

Uji Aktivitas Antioksidan Sediaan *Clay Mask Stick* Ekstrak Daun Buas-Buas (*Premna Serratifolia* L.) Dengan *Green Solvent*

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ABSTRAK

Daun buas-buas (*Premna Serratifolia* L.) merupakan tanaman obat yang mengandung flavonoid dan senyawa fenolik yang berpotensi sebagai antioksidan untuk melindungi kulit dari kerusakan akibat radikal bebas. Pemanfaatan *green solvent* menjadi alternatif dalam pengembangan sediaan kosmetik yang lebih aman. Penelitian ini bertujuan untuk menentukan nilai IC₅₀ ekstrak *green solvent* daun buas-buas serta memformulasikan ekstrak terbaik ke dalam sediaan *clay mask stick*. Ekstraksi dilakukan menggunakan metode *Microwave-Assisted Extraction* (MAE) dengan pelarut madu yang dicampur dengan aquadest (1:20) dan *Natural Deep Eutectic Solvent* (NADES) berbasis asam sitrat dan glukosa (1:1). Aktivitas antioksidan ekstrak dan sediaan diuji menggunakan metode DPPH untuk menentukan nilai IC₅₀. Ekstrak dengan aktivitas terbaik diformulasikan dalam sediaan dan dievaluasi stabilitas fisik serta kimianya menggunakan metode *cycling test*. Hasil menunjukkan bahwa ekstrak NADES memiliki aktivitas antioksidan sangat kuat dengan nilai IC₅₀ sebesar 31.53 ppm, sedangkan ekstrak madu alami tergolong sedang dengan nilai IC₅₀ sebesar 108.53 ppm. Sediaan *clay mask stick* pada hari ke-0 memiliki nilai IC₅₀ sebesar 207.94 ppm (F1) dan 171.57 ppm (F2) yang termasuk kategori lemah, serta 110,882 ppm (F3) yang termasuk kategori sedang. Setelah dilakukan *cycling test* hingga siklus ke-6, terjadi peningkatan nilai IC₅₀ menjadi 434.28 ppm (F1) dan 312.55 ppm (F2) yang termasuk kategori sangat lemah, serta 208.48 ppm (F3) yang termasuk kategori lemah, yang menunjukkan adanya penurunan aktivitas antioksidan setelah *cycling test*. Namun seluruh formula tetap stabil secara fisik dan memiliki pH dalam rentang aman untuk kulit. Penelitian ini menunjukkan potensi pengembangan *clay mask stick* berbasis *green solvent* sebagai sediaan kosmetik antioksidan.

Kata kunci: Clay Mask Stick; Green Solvent; Nilai IC₅₀

Antioxidant Activity of Clay Mask Stick with *Premna serratifolia* L. Extract Using Green Solvents

ABSTRACT

Buas-buas leaves are a medicinal plant containing flavonoids and phenolic compounds with potential antioxidant activity to protect the skin from damage caused by free radicals. The use of green solvents has emerged as an alternative in developing safer and more sustainable cosmetic formulations. This study aimed to determine the IC₅₀ value of buas-buas leaf extracts obtained using green solvents and to formulate the most active extract into a clay mask stick preparation. Extraction was carried out using the MAE method with natural honey mixed with distilled water (1:20) and NADES based on citric acid and glucose (1:1). Antioxidant activity of both extracts and formulations was evaluated using the DPPH method to determine IC₅₀ values. The extract with the best activity was then formulated and further evaluated for physical and chemical stability using a cycling test method. The results showed that the NADES extract exhibited very strong antioxidant activity with an IC₅₀ value of 31.53 ppm, while the natural honey extract showed moderate activity with an IC₅₀ of

108.53 ppm. The clay mask stick formulations on day 0 had IC₅₀ values of 207.94 ppm (F1) and 171.57 ppm (F2), categorized as weak, and 110.88 ppm (F3), categorized as moderate. After six cycles of the cycling test, IC₅₀ values increased to 434.28 ppm (F1) and 312.55 ppm (F2), categorized as very weak, and 208.48 ppm (F3), categorized as weak, indicating decreased antioxidant activity after accelerated storage. However, all formulations remained physically stable and had pH values within a safe range for skin.

