Ad-Dawaa' Journal of Pharmaceutical Sciences
ISSN: 2654-7392, E-ISSN: 2654-6973
Vol. 9, No. 1, June 2026, p. 93 – 104
DOI: https://doi.org/10.24252/djps.v9i1.59586

Innovation of Microparticle Peel-Off Gel Mask Formulation from Freeze-Dried Kratom Leaf Extract of Kalimantan as an Antioxidant

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Article history:
Submited: 1-2-2026
Revised: 28-2-2026
Accepted: 15-3-2026

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Cite this article: Latifah, N., Fatmasari, E., Purwanto, A., Zamzani, I., Nor, I., Indira, I., Aida, N. (2026). Innovation of Microparticle Peel-Off Gel Mask Formulation from Freeze-Dried Kratom Leaf Extract of Kalimantan as an Antioxidant.. Ad-Dawaa' J. Pharm. Sci. 9(1): 93-104.

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ABSTRACT

Introduction*: Indonesia is rich in natural resources, including various plants with potential as traditional medicinal ingredients. One of these plants is kratom (Mitragyna speciosa), which contains bioactive compounds such as alkaloids and flavonoids, both of which are rich in antioxidants.* ***Aims:*** *The purpose of this study was to determine whether the freeze-dried methanol extract of kratom leaves can be formulated into microparticles, and to then prepare a microparticle peel-off gel mask from the freeze-dried methanol extract of kratom leaves to produce a high-quality preparation.* ***Result:*** *The research method included extraction using maceration, followed by formulating the methanol extract of kratom leaves into microparticles using the freeze-drying method. This was done to enhance the stability of the active compounds and ensure the controlled release of the active ingredients. The results of the basic formula for the peel-off gel mask were adapted from previous research that developed an effective peel-off gel mask base. Microparticles (1,290 µm) of the freeze-dried kratom leaf extract were then added, and physical quality evaluations showed that only Formula 3, with a Polyvinyl Alcohol (PVA) concentration of 16%, met all the physical quality test requirements for the peel-off gel mask preparation.* ***Conclusion:*** *The conclusion of this study is that the freeze-dried methanol extract of kratom leaves can be formulated into microparticles, and the preparation of the microparticle peel-off gel mask from the freeze-dried methanol extract of kratom leaves results in a high-quality preparation in Formula 3.*

KEYWORDS*: Kratom leaves, freeze-dried, microparticles, peel-off gel mask, gel.*

INTRODUCTION

Indonesia is rich in natural resources, including indigenous plants often used as food ingredients and traditional medicines. The use of traditional plant-based medicines continues to increase, making it crucial to develop more practical and consumer-friendly preparations (Mariani, 2021). One region rich in natural resources is Kalimantan, which boasts numerous plants with potential as herbal medicines. Some plants with proven efficacy include tongkat ali (early earth stake), tabat barito (barito tabat), dayak onion, sarang

semut (ant nest), maniran (yellow wood), kratom, and karamunting (Sutomo, 2010).

Kratom (*Mitragyna speciosa*) is a tropical plant native to Southeast Asia, including Indonesia, particularly West Kalimantan. Kratom leaves contain active compounds such as alkaloids and flavonoids, which are known to have various benefits, including antitussive, anesthetic, antinociceptive, stimulant, analgesic, and antioxidant and antibacterial activities (Syafriansyah, et al., 2021; Parthasarathy, et al., 2009). Previous research has shown that methanol extract of kratom leaves has very strong antioxidant activity, with an IC_{50} value at a concentration of 37.08 ± 3.54 µg/mL, indicating its great potential for use as a topical agent to prevent premature aging caused by ultraviolet (UV) exposure (Parthasarathy et al. 2009).

One effective topical formulation for kratom extract is a peel-off gel mask. This gel mask works by drying after application to the skin and can then be peeled off, removing dead skin cells and accumulated surface debris such as excess sebum and environmental pollutants (Santoso et al. 2020). However, to ensure the stability of the active compounds, advanced technologies such as freeze-drying are necessary. This method maintains the stability of active compounds that are easily degraded by heat, thereby preserving the quality of the kratom extract used in topical preparations (Santi, 2015).

