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Formulation and Antioxidant Evaluation of Clay Masks Containing Robusta Coffee Pulp Extract

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Abstract: The pulp of robusta coffee (*Coffea canephora* L.) is known to contain chlorogenic acid compounds that have potential as antioxidants. The content of this compound can be utilized as an active ingredient in cosmetics such as clay masks. The main component that affects the physical properties of a clay mask is the clay base. This study aims to develop a clay mask formulation from robusta coffee pulp extract with variations in the concentration of base combinations, namely bentonite and kaolin, and to observe their effects on the physical properties of the dosage form. The base concentration variations used were 20% (F1), 25% (F2), and 30% (F3) with a bentonite to kaolin ratio of 1:2. The evaluation of physical properties included organoleptic tests, homogeneity tests, pH tests, spreadability tests, adhesion tests, viscosity tests, and drying time measurements. The evaluation results showed that all clay masks were homogeneous with pH values ranging from 5.15 ± 0.04 to 5.29 ± 0.12, spreadability values of 4.60 ± 0.22 to 4.74 ± 0.37 cm, adhesion values of 0.82 ± 0.11 to 1.16 ± 0.15 seconds, and viscosity ranged from 5501 ± 670.22 to 7567.67 ± 220.30 mPa.s, and drying time ranged from 20.60 ± 0.39 to 25.34 ± 0.80 minutes. Results of the antioxidant activity test indicated that the clay mask made from robusta coffee pulp extract is a powerful antioxidant with IC_{50} values of 28.31 ± 4.72 to 30.45 ± 5.69 ppm. Variations in the concentration of the clay mask base significantly ($p < 0.05$) affected the viscosity and drying time of the dosage form. In summary, the clay mask formulations satisfied the essential physical criteria and showed promise for cosmetic application. F3 emerged as the leading formula, exhibiting the highest antioxidant capacity, evidenced by its lowest IC_{50} value.

Keywords: antioxidants, clay mask, coffee bean pulp, formulation, physical properties

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1. INTRODUCTION

Coffee pulp is a by-product of coffee bean processing that possesses various beneficial properties. Robusta coffee pulp contains several secondary metabolites, including caffeine and polyphenolic compounds, which exhibit natural antioxidant activity (Harahap, 2017). The extract of coffee pulp has demonstrated antioxidant potential with an IC_{50} value of 99.78 ppm (Yasir et al., 2023). The antioxidant activity of the extract can be more effectively utilized when formulated into a topical dosage form, as the active compounds can directly interact with the facial skin and remain active for a more extended period, thereby optimizing their antioxidant effects (Kumalasari et al., 2023). A peel-off mask formulation containing 5% coffee pulp extract has been reported to exhibit potent antioxidant activity (Nursal et al., 2024). However, peel-off masks are less effective in performing deep pore cleansing and may irritate the peeling process, particularly on sensitive skin (Zhang et al., 2023). Therefore, the development of a clay mask formulation was carried out to optimize the use of coffee pulp extract as an antioxidant agent.

Clay masks are a type of facial mask renowned for their ability to hydrate and nourish the skin, thanks to the properties of clay bases, which have been proven to help maintain skin moisture (Kumalasari et al., 2023). During application, clay masks dry quickly, cleanse pores effectively, possess excellent absorption capacity, and do not cause irritation on normal skin. The clay base serves as the primary component in clay mask formulations, with kaolin and bentonite being the most commonly used types (Febriani and Sembiring, 2021; Indriastuti et al., 2022).

Kaolin is a natural mineral obtained through purification and drying processes. It is water-dispersible, has a pH similar to that of the skin, and exhibits good permeability and oil absorption capacity (Elfiyani et al., 2023; Indriastuti et al., 2022). According to Elfiyani et al., (2023), the use of kaolin and bentonite at a concentration of 20% produced clay masks that met pharmaceutical standards. However, formulations containing bentonite alone exhibited poor spreadability, necessitating its combination with kaolin to achieve optimal texture and performance (Nugrahaeni et al., 2025; Tungadi et al., 2024).
Based on these findings, this study aims to investigate the effect of varying concentrations of bentonite and kaolin on the physical properties and antioxidant activity of clay masks formulated with Robusta coffee pulp extract. The base concentrations used were 20%, 25%, and 30%, with a bentonite-to-kaolin ratio of 1:2.