Keywords: Clay Mask Stick; Green Solvent; IC₅₀ Value

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INTRODUCTION

The skin is the outermost organ that serves as a protective barrier against environmental exposures such as ultraviolet (UV) radiation, pollution, and dust particles. These exposures can induce the excessive generation of reactive oxygen species (ROS), leading to oxidative stress, which consequently results in skin cell damage, premature aging, hyperpigmentation, acne, and a decline in overall skin quality. Antioxidants play a crucial role in neutralizing free radicals through electron or hydrogen atom donation mechanisms, thereby helping to preserve the integrity and physiological functions of the skin (Febriani *et al.*, 2022). The development of safe and effective natural antioxidant active compounds has become an important focus in the fields of cosmetics and pharmaceutical sciences. *Premna serratifolia* L. leaves are recognized as a potential natural source of antioxidants and are reported to contain secondary metabolites such as flavonoids and phenolic compounds. Flavonoids, including apigenin, luteolin, and quercetin, contribute to free radical scavenging activity through electron stabilization, thereby exhibiting strong antioxidant properties. Previous studies have reported that the ethanolic Buas-Buas Leaf Extract demonstrates very strong antioxidant activity, with an IC₅₀ value of 20.66 µg/mL. However, the use of conventional organic solvents such as ethanol and methanol in such studies still presents limitations, including toxicity, high volatility, and adverse environmental impacts (Adahane *et al.*, 2024).

Green solvents have been developed as environmentally friendly and sustainable alternatives for the extraction of bioactive compounds. In addition to its extraction efficiency, the safety profile of NADES is an important consideration in the development of topical formulations. The NADES used in this study was composed of citric acid and glucose, both of which are naturally occurring compounds that have been widely utilized in pharmaceutical and cosmetic applications due to their low toxicity and favorable biocompatibility. Although residual components of NADES may remain in the extract after the extraction process, citric acid is commonly employed as a safe excipient and pH-adjusting agent in topical products. Therefore, citric acid glucose-based NADES demonstrates considerable potential as an environmentally friendly and safe solvent system for the extraction of bioactive compounds intended for topical herbal formulations. (Martinovi & Neši, 2022). Diluted honey solutions with aquadest (1:20) also have the potential to serve as green solvents due to their sugar and organic acid content, which can improve the solubility and stability of bioactive compounds (Kolayli, 2023). In addition, Microwave-Assisted Extraction (MAE) has been shown to improve extraction efficiency by reducing extraction time, minimizing solvent usage, and preserving the quality of active

compounds (Yusuf *et al.*, 2021). However, studies combining the use of green solvents with MAE for the Buas-Buas Leaf Extract remain limited.

Clay mask stick is an innovative topical dosage form based on mineral ingredients such as kaolin and bentonite, designed in a stick format to enhance practicality, hygiene, and ease of application (Maharani *et al.*, 2024). This formulation functions as a cleanser and sebum absorber for the skin. The incorporation of Buas-Buas Leaf Extract is expected to enhance the functional properties of the formulation through its antioxidant activity. Previous studies have primarily focused on the antioxidant activity of the extracts themselves, whereas the evaluation of antioxidant activity after formulation into a clay mask stick remains limited. This situation indicates a research gap regarding the influence of formulation on the antioxidant activity of the final product. This study presents a novel approach by utilizing a combination of green solvents, namely citric acid–glucose-based Natural Deep Eutectic Solvent (NADES) and honey diluted with aquadest, applied in conjunction with the Microwave-Assisted Extraction (MAE) method for the extraction of Buas-Buas Leaf, as well as their application in a clay mask stick formulation. This study aims to evaluate the antioxidant activity of both the extract and the clay mask stick based on IC_{50} values, to analyze the effect of green solvent usage on the resulting antioxidant activity, and to assess the physical stability of the formulation through a cycling test to ensure product stability during storage.