To produce a peel-off gel mask preparation with freeze-dried kratom leaf extract containing powerful antioxidants and microparticle size, a thorough evaluation of the preparation's physical quality is necessary. Previously, the authors developed a natural peel-off gel mask base that met physical parameters such as homogeneity, viscosity, pH, spreadability, and drying time (Latifah et al., 2025). Based on these results, this research continued with the innovation of adding active ingredient extracts in microparticle form to increase the effectiveness and stability of the antioxidant. Because the stability of antioxidants from kratom leaves is highly influenced by the environment, a microparticle formulation is considered an appropriate approach (Aulia & Sriwidodo, 2022).

Furthermore, to improve the stability and control of active ingredient release, a microparticle delivery system is an appropriate choice. Microparticles measuring 1–1000 µm can control the gradual release of active ingredients, increasing the effectiveness and stability of topical preparations (Aulia & Sriwidodo, 2022). Although nanoparticles are widely explored in topical delivery, this study employed a microparticle system to ensure greater physical stability within the gel matrix and to minimize excessive dermal penetration. In peel-off cosmetic formulations, the primary objective is controlled surface release and localized antioxidant activity rather than systemic absorption. Therefore, microparticles were considered more appropriate to balance stability, safety, and functional performance (Silva et al. 2023). So that, this study aims to

Table 1. Microparticles formulation of freeze-dry kratom leaves extract

Material	Amount	Solvent
Freeze-dried kratom leaf extract	3.7 mg	2 mL
Chitosan	0.02 g	
Acetic acid	0.05 g	10 mL
Tween 80	0.01 g	
Sodium tripolyphosphate (NaTPP)	2 mg	2 mL
Distilled water (aquadest)	14 mL	-

develop a peel-off gel mask preparation based on freeze-dried kratom leaf extract microparticles, which is expected to increase antioxidant effectiveness and provide optimal cosmetic benefits.

The purpose of this study was to determine whether freeze-dried methanol extract of kratom leaves can be formulated into microparticles and to determine whether the freeze-dried methanol extract of kratom leaves produces a good peel-off gel mask preparation.

MATERIAL AND METHODS

Equipment

The following equipment was used in this study: Homogenizer (IKA), Ultraturax (IKA T25), Dehydrator (Memmert), Oven (Memmert), pH Meter (Hanna Instruments), Viscometer (Brookfield), Spectrophotometer (Shimadzu), Magnetic Stirrer (IKA), and Spreadability Test Equipment.

Materials

Kratom leaves, methanol, ethanol, Polyvinyl Alcohol (PVA), Hydroxypropyl Methylcellulose (HPMC), propylene glycol, methyl paraben, fragrance, aquadest, chitosan, Sodium tripolyphosphate (NaTPP), glacial acetic acid.



Figure 1. Kratom leaves

Preparation of Simplisia

A total of 10 kg of fresh kratom leaves were dried at a temperature of 16-18°C. The dried simplisia was powdered using a blender until a fine simplisia powder was obtained, which was then ready for maceration.

Maceration Extraction Method

A total of 1,200 grams of dried simplicia powder kratom leaves are coated with methanol as much as 12 L at room temperature for 3 days. Maserat is then concentrated with a water bath until a thick extract is obtained. The concentrated extract was subsequently subjected to freeze-drying at −40°C and then formulated into a microparticle-based peel-off gel.

Table 2. Formulation of Microparticle Peel-Off Gel Mask from Freeze-Dried

Material	Formulation (g)		
	F1	F2	F3
Freeze-dried kratom leaf extract microparticles	0.0037	0.0037	0.0037
Polyvinyl Alcohol (PVA)	12	14	16
Hydroxypropyl Methylcellulose (HPMC)	3	3	3
Propylene Glycol	10	10	10
Methyl Paraben	0.2	0.2	0.2
Fragrance	qs	qs	qs
Etanol	2	2	2
Distilled Water (aquadest) ad	100	100	100

Microparticles Freeze-Dry Extract Kratom Leaves

Freeze-dry extract from kratom leaves is weighed and dissolved into ethanol, then added aquades. Chitosan with various variations of weight is dissolved into 1%glacial acetic acid. Variations in composition comparison of extracts, chitosan, and NATPP in the solution, namely in the ratio (1:1:1), (1:5:1), and (1:10:1). After the material is mixed, the stirring is continued using a magnetic stirrer for 2 hours, then evaporation is carried out using the Solvent Evaporation method (Fitri, 2020).