2. MATERIAL AND METHODS

2.1 Material

The materials used in this study included dried Robusta coffee (Coffea canephora L.) pulp obtained from Sukawangi Village, Bogor Regency; bentonite (PT. Palapa Muda Perkasa); kaolin (PT. Yudian Kawan Mineral); xanthan gum (PT. Fufeng); propylene glycol (PT. Palapa Muda Perkasa); triethanolamine (PT. Palapa Muda Perkasa); methylparaben (PT. Palapa Muda Perkasa); distilled water (PT. OneMed); ethanol 96%; DPPH powder (Sigma-Aldrich); $FeCl_3$ (PT. Merck); ascorbic acid (PT. Merck); and methanol p.a (PT. Merck).

2.2 Methods

This study employed an experimental method with the concentration of the clay base as the independent variable. The clay base used was a combination of bentonite and kaolin at a ratio of 1:2 with varying total concentrations of 20% (F1), 25% (F2), and 30% (F3). The active ingredient incorporated into the clay mask formulation was a concentrated extract of coffee pulp at a concentration of 5%.

a. Preparation and Characterization of Coffee Pulp Extract

The coffee pulp simplicia obtained was first subjected to plant determination prior to extraction. The determination was conducted at the National Research and Innovation Agency (BRIN), Cibinong. The extraction process was carried out using the maceration method with 96% ethanol as the solvent at a 1:10 ratio. A total of 300 grams of simplicia was weighed, placed in a brown glass container, and soaked in 96% ethanol until fully submerged. The sample was macerated for 3 × 24 hours with occasional stirring every six hours, followed by filtration using filter paper and a sterile funnel to separate the filtrate from the residue. The residue was then remacerated with a fresh portion of solvent. The combined filtrates were concentrated using a rotary evaporator at 50°C. The concentrated extract obtained was weighed, and its percentage yield was calculated. The concentrated extract was characterized by organoleptic evaluation, qualitative analysis of phenolic compounds, determination of ash content, and loss-on-drying analysis using a moisture balance (Mettler Toledo HB-43S) (Nursal et al., 2024).

b. Preparation of Robusta Coffee Pulp Extract Clay Mask

The first step in preparing the clay mask was to sieve bentonite and kaolin using a 100-mesh sieve. All ingredients were weighed using an analytical balance (OHAUS) according to the formulation shown in

Table 1. The base was prepared by dispersing bentonite and kaolin in a portion of distilled water in a glass beaker, then homogenization with a homogenizer (Heidolph Torque) at 500 rpm until a uniform mixture was obtained. Xanthan gum, previously dispersed in distilled water, was then added to the base and mixed until a homogeneous mixture was achieved (M1). In a separate porcelain dish, methylparaben was dissolved in propylene glycol (M2). The next step was to mix M2 with the extract until fully dissolved (M3). Finally, M1 and M3 were combined and homogenized at 1000 rpm, followed by the addition of the remaining distilled water and triethanolamine (TEA) (Elfiyani et al., 2023; Nugrahaeni et al., 2025). The final mixture was re-homogenized, assessed for physical properties, and assayed for antioxidant activity using the DPPH method.

Table 1: Formulation of Robusta Coffee Pulp Extract Clay Mask

Component	Concentration (% w/w)			Function
	F1	F2	F3	
Robusta Coffee Pulp Extract	5	5	5	Active ingredient
Bentonite	7	8.3	10	Mineral clay
Kaolin	13	16.7	20	Mineral clay
Xanthan Gum	1	1	1	Thickening agent
Propylene Glycol	7	7	7	Humectant
TEA	0.7	0.7	0.7	Alkalizing agent
Metilparaben	0.2	0.2	0.2	Preservative
Distilled water ad	100	100	100	Dispersing agent

