

Improving Production Strategies through Monitoring Visual and Physicochemical Changes in Gambier Catechin-Based Shampoo Bars under Practical Storage Conditions

Rheinia Valeni¹, Rini¹, Ridwan Rizkyanto¹, Adila Adisti², Najla Rosyadah¹, Diah Ayu Natasya¹, Tuty Anggraini¹, Dina Afifah Zakia¹, Iin Hartati Wibisono³, Daimon Syukri^{1*}

¹Department of Food and Agricultural Product Technology, Universitas Andalas, Padang, Indonesia

²Department Economics Faculty Economics and Business, Universitas Andalas, Padang, Indonesia

³IUMI Organic Skin Care, Cibubur, Indonesia

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Corresponding Author:

Daimon Syukri

dsyukri@ae.unand.ac.id

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Abstract: The development of eco-friendly cosmetics using local bioresources like *Uncaria gambir* Roxb. (gambir) holds strong promise for sustainable innovation. This study formulates and evaluates a catechin-based solid shampoo enriched with purified gambir extract, aiming to enhance product stability. A 14-day storage test was conducted at 20 °C and 40 °C using three types of packaging: translucent plastic, aluminum foil, and amber glass. Visual observations revealed that higher temperatures, especially in translucent plastic, accelerated color degradation due to catechin instability. Amber glass was the most effective in preserving the shampoo's original color. Further, aqueous stability was assessed through two wet-use simulations: soaking (full immersion in distilled water) and dipping (partial immersion daily over four days). The shampoo maintained physical integrity during both tests, showing no softening or structural deformation. However, water discoloration increased over time, indicating catechin release and oxidation. The extent of browning was influenced by the pH of the water, suggesting that both environmental pH and oxidative exposure affect catechin behavior during use. These results demonstrate the solid shampoo's physical resilience and moderate color stability under thermal and aqueous stress. While catechin oxidation is unavoidable, its rate can be mitigated through optimal packaging and pH management. The gambir-based solid shampoo shows potential as a natural, environmentally friendly personal care product, although further formulation refinement is needed to improve color retention during storage and use.

Keywords: Application; Catechin; Field; Packaging; Stability

Introduction

Introduction The global cosmetics and personal care industry has experienced rapid growth, driven by increasing consumer awareness of health, sustainability, and a preference for natural-based products. Modern consumers are becoming more selective, seeking safe, eco-friendly formulations with proven functional benefits. This shift presents an opportunity for countries

rich in biodiversity, such as Indonesia, to develop high-value innovations based on local natural resources (Atmaja et al., 2025; Gunawan et al., 2024). One promising Indonesian commodity is *Uncaria gambir* Roxb., commonly known as gambir, a plant predominantly cultivated in West Sumatra and contributing up to 90 % of the nation's gambir exports. Gambir is well known for its high catechin content, a natural compound with strong antioxidant,

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antimicrobial, and antifungal properties (Bae et al., 2020; Gopal et al., 2016). In addition to catechin, gambir also contains tannins, pyrocatechol, and quercetin, which further enhance its functional potential (Mita et al., 2025; Munggari et al., 2022). These bioactive components make gambir extract highly suitable for use in cosmetic products, particularly natural hair care solutions (Goyal et al., 2022; Rónavári et al., 2021). In recent years, solid shampoo formulated with gambir extract has emerged as an eco-friendly and effective alternative to conventional liquid shampoos. Its compact form, minimal packaging, and plant-based ingredients align well with global sustainability trends. Previous product development efforts have successfully produced prototypes of gambir-based personal care items, including antiseptic liquid soap, bar soap, and solid shampoo, with promising results (Couceiro et al., 2025; Sayeed & Goyal, 2025).