RESEARCH METHOD

Study Site and Period

This study was conducted at the Biology Laboratory, Chemistry Laboratory, and Pharmaceutical Technology Laboratory, Faculty of Medicine, Tanjungpura University, from October 2025 to January 2026.

Materials

The materials used in this study included *Premna serratifolia* L. leaves, honey, aquadest, glucose, citric acid, analytical grade methanol (p.a.), DPPH (2,2-diphenyl-1-picrylhydrazyl), and vitamin C as a positive control. The clay mask stick base consisted of kaolin, bentonite, cetyl alcohol, glycerin, isopropyl myristate, carnauba wax, lanolin, methyl paraben, and menthol.

Instruments

The equipment used included a set of laboratory glassware, analytical balance, 60 mesh sieve, blender, food dehydrator, microwave extractor, magnetic stirrer, digital pH meter, micropipette, water bath, sonicator, and UV-Vis spectrophotometer.

Methods

Preparation of Simplicia

Buas-Buas Leaf were sorted, washed under running water, cut into smaller pieces, and dried at approximately 50°C until the moisture content decreased. The dried material was then ground and sieved using a 60 mesh sieve to obtain a uniform particle size powder, which was subsequently stored in a closed container protected from light.

Preparation of Green Solvents

Honey solution was prepared by mixing honey with aquadest at a ratio of 1:20, followed by homogenization using a magnetic stirrer at approximately 40°C. NADES (Natural Deep Eutectic Solvent) was prepared by mixing citric acid and glucose at a 1:1 ratio, followed by heating and stirring until a homogeneous solution was formed.

Extraction Process

A total of 15 g of powdered simplisia was extracted with 150 mL of green solvent using the Microwave-Assisted Extraction (MAE) method at 100 watts for 10 minutes. The resulting extract was filtered, and the filtrate was stored in a dark container to prevent degradation due to light exposure.

Phytochemical Screening

Phytochemical screening was conducted to identify the presence of secondary metabolites. Flavonoids were indicated by the formation of a red coloration following the addition of Mg and HCl to 1 mL of extract. Phenolic compounds were identified by a color change from blue to purple after the addition of FeCl₃ to 1 mL of extract. Saponins were indicated by the formation of stable foam after the extract was mixed with aquadest and shaken. Meanwhile, tannins were identified by the appearance of a blue-black or dark green coloration after the addition of FeCl₃ to 1 mL of extract.

Antioxidant Activity Assay Using DPPH Method

Determination of Maximum Wavelength and Incubation Time

A 40 ppm DPPH solution was scanned within the wavelength range of 400–800 nm using a UV-Vis spectrophotometer to determine the maximum absorption wavelength. The DPPH solution was then reacted with the sample solution, and absorbance was measured at 5-minute intervals up to 60 minutes to determine the optimum incubation time, defined as the point at which absorbance reached a stable value.

Preparation of Stock Solutions

Stock solutions of the extract and vitamin C (1000 ppm) were prepared by dissolving 50 mg of each sample in methanol and diluting to a final volume of 50 mL. The solutions were homogenized and stored in dark containers. Working solutions of vitamin C were prepared at concentrations of 2, 4, 6, 8, and 10 ppm. For each concentration, 2 mL of solution was mixed with 2 mL of 40 ppm DPPH, incubated for 30 minutes in the dark, and measured at the maximum wavelength.

Determination of Antioxidant Activity of Extracts

Buas-buas leaf extracts obtained using NADES and honey solution as green solvents were evaluated for antioxidant activity. The NADES extract was tested at concentrations of 20, 30, 40, 50, and 60 ppm, whereas the honey-solvent extract was tested at concentrations of 20, 40, 60, 80, and 100 ppm. For each concentration, 2 mL of sample solution was mixed with 2 mL of 40 ppm DPPH solution, incubated in the dark for 30 minutes, and observed for a color change from purple to yellow, indicating free radical scavenging activity. Absorbance was measured at the maximum wavelength, and all measurements were performed in triplicate.