Characterization of Microparticles Freeze-Dry Extract

Evaluation of Particle Size

Particle size distribution was analyzed using a laser diffraction Particle Size Analyzer (Beckman Coulter LS 13 320, USA) equipped with a Universal Liquid Module. The measurement was performed using the Fraunhofer optical model over a detection range of 0.017–2000 μm. The sample was dispersed in distilled water (H_2O) and measured under continuous stirring to ensure homogeneity. The results were expressed as mean particle size and distribution parameters (d10, d50, and d90).

Evaluation of SEM

The microparticles obtained are characterized using SEM (scanning electron miscroscopy). Arrange the lens and pay attention to the monitor until the sample looks focused, then enlarged and clarified the image. This SEM is used to see the morphology of microcapsules (Mahl, et al. 2011).

Formulation of Microparticle Peel-Off Gel Mask

The basic formula of the peel-off gel mask in this study refers to the composition that was developed and evaluated in previous research (Latifah et al., 2025), with the addition of an active phase in the form of microparticles from freeze-dried kratom leaf extract from Kalimantan.

Preparation of Microparticle Peel-Off Gel Mask from Freeze-Dried Kratom Leaf Extract

The process of making the microparticle peel-off gel mask from freeze-dried kratom leaf extract begins by dissolving methyl paraben in a glass beaker with aquadest at a temperature of 90°C. After that, HPMC is added and left to expand completely. Separately, PVA is developed in hot aquadest at a temperature of 90°C until it fully expands, then stirred. The PVA and HPMC are then combined with constant stirring until they swell (first mixture).

The freeze-dried kratom extract microparticles were dispersed in ethanol and stirred using a magnetic stirrer at controlled speed to obtain a homogeneous suspension while preserving particle structure. After mixing, propylene glycol is added, and the mixture is stirred again until homogeneous (second mixture). The second mixture is then added to the first mixture, a few drops of fragrance are added, and it is stirred until homogeneous. Store the mixture in a container and seal it tightly. Finally, perform a physical quality evaluation of the gel mask preparation.

Physical Evaluation of Microparticle Peel-Off Gel Mask from Freeze-Dried Kratom Leaf Extract

The physical quality evaluation of the microparticle peel-off gel mask from freeze-dried kratom leaf extract refers to the study by Harliantika and Noval (2021) and includes organoleptic, homogeneity, pH, viscosity, adhesion, Spreadability , and drying time tests (3x replication).

Organoleptic test. Observation of color, form, and aroma of the preparation.

Homogeneity test. This is done by applying the gel to three parts: the top, middle, and bottom. The peel-off gel mask preparation is placed on a transparent glass, then covered with a glass slide to observe the clarity and the presence of aggregates in the gel mask preparation (Latifah, Patimah and Kurnia, 2025).

pH test. Measured by inserting a pH meter into the test sample. The pH meter used in this study had been calibrated with a standard solution. The required pH range is 4.5–6.5 (Latifah et al. 2025).

Spreadability test. The spreadability test is conducted by taking 0.5 grams of the peel-off gel mask and placing it on a glass with a 15 cm diameter. The top part of the glass is weighed and then placed on top of the sample (Leave it for 1 minute). After that, apply a 50-gram load, then let it sit for 1 minute. The spreadability is recorded each time the load is added. The total load added is 100 grams, and the required spreadability is 3-5 cm (Saputri et al. 2020).

Adhesion test: This is done by taking 0.2 g of the peel-off gel mask and placing it between two glass slides. A 1 kg weight is placed on top of the glass slide and left for 5 minutes. After that, the weight is removed, and the time it takes for the two glass slides to separate is recorded. The required adhesion time is no less than 4 seconds (Hidayat et al. 2021).