c. Evaluation of the Robusta Coffee Pulp Extract Clay Mask

The clay mask formulations were evaluated through a series of physical tests. Organoleptic assessment was carried out by observing the color, odor, and overall appearance of the product. Homogeneity was assessed by spreading 1 g of the formulation onto a glass slide and inspecting its uniformity under a light to ensure a homogeneous texture. The pH value was determined using a calibrated pH meter (Hanna) with buffer solutions of pH 4 and pH 7. The electrode was immersed in the clay mask formulation until the pH reading on the monitor stabilized (Fauziah et al., 2022; Nugrahaeni et al., 2025). The spreadability test was conducted by placing 1 gram of the formulation between two glass plates. After one minute, the diameter of the spread was measured using a ruler. Additional weights of 50, 100, and 150 grams were subsequently placed on the upper glass plate for one minute each, and the resulting diameters were remeasured. The obtained values were averaged and recorded as the clay mask's spreadability (Ardhany et al., 2022; Nugrahaeni et al., 2025). The adhesion test was performed in triplicate by placing 1 g of the formulation between two glass slides, which were then compressed with a 1 kg weight for 1 minute. The weight was then removed, and the time required for the two slides to separate was recorded (Nugrahaeni et al., 2025). Viscosity was measured to determine the consistency of the formulation using a viscometer (Anton Paar QC 300-R) equipped with spindle No. 6 RH. The sample was placed in a glass beaker, and the spindle was rotated at a speed of 50 rpm (Nursal et al., 2024). Drying time was evaluated by applying 0.5 g of the formulation to a 2 × 3 cm area on the back of the hand and recording the time needed for the mask to become non-sticky, change color, and form cracks, with an acceptable drying duration of 15–30 minutes (Fauziah et al., 2022; Nugrahaeni et al., 2025).

d. Antioxidant Activity Test

The antioxidant activity was evaluated using the DPPH method with vitamin C as the standard, following a modified procedure from Nursal et al., (2024). The test was performed on both the coffee pulp extract and the three clay mask formulations. A 50 mg extract was weighed using an analytical balance (Mettler Toledo MS204) and dissolved in methanol in a 50 mL volumetric flask to obtain a stock solution with a concentration of 1000 ppm. Serial dilutions were then prepared in 10 mL volumetric flasks to yield final concentrations of 12, 16, 20, 24, and 28 ppm. From each concentration, 1 mL of sample solution was transferred into a 10 mL volumetric flask, followed by the addition of 5 mL of DPPH solution. The volume was then adjusted to the mark with methanol, vortexed, and incubated in the dark at 37°C for 30 minutes. The absorbance of each solution was measured using a UV-Vis spectrophotometer (Shimadzu UV-1900i) at a wavelength of 515 nm. The same procedure was applied to the clay mask formulations. However, prior to absorbance measurement, the samples were filtered to ensure solution clarity. The percentage of inhibition (% inhibition) was calculated using the following equation:

$$\% \text{ Inhibition} = \frac{A_{blank} - A_{sample}}{A_{blank}} \times 100$$

The IC_{50} value was determined from the linear regression equation obtained from the relationship between sample concentration (x) and percentage of inhibition (y).

e. Data Analysis

Data analysis was performed on the results of the physical evaluations, including spreadability, adhesion, pH, viscosity, drying time, and antioxidant activity. The data were analyzed using a one-way ANOVA test (IBM SPSS Statistics 25.0) with a 95% confidence level ($\alpha = 0.05$). When significant differences were observed among the formulations, further analysis was conducted using Tukey's HSD post hoc test to determine the specific differences between formulations.

3. RESULTS AND DISCUSSION

a. Characterization of Robusta Coffee Pulp Extract

The coffee pulp used in this study was confirmed to originate from the Coffea robusta plant, as evidenced by the determination certificate No. B-256/II.6.2/IR.01.02/1/2025. The results of the extract characterization are presented in Table 2. The yield obtained was 16.28%, corresponding to 97.7 grams of concentrated extract from 600 grams of simplicia. The yield value met the standard requirement for good extraction yield, which is greater than 10%. A higher yield indicates that a larger amount of extractable compounds was obtained (Saerang et al., 2023). An ash content test was subsequently conducted to determine the mineral content present both within and outside the extract after the destruction process. The ash content of the Robusta coffee pulp extract was 9.69% ± 0.11%, which meets the maximum permissible limit of <10% (Kemenkes RI, 2017).