Formulation of Clay Mask Stick

The preparation of the clay mask stick began with weighing all ingredients according to the formulation. Bentonite was first dispersed with aquadest in a mortar and allowed to swell. Subsequently, kaolin was added and triturated until a smooth, lump-free paste was formed. Zinc oxide, methyl paraben, and glycerin were then incorporated and mixed thoroughly (Phase 1). In a separate process, cetyl alcohol, lanolin, isopropyl myristate, and carnauba wax were melted using a water bath at approximately 70°C until fully liquefied and well combined (Phase 2). The molten Phase 2 was then gradually added to Phase 1 under continuous stirring until a homogeneous mixture was obtained. Afterward, Buas-Buas Leaf Extract and menthol were added and mixed until evenly distributed. The final mixture was poured into molds and allowed to solidify into stick form at room temperature (Aan *et al.*, 2025). The resulting formulation was evaluated for organoleptic properties, homogeneity, pH, spreadability, adhesion, and drying time. Stability testing was performed using a cycling test method, involving alternating storage at low temperature ($\pm 4^{\circ}\text{C}$) and high temperature ($\pm 40^{\circ}\text{C}$). Antioxidant activity was also assessed using the same DPPH method as applied to the extract. Prior to stability testing (H0), concentrations of 50, 100, 150, 200, and 250 ppm were used. After six cycles of the cycling test, concentrations of 100, 200, 300, 400, and 500 ppm were applied. The increase in concentration after the cycling test was necessary because storage under alternating temperature conditions may cause degradation of antioxidant compounds, resulting in decreased antioxidant activity. Consequently, the lower concentration range used before storage produced inhibition values that were too low for accurate IC_{50} determination. Therefore, higher concentrations were used to obtain measurable inhibition responses and ensure reliable antioxidant activity analysis.

Experimental Design

This study employed a comparative experimental design to evaluate two types of green solvents, namely NADES and honey solution, for the extraction of buas-buas leaves. The antioxidant activity of the resulting extracts was determined using the DPPH method through IC_{50} value determination. The extract with the best antioxidant activity was subsequently formulated into a clay mask stick and evaluated for its physical characteristics, including organoleptic properties, homogeneity, pH, spreadability, adhesion, drying time, and stability using the cycling test method. All measurements were performed in triplicate.

Data Analysis

Data were analyzed using descriptive and quantitative approaches with SPSS software. IC_{50} values were determined through linear regression analysis between concentration and percentage inhibition. Stability data were analyzed based on changes in physical parameters before and after the cycling test, and statistical tests were conducted to determine significant differences.

RESULTS AND DISCUSSION

Phytochemical screening is a preliminary qualitative analysis used to identify the presence of secondary metabolites in an extract, providing an initial indication of its potential bioactivity (Aristyawan *et al.*, 2024). The results indicated that the Buas-Buas Leaf Extract Obtained Using NADES was positive for flavonoids, saponins, phenolics, and tannins,

whereas the Buas-Buas Leaf Extract Obtained Using Honey Solution did not exhibit the presence of tannins. Tannins are phenolic compounds characterized by relatively high molecular weight and specific polarity, which makes them more compatible with the NADES solvent system than with honey-based solvents. The presence of tannins in the NADES extract is expected to contribute to its higher antioxidant activity, as tannins function as effective electron donors and free radical scavengers (Adyitia *et al.*, 2017). The phytochemical screening results for each extract are presented in Table 1.

Table 1. Phytochemical Screening Results of *Premna serratifolia* L. Leaf Extract

Compound	Positive Indicator	NADES extract	Honey Extract
Flavonoids	Red to orange coloration	+	+
Phenolics	Formation of foam	+	+
Saponins	Blue, green, purple, or reddish coloration	+	+
Tannins	Dark green to blackish coloration	+	-

Positive (+) = Indicates the presence of secondary metabolites.

Negative (-) = Indicates the absence of secondary metabolites.