Viscosity test. The Stormer viscometer is used with spindle number 4 to determine the viscosity of each formula. The test requirement is 4,000 – 50,000 cps (Laila et al. 2022)

Drying time. The test is carried out by applying 0.5 grams of each formula preparation to the back of the hand with a size of 4 × 4 cm, then observed using a stopwatch. The time required for the preparation to dry is the time until the preparation forms a film layer (Maharani et al. 2019). This study protocol involving human subjects was approved by the Institutional Ethics Committee of Universitas Muhammadiyah Banjarmasin, with ethical clearance number No. 490/UMB/KE/VIII/2024.

RESULTS AND DISCUSSION

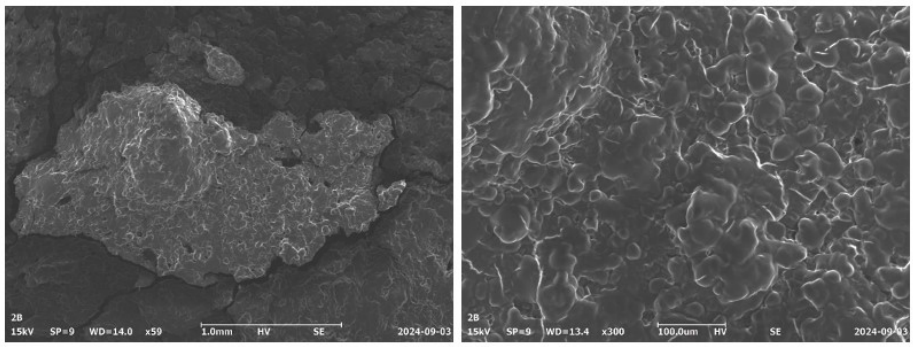
Extraction is a crucial step in isolating bioactive compounds from natural materials, as it determines the phytochemical composition that will be obtained. In this study, kratom leaf extraction was carried out using methanol as a solvent. Methanol is known as a strong polar solvent that is effective in extracting phenolic compounds, flavonoids, and alkaloids—secondary metabolites with high antioxidant activity.

According to Rahman et al. (2022), the use of methanol as a solvent result in higher extract yields compared to ethanol or water, particularly for plants that contain aromatic and nitrogen compounds, such as alkaloids. This is consistent with the characteristics of kratom leaves (*Mitragyna speciosa*), which contain mitragynine, flavonoids, and other phenolic compounds. Additionally, methanol has a high solubility for semi-polar to polar components, which is believed to play a significant role in antioxidant activity.

However, the main drawback of using methanol is its toxicity and potential carcinogenicity if residues remain in the preparation. Therefore, in this study, a subsequent process of freeze-drying was conducted. Freeze-drying not only efficiently removes the solvent but also preserves the stability of active compounds that are easily degraded by heat (Bhardwaj et al., 2021). This process ensures the safety of the extract for further use in topical cosmetic formulations.

Dried kratom leaves were powdered and extracted by maceration using methanol (1:10 w/v) for 3x24 hours at room temperature with occasional stirring. The filtrate was collected and concentrated under reduced pressure using a rotary evaporator at 40°C to obtain a thick extract. The concentrated extract was then subjected to freeze-drying at −40°C to produce a dry extract powder. The extraction yield was calculated based on the initial weight of dried plant material and the final weight of dried extract. The percentage yield obtained was 11.21% (w/w).

After extraction with methanol solvent, the freeze-drying stage is carried out to produce a dry extract that is stable and safe for use. Freeze-drying effectively removes harmful solvents such as methanol and preserves the



(a) (b)

Figure 2. Scanning electron microscope microparticle kratom leaf extract with magnification (a) ×59 and (b) ×300

chemical structure of active compounds, especially flavonoids and phenolics, which are susceptible to degradation due to heat (Kumar et al., 2021). The main advantage of this method is the sublimation process, which operates at low temperatures and under vacuum pressure, making it suitable for preserving the bioactivity of antioxidant compounds. Several studies have shown that freeze-dried plant extracts have higher total phenol content and antioxidant activity compared to conventional drying methods (Kim et al., 2023). This supports the use of freeze-drying in the initial process of formulating natural-based cosmetics.