Among them, the gambir-based solid shampoo has shown particular potential as a natural anti-dandruff treatment. Early trials have demonstrated its functional effectiveness and consumer interest, especially through limited production and marketing collaboration with local cosmetic producers such as CV IUMI Organic. Despite its promising qualities, one of the major obstacles to broader commercialization is the product's color instability during storage. The shampoo tends to turn brown over time, a change that negatively affects consumer perception and reduces its market appeal. This discoloration is suspected to result from the degradation of catechins, especially under suboptimal environmental conditions. To support the wider commercialization of gambir-based solid shampoo, this study aims to provide scientific insights into one of the key challenges: maintaining product stability, particularly its color, during storage and daily use. Discoloration over time can undermine product quality and reduce consumer acceptance. This research explores how the shampoo's appearance is affected by different storage conditions and packaging methods, such as whether the product is fully sealed, partially wrapped, or left unpackaged.

Simulated usage tests were also conducted to observe how the product responds to contact with water, mimicking real-life conditions. In addition, the study investigates how catechin—the main active compound in gambir—behaves under varying environmental factors, including temperature and pH, to better understand its contribution to color changes (Wen et al., 2025; Xue et al., 2024). These findings are expected to guide improvements in both formulation and packaging strategies to preserve the product's quality over time. By strengthening the scientific foundation for product stability, this research supports the development of

gambir-based solid shampoo that is more appealing, reliable, and ready for broader distribution in the growing natural cosmetics market.

Method

Materials

The equipment utilized in this study included an incubator, a HunterLab ColorFlex EZ spectrophotometer, beakers, and test tubes. The primary materials comprised catechin-based solid shampoo bars obtained directly from CV IUMI (Bogor, Indonesia), demineralized water (aquadest), sodium hydroxide (NaOH), and pure catechin powder.

Methods

This study aimed to evaluate the visual and physical stability of catechin-enriched solid shampoo bars under varying storage conditions. The shampoo bars were subjected to color stability testing at controlled temperatures of 20 °C, 30 °C, and 40 °C. Three packaging treatments were applied, namely: Sample Code 1, which consisted of shampoo bars with complete packaging (aluminum tube, plastic wrap, and silica gel); Sample Code 2, shampoo bars wrapped only in plastic; and Sample Code 3, shampoo bars without any packaging. In addition to storage-based stability testing, supplementary tests were conducted to simulate real-world use and examine the behavior of catechin under different conditions. These included: Soak Test (Leachate simulation) Dip Test (Repeated exposure to water) Catechin Solution Stability Test under neutral and alkaline conditions Color changes in all tests were monitored using a HunterLab ColorFlex EZ spectrophotometer.

Test Parameters and Procedures

Color Stability Test (Storage Conditions)

Shampoo bars under three different packaging conditions (fully packaged, plastic-wrapped, and unpackaged) were stored at 20 °C and 40 °C for four weeks. Daily visual observations and instrumental color measurements were conducted to monitor discoloration trends over time.



Figure 1. Documentation of measured product

Soak Test

One gram of shampoo was dissolved in 5 mL of demineralized water (1:5 w/v ratio) and left to stand. Color measurements were taken daily using the HunterLab instrument over a total period of 217 hours to evaluate color leaching into water.

Dip Test (Simulated Daily Use)

Unpackaged shampoo bars were immersed in demineralized water in a beaker for 1–2 minutes, twice daily (at 9:00 AM and 3:00 PM), simulating routine usage. Color measurements were conducted the following day to assess surface discoloration due to water exposure.

Catechin Solution Stability Test

A 100 ppm catechin solution was prepared by dissolving 10 mg of catechin powder in 100 mL of demineralized water. Two formulations were tested: Neutral solution: Catechin in water only Alkaline solution: Catechin solution with 2 g NaOH added Both solutions were stored under ambient conditions, and visual color changes were recorded daily to evaluate the effect of pH on catechin stability