Antioxidant Activity of Extracts

Antioxidant activity reflects the ability of a compound to neutralize free radicals involved in oxidative stress through electron or hydrogen atom donation mechanisms (Meigaria *et al.*, 2018). In this study, the DPPH method was employed as it provides a quantitative evaluation based on the IC₅₀ value, defined as the concentration required to inhibit 50% of free radicals. Method validation was initiated by determining the maximum absorption wavelength (λ_{max}) of DPPH within the range of 400–800 nm, which showed a peak absorbance at 515 nm. This result is consistent with the theoretical maximum absorption of DPPH, typically reported within the range of 515–520 nm (Muliasari *et al.*, 2023). As illustrated in Table 2, vitamin C, used as a positive control, exhibited an IC₅₀ value of 4.056 ± 0.068 ppm. An IC₅₀ value below 50 ppm is classified as very strong antioxidant activity. In addition, the correlation coefficient ($r \geq 0.995$) indicated a very strong linear relationship between concentration and percentage inhibition, demonstrating good analytical accuracy and precision (ICH, 2005). The IC₅₀ values of vitamin C are presented in Table 2.

Table 2. Antioxidant Activity of Vitamin C

Replicate	IC ₅₀	mean IC ₅₀ $\pm$ SD	Category
1	3.992	4.056 $\pm$ 0.068	Very strong
2	4.124		
3	4.022		

The NADES Buas-Buas Leaf Extract L. leaves exhibited very strong antioxidant activity, as indicated by its lower IC₅₀ value, whereas the honey-based extract was categorized as having moderate activity (Tarung *et al.*, 2023). This indicates that the antioxidant activity of the Buas-buas Leaf Extract Obtained Using NADES is higher than that of the honey-based extract. The difference is influenced by the characteristics of the solvent used, where NADES has the ability to form a hydrogen-bonding network with tunable polarity, making it more effective in extracting bioactive compounds, particularly phenolics and flavonoids. In contrast, the honey-based solvent system at a ratio of 1:20 is

predominantly composed of distilled water, which is highly polar. This condition reduces the extraction efficiency for compounds with a wide range of polarities. Furthermore, the presence of sugars in honey, along with the high proportion of distilled water, may lead to a dilution effect, thereby lowering the concentration of active compounds in the extract and resulting in reduced antioxidant activity (Adyittia *et al.*, 2017). Flavonoid and phenolic compounds present in Buas-buas Leaf. leaves contribute to antioxidant activity through mechanisms involving hydrogen atom or electron donation to stabilize free radicals, as well as by inhibiting the formation of new radicals via metal ion chelation. Based on the IC₅₀ values, the NADES extract was selected for further formulation into a clay mask stick, as it is expected to provide more optimal antioxidant efficacy (Dimitriu et al., 2023). The IC₅₀ values of the NADES and honey extracts are presented in Table 3.

Table 3. Antioxidant Activity of Buas-Buas Leaf Extracts Using NADES and Honey Solution

Sample	Replicate	IC ₅₀	mean IC ₅₀ ±SD	Category
NADES	1	31.209	31.53 ± 0.1611	Very strong
Based	2	31.531		
Extract	3	31.376		
Honey	1	109.11	108.53 ± 0.693	Moderate
Based	2	108.70		
Extract	3	107.76		

The formulation developed in this study was a clay mask stick incorporating Buas-Buas Leaf Extract Obtained Using NADES. leaves as the active ingredient. The selection of the NADES extract was based on its high antioxidant activity (low IC₅₀ value), which is expected to provide protective effects on the skin. The mask base consisted of a combination of kaolin and bentonite, which function as adsorbents to remove impurities and excess sebum. Carnauba wax and cetyl alcohol were utilized to form a solid stick dosage form that is practical and easy to apply. Carnauba wax was selected due to its high melting point (approximately 82–86°C), which contributes to the hardness and structural stability of the formulation. Meanwhile, cetyl alcohol (approximately 49–52°C) acts as a co-emollient, improving consistency and enhancing the smoothness of application. The combination of these components provides a balance between hardness and spreadability, ensuring that the formulation remains stable, does not easily melt, and is comfortable to use without being excessively rigid. As shown in Figure 1, the formulations in this study consisted of several variations in active ingredient concentration, namely F0 (without extract), F1 (1%), F2 (3%), and F3 (5%) of Buas-Buas Leaf Extract Obtained Using NADES. leaves, in order to evaluate the effect of concentration on the characteristics and antioxidant activity of the formulation (Aan *et al.*, 2025).