Table 2: Characterization Results of *Robusta Coffee* (Coffea canephora L.) Pulp Extract

Characterization	Observation Result	Specification
Form	Viscous	-
Color	Dark brown	-
Odor	Characteristic coffe aroma	-
Yield	16.28 %	>10% (Saerang et al., 2023)
Ash Content	9.69 % ± 0.11 %	<10% (Kemenkes RI, 2017)
Loss on Drying	11.03 % ± 2,48 %	<10% (Kemenkes RI, 2017)
Phenolic Test	(+) Dark green	Dark green (Bawekes et al., 2023)

The loss on drying Test yielded an average value of 11.03% ± 2.48%, which slightly exceeded the required limit of <10%. A high loss-on-drying value indicates a high residual moisture content, which can promote microbial growth in the extract. This condition may result from incomplete drying of the simplicia, leaving residual water in the extract (Krismayadi et al., 2024). The qualitative phenolic Test showed a color change to dark green after the addition of 1% $FeCl_3$ reagent, indicating the presence of phenolic compounds in the Robusta coffee pulp extract. The Fe^{3+} ions in $FeCl_3$ react with hydroxyl groups of phenolic compounds, forming a complex that produces a dark green coloration (Bawekes et al., 2023).

b. Organoleptic and Homogeneity Evaluation of Clay Mask Containing Robusta Coffee Pulp Extract

The organoleptic evaluation of the clay mask formulated with Coffea robusta husk extract revealed a thick consistency, a grayish-green color, and a characteristic coffee aroma. As shown in Table 3, formulations F1 and F2 exhibited a thick consistency, while F3 demonstrated a very thick texture. This variation in consistency may be attributed to differences in base concentration. An increase in the base concentration results in a denser clay mask, as the amount of water used decreases (Elfiyani et al., 2023). A homogeneity test was subsequently performed to ensure uniformity of texture across the formulations. The results indicated that all three formulations (F1, F2, and F3) were physically homogeneous, as no particle granules were observed and the color of the clay mask appeared uniformly distributed.

Table 3: Organoleptic Evaluation of Clay Mask Containing Robusta Coffee Husk Extract (n = 3)

Formula	Concistency	Color	Odor
F1	Thick	Dark gray	Characteristic coffe aroma
F2	Thick	Dark gray	Characteristic coffe aroma
F3	Very thick	Dark gray	Characteristic coffe aroma

c. pH Evaluation of Clay Mask Containing Robusta Coffee Pulp Extract

The pH measurement was conducted to ensure that the formulated product possessed a pH value suitable for skin application, typically within the range of 4.5–6.5 (Fauziah et al., 2022). The results demonstrated that all clay mask formulations complied with the acceptable pH range for topical products. The pH values obtained are presented in Figure 1.

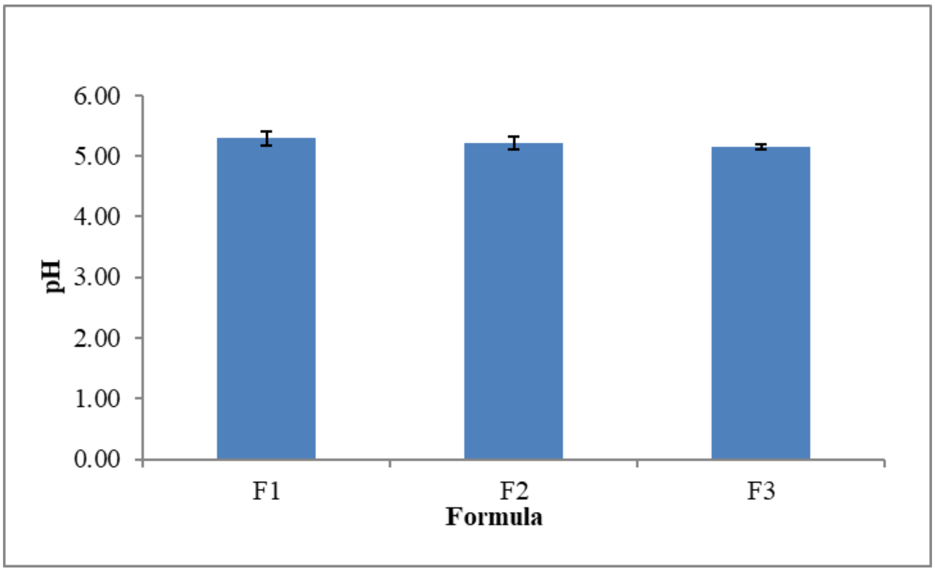

Figure 1. pH Evaluation Results of Clay Mask Containing Robusta Coffee Pulp Extract (n = 3)