To further enhance the stability of the extract and prolong the release of active ingredients on the skin, the freeze-dried extract is formulated into microparticles. Microparticles function as a delivery system that can control the gradual release of bioactive compounds, enhance permeation, and protect the compounds from oxidative degradation and photolysis (Sah & Prabaharan, 2020).

The particle size analyzer test results that the microparticles of the freeze-dried kratom leaf extract have an average (mean) size of 1,290 µm with a median (d50) of 1,008 µm. The d10 and d90 values are 0,604 µm and 2,176 µm; respectively, indicating a particle distribution that falls within the ideal range for topical applications. The distribution is bimodal, with a C.V. value of 49.3%, indicating variation in particle sizes. Particles smaller than 10 µm are considered suitable for the peel-off gel mask as they can spread evenly on the skin and allow for the gradual release of active ingredients (Chen et al., 2022; Alshammari et al., 2023).

The SEM results in the Figure 2 at magnifications of ×59 and ×300 show that the microparticles of the freeze-dried kratom leaf extract have a relatively uneven and indented surface, with a morphology that tends to be irregular. However, the particles are well-dispersed. This wrinkled surface can increase the contact surface area and support the gradual release of active compounds when applied to the skin.

The particle structure appears to be partially adhered to each other but does not form large aggregates, indicating that the drying system

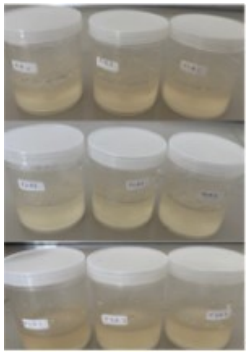


Figure 3. Formula preparations of the peel-off gel mask

successfully produced particles in the appropriate micro-size range for topical preparations. This morphology is consistent with the PSA results, which show particle sizes of <10 µm. According to Sah & Prabaharan (2020), microparticle morphology that is not overly smooth and has light porosity can enhance adhesion and the release of active ingredients on the skin's surface.

The homogeneity test in the Figure 3 results show that all formula preparations of the peel-off gel mask have a uniform microparticle distribution, without any sedimentation or clumping after storage. This indicates that the microparticles of freeze-dried kratom leaf extract are well-dispersed in the gel matrix. The success of this homogeneity test demonstrates that the formula used is effective in maintaining the uniformity of the preparation, ensuring a consistent dose of antioxidant active ingredients in each application. These results are consistent with findings from previous studies by Afifah et al. (2020) and Latifah et al. (2025), who also reported good homogeneity in gel bases with evenly distributed active ingredients.

The homogeneity test in the figure 5 results show that all formula preparations of the peel-off gel mask have a uniform microparticle distribution, without any sedimentation or clumping after storage. This indicates that the microparticles of freeze-dried kratom leaf extract are well-dispersed in the gel matrix. The success of this homogeneity test demonstrates that the formula used is effective in maintaining the uniformity of the preparation, ensuring a consistent dose of antioxidant active ingredients in each application. These results are consistent with findings from previous studies by Afifah et al. (2020) and Latifah et al. (2025), who also reported good homogeneity in gel bases with evenly distributed active ingredients.

The pH test results (Table 3) show that all formulas have a pH within the safe range for the skin, which is between 4.5–6.5. Formula 2 has the highest pH of 4.90 ± 0.05, followed by Formula 1 (4.79 ± 0.04) and Formula 3 (4.72 ± 0.05). Statistical analysis shows a significant difference between the formulas ($p<0.05$). This interpretation indicates that the addition of microparticles from freeze-dried kratom leaf extract can influence the final pH of the preparation. The pH variation is suspected to stem from the acidic or basic nature of the metabolite compounds in the extract, which interact with the gel base, such as PVA or other compounds. These findings are consistent with the research of Wahyuni et al. (2023), which states that the addition of plant extracts

Tabel 3. Phisical evaluation of microparticle peel-off gel mask

Physical evaluation	Formula 1	Formula 2	Formula 3
pH	4.79±0.04*	4.90±0.05*	4.72±0.05*
Adhesion (minutes)	1.71±0.60*	2.76±0.57*	4.74±1.95*
Spreadability (cm)	4.8±0.529*	4.37±0.231*	3.63±0.45*
Viscosity	1.411±1.08ns	2.831±0.85ns	4.451±2.12ns
Drying time	18.67±1.53*	19.67±2.31*	17.33±4.16*

n = 3. * mean significantly different (<0.05), ns mean no significant different.