Figure 1. Clay Mask Stick Formulation

Organoleptic evaluation showed that all formulations (F1–F3) remained stable throughout six storage cycles, with no observable changes in odor and texture. The aroma was predominantly mint, with a slight characteristic odor of the extract observed in F3, while all formulations maintained a consistent and stable semi-solid texture. Differences were observed only in color, where increasing extract concentration resulted in a progressively darker appearance. The stability of the texture is attributed to the presence of carnauba wax and cetyl alcohol, which function as solid matrix-forming agents, while kaolin and bentonite act as adsorbents. The higher proportion of kaolin (15%) compared to bentonite (1%) suggests that the formulation is more suitable for normal to dry skin types. Homogeneity testing indicated that all formulations remained uniform during storage, as evidenced by the absence of lumps or phase separation, confirming an even distribution of active ingredients and excipients as well as a stable formulation system (Darmadinata & Sulistyaningsih, 2019).

pH evaluation was performed to ensure the safety of the clay mask stick for topical application. The initial pH ranged from 5.12 to 5.41, which falls within the acceptable range (4.5–8) and is close to the physiological pH of the skin. During the cycling test, a slight increase in pH was observed; however, the values remained stable and within the safe range (Fitria *et al.*, 2025). This change may be attributed to the presence of flavonoid compounds, which exhibit weak acidic properties. Statistical analysis revealed significant differences between cycles and formulations ($p < 0.05$), although these variations remain practically acceptable. As a topical formulation acting primarily on the epidermal layer, the product is considered safe with minimal risk of excessive penetration (Sawij & La, 2019). The pH values during storage are presented in Table 4.

Table 4. pH Values of Clay Mask Stick During 6 Storage Cycles

Formulation	Cycle					
	1	2	3	4	5	6
F1	5.64	6.06	6.22	6.34	6.52	7.01
F2	5.53	5.82	6.05	6.19	5.97	6.38
F3	5.50	5.71	5.90	6.02	6.12	6.21

Spreadability testing was conducted to evaluate the ability of the formulation to spread on the skin surface during application. At the initial observation (day 0), spreadability values were 4.65 cm (F1), 4.70 cm (F2), and 4.80 cm (F3). During the cycling test, all formulations met the acceptable standard range (3–5 cm), although a gradual decrease was observed over time. This reduction is associated with increased viscosity due to the swelling

properties of bentonite and kaolin, as well as solvent evaporation during storage. Statistical analysis indicated significant differences between cycles and formulations ($p < 0.05$), suggesting that composition influences spreadability. Overall, all formulations demonstrated good spreadability, indicating ease of application and uniform distribution on the skin (Fiskia *et al.*, 2024). Results are shown in Table 5.

Tabel 5. Spreadability of Clay Mask Stick During 6 Storage Cycles

Formulation	Cycle					
	1	2	1	4	1	6
F1	4.430	4.467	4.253	3.763	3.387	3.353
F2	4.633	4.510	4.297	3.797	3.650	3.500
F3	4.663	4.520	4.280	4.073	3.830	3.697

Adhesion testing was performed to determine the duration of adherence of the formulation on the skin after application. Initial adhesion times were 35.12 s (F1), 33.23 s (F2), and 28.01 s (F3). All formulations exhibited good adhesion (>4 seconds), with values ranging from 31–60 seconds (Qoriati *et al.*, 2024). Adhesion increased during storage, likely due to increased viscosity and the formation of a colloidal structure by bentonite and kaolin. Statistical analysis showed significant differences ($p < 0.05$). These results indicate that all formulations possessed adequate adhesive properties, although a decreasing trend was observed with increasing extract concentration (Fiskia *et al.*, 2024). Results are presented in Table 6.