The results showed that the pH values of the clay mask formulations ranged between 5.15 ± 0.04 and 5.29 ± 0.12. Based on the evaluation, the pH of the clay mask decreased as the concentration of the base increased. This phenomenon can be attributed to the higher proportion of kaolin used at higher base concentrations. Kaolin exhibits a slightly acidic pH (4–5.5), whereas bentonite tends to be alkaline (pH 9–10.5). Therefore, increasing the proportion of kaolin in the formulation results in a lower overall pH. Nevertheless, all formulations remained within the acceptable pH range for topical products. A formulation that is too acidic may cause skin irritation, while an excessively alkaline formulation can lead to skin dryness (Fauziah et al., 2022). These findings are consistent with the report by Syamsidi et al. (2021), which indicated that clay mask formulations incorporating a kaolin–bentonite base with a

higher kaolin proportion tend to produce preparations with pH values approaching neutrality.
The pH data were further analyzed statistically using one-way ANOVA. The normality and homogeneity tests indicated that the data were normally distributed and homogeneous ($p > 0.05$), allowing for subsequent analysis using ANOVA. The ANOVA results showed a significance value greater than 0.05, indicating no statistically significant differences among the pH values of the clay mask formulations. These findings suggest that variations in base concentration did not significantly affect the pH of the clay mask containing Coffea robusta husk extract.

d. Spreadability Test of the Clay Mask Containing Robusta Coffee Pulp Extract

The spreadability test was designed to assess the formulation's ability to spread when applied to the skin. The results obtained for all formulations ranged from 4.6 to 4.74 cm, as presented in Table 4. The spreadability test results indicated that the formulated clay mask exhibited a rigid consistency, as reflected by its spreadability value of less than 5 cm (Amalia et al., 2021). Nevertheless, the obtained values still met the acceptable spreadability criteria for clay masks, as reported by Syamsidi et al. (2021).

Table 4: Results of Spreadability, Adhesion, and Drying Time Tests (n = 3)

Formula	Spreadability (cm)	Adhesion (s)	Drying time (minutes)
F1	4.73 ± 0.28	0.97 ± 0.23	25.34 ± 0.80
F2	4.74 ± 0.37	0.82 ± 0.11	22.54 ± 0.52
F3	4.60 ± 0.22	1.16 ± 0.15	20.60 ± 0.39

Based on the results obtained, F2 exhibited the highest spreadability value. This may be attributed to the use of kaolin, which is less prone to swelling compared with bentonite and therefore tends to form a looser matrix. Such characteristics can increase the clay mask's spreadability (Shakeel et al., 2021). The normality test yielded a significance value of 0.341 ($p > 0.05$), indicating that the data were normally distributed. The homogeneity test also showed homogeneous data ($p > 0.05$). Subsequent one-way ANOVA analysis revealed a significance value of 0.833 ($p > 0.05$), indicating no significant differences among formulations. Thus, variations in base concentration did not affect the spreadability of the clay mask formulations.

e. Adhesion Test of the Clay Mask Containing Robusta Coffee Pulp Extract

The adhesion test was conducted to evaluate the formulation's ability to adhere to the skin surface upon application. The longer the contact time between the formulation and the skin, the greater the potential for active ingredient absorption (Tungadi et al., 2024). The clay mask is considered to have good adhesion if the result is greater than 1 second (Dipahayu and Lestari, 2021). The adhesion values of the formulations ranged from 0.97 to 1.16 seconds (Table 4). F3, with a 30% base concentration, exhibited the highest adhesion time of 1.16 seconds. The results of F1 and F2 did not meet the requirements, likely due to the lower base concentration, which made the formulation less viscous, and the insufficient xanthan gum concentration as a thickening agent (Widyaningrum et al., 2012).
Statistical analysis using one-way ANOVA revealed that the adhesion data were normally distributed and homogeneous ($p > 0.05$). The ANOVA test showed a significance value greater than 0.05, indicating no significant differences among the adhesion values of the clay mask formulations. Therefore, variations in base concentration did not affect the adhesion of the Coffea robusta husk extract clay mask formulations.

f. Viscosity Test of the Clay Mask Containing Robusta Coffee Pulp Extract

The viscosity test was conducted to determine the thickness of the formulation, as viscosity influences flow behavior—higher viscosity results in slower flow (Elfiyani et al., 2023). The acceptable viscosity range for clay mask formulations is 4,000–40,000 mPa·s (Zainal et al., 2023). As shown in Figure 2, the viscosity values of all formulations fell within this range, ranging from 5,501 to 7,567.67 mPa·s.

