso et al., 2020).

The stability of peel-off masks during storage can be improved by adding some additives such as phenoxyethanol. The results of stability tests at low temperature ($5 \pm 1^\circ\text{C}$) and room temperature, and protected from sunlight ($22 \pm 2^\circ\text{C}$) did not affect the stability of the formulation, and that phenoxyethanol at 1.0% w/w could preserve the formulation from microbial degradation under various storage conditions (Salmannejad et al., 2024).

The irritation test of the preparation was also carried out on 12 respondents, by applying the preparation behind the ear, the results showed no reaction. This irritation test is to determine the safety level of the preparation, the results that do not show irritation are also influenced by the pH of the preparation which has met for topical preparations. The results of the irritation test are very good with peel-off mask preparations that meet the pH according to the pH range of the skin (Rosaini et al., 2021).

The peel mask is proven to have a pH with a safe range as a topical preparation and also has very good irritation test results after 12 days of monitoring, the high antioxidant content of robusta coffee is believed to be useful as anti-aging and also maintains skin brightness. Robusta coffee skin extract has the potential to be a natural antioxidant and can be made into a facial serum gel preparation (Maysarah et al., 2024). Coffee is also believed to have promising biological activities as a candidate for the development of functional foods and/or medicinal ingredients in the fight against free radicals that cause the aging process (Hayes et al., 2023).

CONCLUSION

The results of this study showed that homogeneity, organoleptic, and pH test results have shown the safety of robusta coffee extract peel-off masks as one of the topical preparation formulas. In addition, the results of the irritation test also showed that 12 days of use did not show symptoms of erythema, pruritus, and edema on the skin. Based on the results of this study, it can be concluded that robusta coffee bean extract (*Coffea robusta* Lindl. Ex De Will) can be formulated in the form of a peel-off mask preparation as an effective anti-aging that is stable and safe for use from skin irritation.

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