Table 6. Adhesion of Clay Mask Stick During 6 Storage Cycles

Formulation	Cycle					
	1	2	1	4	1	6
F1	38.70	41.62	44.98	49.88	54.25	60.03
F2	36.01	38.97	41.90	46.16	53.16	57.53
F3	30.08	33.11	38.20	42.71	44.56	48.36

Drying time testing was conducted to determine the time required for the formulation to dry on the skin surface after application. Initial drying times were 27.01 minutes (F1), 26.11 minutes (F2), and 25.24 minutes (F3). During the cycling test, all formulations remained within the ideal range (15–30 minutes) and tended to decrease over time. This trend is influenced by solvent evaporation and increased solid content due to temperature fluctuations. Statistical analysis showed significant differences ($p < 0.05$) (Zehan *et al.*, 2024). Results are presented in Table 7.

Table 7. Drying Time of Clay Mask Stick During 6 Storage Cycles

Formulation	Cycle					
	1	2	1	4	1	6
F1	24.90	23.22	20.65	18.87	17.17	15.71
F2	26.16	25.05	22.14	20.60	19.70	17.03
F3	25.62	23.27	22.56	18.54	18.20	15.09

Antioxidant Activity of Clay Mask Stick

The antioxidant activity of the clay mask stick showed that, at day 0, activity increased with increasing extract concentration. Formulations F1 and F2 were categorized as weak, whereas F3 (5%) exhibited the highest activity, classified as moderate ($IC_{50} = 110.88$ ppm). The negative control (F0) showed negligible activity ($IC_{50} = 460.21$ ppm), confirming that antioxidant activity is directly proportional to extract concentration. After six cycles of stability testing, all formulations exhibited increased IC_{50} values, indicating a decline in antioxidant activity. F1 and F2 shifted to very weak or inactive categories, while F3 remained in the weak category, indicating relatively better performance compared to other formulations. The observed reduction in antioxidant activity is primarily attributed to the oxidative degradation of flavonoid and phenolic compounds, which are the major antioxidant constituents of *buas-buas* leaf extract. Repeated temperature fluctuations during the cycling test can accelerate oxidation reactions, resulting in the degradation of hydroxyl-containing phenolic structures that are responsible for free radical scavenging activity. Consequently, the ability of the formulation to neutralize DPPH radicals decreases, leading to an increase in IC_{50} values after storage. Therefore, oxidative degradation of flavonoid and phenolic compounds is considered the predominant mechanism responsible for the decline in antioxidant activity observed in this study. These factors contribute to reduced antioxidant effectiveness and highlight the importance of storage conditions (Nugroho & Wahidin, 2024).The results are presented in Table 8.

Table 8. IC_{50} Values of Clay Mask Stick Formulations (Day 0 and Cycle 6)

Sample	Formulation	IC_{50} (ppm)	Category
Formulation at Day 0	F1	207.94	Weak
	F2	171.57	Moderate
	F3	110.88	Moderate
Formulation at Cycle 6	F1	434.28	Inactive
	F2	312.55	Very Weak
	F3	208.48	Weak

CONCLUSION

The extract of *Premna serratifolia* L. leaves obtained using NADES (citric acid–glucose) exhibited very strong antioxidant activity ($IC_{50} = 31.53$ ppm), which was superior to that obtained using honey-based solvent ($IC_{50} = 108.53$ ppm, moderate category). In the clay mask stick formulation, F3 (5%) demonstrated the highest antioxidant activity; however, a decrease was observed after the cycling test, as indicated by an increase in IC_{50} from 110.88 ppm to 208.48 ppm. Overall, the formulation exhibited good physical stability throughout the evaluation, including organoleptic properties, pH, spreadability, adhesion, and drying time, despite statistically significant differences that remained within acceptable limits. The use of green solvents was shown to enhance antioxidant activity by improving the extraction efficiency of phenolic and flavonoid compounds, as reflected by lower IC_{50} values.

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