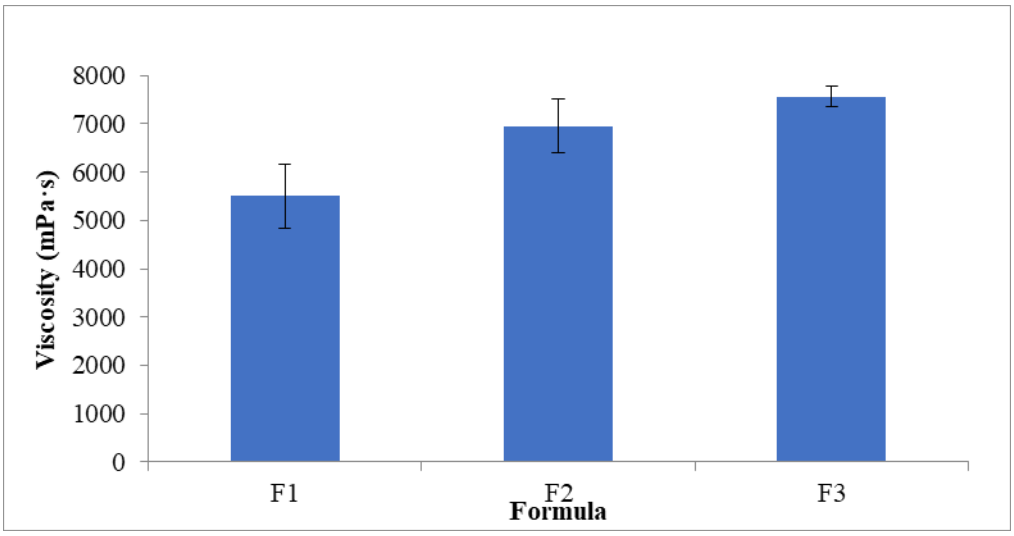

Figure 2. Viscosity Test Results of Clay Mask Containing *Robusta Coffee* Pulp Extract (n = 3)

Normality and homogeneity tests yielded significance values greater than 0.05, indicating that the data were normally distributed and homogeneous. Therefore, a one-way ANOVA was performed, yielding a significance value of 0.01 ($p < 0.05$), indicating significant differences among formulations. Further analysis using Tukey's post hoc test revealed significant differences between F1 and F2 and between F1 and F3 ($p < 0.05$). These findings indicate that variations in base concentration significantly affected the viscosity of the clay mask formulations. This can be attributed to the characteristics of bentonite and kaolin, which are mineral clays with high water absorption and swelling capacities. Bentonite contains interlayer spaces that trap water molecules, forming colloidal dispersions, while kaolin contains hydroxyl (-OH) groups that bind water, increasing viscosity (Elfiyani et al., 2023). Therefore, increasing the base concentration leads to higher viscosity (Zainal et al., 2023).

g. Drying Time Test of the Clay Mask Containing Robusta Coffee Pulp Extract

The drying time test was conducted to determine the time required for the mask to dry, measured from the moment of application to the skin until the mask layer was dehydrated. A clay mask is considered dry when the applied layer is no longer wet, its color changes, and small cracks appear. The ideal drying time for a clay mask is 15–30 minutes (Fauziah et al., 2022). All formulations (F1–F3) met this requirement. Formula F1 had the longest drying time, while F3 dried the fastest (Table 4). Normality and homogeneity tests showed significance values greater than 0.05, indicating that the data were normally distributed and homogeneous. A one-way ANOVA revealed a significance value of $p < 0.05$, indicating a significant difference in drying time among formulations. Tukey's post hoc test confirmed that variations in base concentration significantly affected drying Time. The base concentration can explain the differences: higher base concentrations resulted in shorter drying times (Fauziah et al., 2022). Kaolin forms a film easily and absorbs water and oil from the skin surface, thereby accelerating the drying process. Meanwhile, bentonite acts as an adsorbent, reducing water content in the formulation and further shortening drying time ((Elfiyani et al., 2023).

h. Antioxidant Activity Test

The antioxidant activity test was conducted to evaluate the antioxidant potential of the clay mask containing Coffea robusta pulp extract using the DPPH method. The principle of the DPPH assay involves measuring the discoloration of the purple DPPH radical solution as antioxidants donate hydrogen atoms to neutralize free radicals, resulting in a color change from purple to yellow (Jami'ah et al., 2018). The maximum absorbance wavelength for DPPH was found at 515 nm with an absorbance value of 0.457. Methanol was used as the blank and solvent, while vitamin C served as the reference due to its high water solubility and polarity, characteristics similar to the extract tested. Vitamin C is a natural antioxidant with extreme radical scavenging activity against DPPH radical (Wulan et al., 2019).