.containing phenolic compounds and alkaloids can lower the pH of the formulation due to the acidic nature of these active compounds. Formulations with a pH close to that of the scalp are considered more suitable for topical use to minimize the risk of irritation.

The adhesion test results show the highest value in Formula 3 (4.74 ± 1.95 minutes), followed by Formula 2 (2.76 ± 0.57 minutes), and the lowest in Formula 1 (1.71 ± 0.60 minutes). Statistical analysis shows a significant difference between the formulas ($p < 0.05$). The interpretation of these results indicates that the concentration of microparticles in the extract is directly proportional to the increased adhesion time. This phenomenon may be attributed to interactions between the microparticles and the polymeric gel matrix, leading to increased viscosity and improved adhesive properties. Research by Hidayat et al. (2021) supports these findings, suggesting that higher local viscosity or the amount of active compounds that can interact with the gel matrix will enhance the adhesion of the peel-off preparation, thereby extending the contact time on the skin and potentially improving efficacy.

Formula 1 has the highest spreadability (4.8 ± 0.529 cm), followed by Formula 2 (4.37 ± 0.231 cm), and Formula 3 (3.63 ± 0.45 cm). There is a significant difference between the formulas ($p < 0.05$). The interpretation of these results indicates that an increase in microparticle concentration leads to a decrease in spreadability. This can be attributed to the increased local viscosity and the reduced ability of the preparation to spread across the skin surface. These findings align with research by Saputri et al. (2020) and Lestari et al. (2020), which state that solid particles in the gel base can form a denser structure and hinder water diffusion, thereby reducing the spreadability of the preparation.

The viscosity test results show that Formula 3 has the highest viscosity (4,451 ± 2.12 cps), followed by Formula 2 (2,831 ± 0.85 cps), and Formula 1 (1,411 ± 1.08 cps). However, statistical analysis shows that there is no significant difference between the formulas ($p > 0.05$), and all are still below the standard viscosity range for gel preparations (4,000–50,000 cps). The interpretation of these results indicates that the microparticles of kratom leaf extract have the potential to increase viscosity, but it is not yet sufficient to produce a statistically significant difference. The low

viscosity may indicate that the polymer concentration or cross-linking is not yet optimal. According to Laila et al. (2022), it is stated that the combination of PVA and active compounds can synergistically increase viscosity if there is sufficient ionic or hydrophobic interaction. The lack of significance in this study could be due to the formula variations not being extreme enough.

The drying time test results show that Formula 2 has the longest drying time (19.67 ± 2.31 minutes), followed by Formula 1 (18.67 ± 1.53 minutes), and Formula 3 (17.33 ± 4.16 minutes). There is no significant difference between the formulas ($p>0.05$). This interpretation indicates that the addition of microparticles does not significantly affect the drying time. The drying times, ranging from 17 to 20 minutes, are still considered suitable for peel-off preparations and not too long for practical use. A study by Maharani et al. (2019) shows that the drying time of gel masks is highly influenced by environmental humidity, water content in the formula, and the type of active ingredients. Since the formulas used have the same base and solvent content, the drying time results are relatively homogeneous.

CONCLUSION

In conclusion, the freeze-dried methanol extract of kratom leaves was successfully formulated into microparticles and incorporated into a peel-off gel mask system. Among the tested formulations, Formula 3 demonstrated the most favorable physicochemical characteristics, particularly in terms of pH stability, viscosity, and drying time.

ACKNOWLEDGEMENT

The author expresses the highest appreciation to the Higher Education Research and Development Council (DIKTILITBANG) of the Muhammadiyah Central Leadership for their funding support through the RisetMu Grant Program (National Competitive Research of Muhammadiyah 'Aisyiyah Universities) Batch VIII in 2024.

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