The IC_{50} values for vitamin C and Coffea robusta husk extract were 20.89 ppm and 26.97 ppm, respectively. IC_{50} values below 50 ppm indicate very strong antioxidant activity (Winahyu et al., 2021). The extract of Coffea robusta pulp exhibits very strong antioxidant activity due to its phenolic content. Phenolic compounds act as antioxidants by donating hydrogen atoms to neutralize free radicals, forming stable phenoxyl radicals through resonance delocalization. Moreover, phenols can chelate transition metal ions such as Fe^{2+} and Cu^{2+}, thereby inhibiting the formation of new free radicals through the Fenton reaction (Shahidi and Ambigaipalan, 2015).

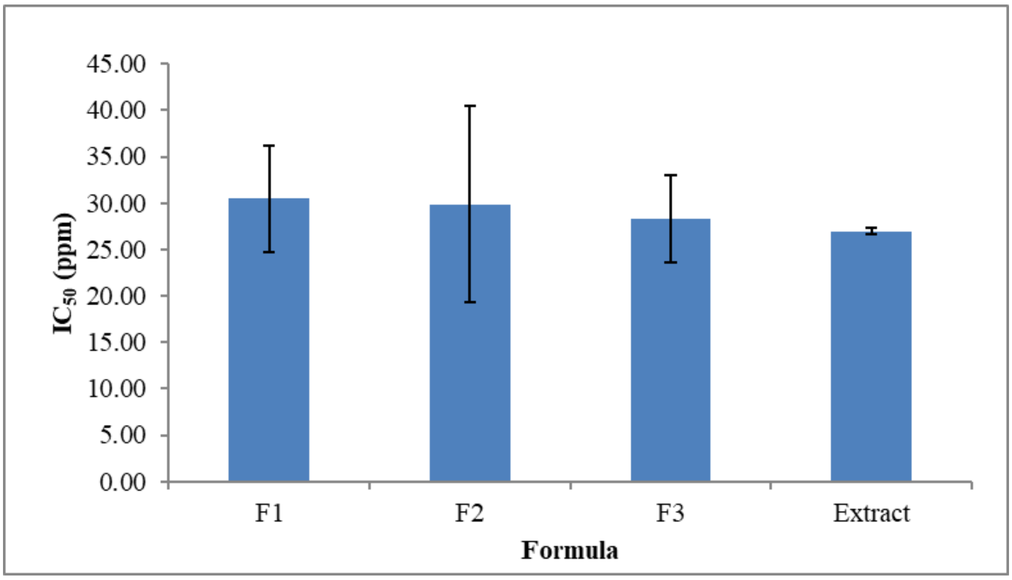

Figure 3. Antioxidant Activity Results of Extract (F4) and Clay Mask Containing Robusta Coffee Pulp Extract (n = 3)

The IC_{50} values of the clay mask formulations were 30.45 ± 5.69 ppm (F1), 29.87 ± 10.63 ppm (F2), and 28.31 ± 4.72 ppm (F3). All formulations demonstrated very strong antioxidant activity. One-way ANOVA analysis yielded a significance value of 0.909 ($p > 0.05$), indicating no significant differences in the antioxidant activities among the formulations. These findings confirm that variations in base concentration did not affect antioxidant activity. Furthermore, no significant difference was observed between the antioxidant activities of the formulations and the pure extract, suggesting that the extract retained its strong antioxidant potential even after being incorporated into a cosmetic formulation, as indicated by IC_{50} values below 50 ppm.

4. CONCLUSION

Variations in base concentration significantly affected the viscosity and drying time of the clay mask formulations but did not influence their pH, spreadability, or adhesion. The Coffea robusta pulp extract clay mask exhibited strong antioxidant activity with IC_{50} values ranging from 28.31 to 30.45 ppm, indicating its potential for further development as a natural-based anti-aging or brightening skincare product. In summary, the clay mask formulations developed in this work met the established physical quality parameters and demonstrated clear potential for advancement into cosmetic applications. Among the formulations evaluated, F3, with the highest concentration of the base, was identified as the most favorable option due to its superior antioxidant performance, as indicated by the lowest IC_{50} value.

5. ACKNOWLEDGMENT

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