Results and Discussion

Figure 2 indicates the progressive deterioration in the visual quality of the shampoo bars across all packaging treatments. The field stability of catechin-enriched shampoo bars was evaluated under three different packaging treatments: Sample Code 1 (complete packaging with aluminum tube, plastic wrap, and silica gel), Sample Code 2 (wrapped in plastic only), and Sample Code 3 (unpackaged). Visual and physical changes were observed over a 60-day storage period under practical conditions. At the initial observation (Day 0), all samples exhibited similar appearances—uniform shape, color, and texture—indicating homogeneity of formulation and absence of early degradation. By Day 15, noticeable differences began to emerge. Sample Code 3, the unpackaged shampoo bar, started to show surface dullness and minor cracking, likely due to moisture loss and oxidation. Sample Code 2 showed slight discoloration but maintained structural integrity. Meanwhile, Sample Code 1 remained stable with minimal changes, indicating the protective effect of multilayer packaging.

At Day 30, the degradation in Sample Code 3 became more evident, with increased surface cracks, fading color, and brittleness, suggesting exposure to environmental stress such as humidity, air, and light. Sample Code 2 exhibited moderate discoloration and mild surface deformation, particularly around the

edges. In contrast, Sample Code 1 maintained its original appearance, with only minor dulling observed, likely mitigated by the presence of the silica gel as a moisture absorber (Kešelj et al., 2025). By Day 60, the visual degradation was most severe in Sample Code 3, which showed complete surface roughening and color changes that potentially reflect catechin oxidation and base matrix instability (Munteanu & Apetrei, 2022; Tkacz et al., 2025). Sample Code 2 also showed progressive degradation, though less extensive than the unpackaged sample. Sample Code 1 continued to demonstrate the best stability, with only slight discoloration, supporting the hypothesis that combination packaging significantly extends product shelf life (Siddiqui et al., 2024; Turan et al., 2024).

These results highlight the importance of effective packaging in maintaining the physical integrity and visual appeal of shampoo bars, especially those enriched with natural antioxidants such as catechin, which may be susceptible to environmental degradation (Taarji et al., 2026). Multilayer packaging, including a desiccant, serves not only as a physical barrier but also as a stabilizing microenvironment for such bioactive formulations (Hu et al., 2025; Zhao et al., 2023).

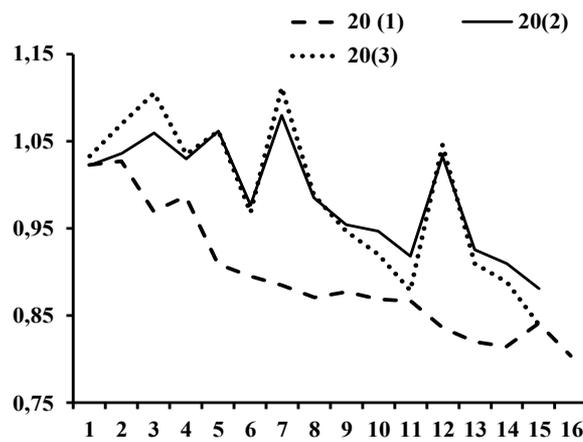


Figure 2. The changes of colour of gambier shampoo bar stored at 20 °C

Figure 3 presents the temperature profile during the 15-day storage of the catechin-based solid shampoo bar at 40 °C, monitored across three replicates (40(1), 40(2), and 40(3)). The data confirm that the storage conditions remained consistently stable, with all replicates showing minimal fluctuation, thereby validating the reliability of the accelerated aging environment. The close alignment of temperature curves among the three samples indicates uniform thermal exposure, which is essential for ensuring consistent simulation of elevated-temperature conditions aimed at

evaluating the shampoo bar’s thermal resilience (Diwoux et al., 2024; Rosik et al., 2025). Maintaining this stable 40 °C condition is critical to assessing the physicochemical stability of the product, including parameters such as hardness, color, fragrance retention, and the integrity of active compounds, particularly catechin, which is heat-sensitive and prone to degradation through oxidation and volatilization (Rezagholizade-shirvan et al., 2024). Elevated storage temperatures are known to accelerate reactions that may compromise both the functional and sensory qualities of cosmetic formulations (Vieira et al., 2024). Therefore, this temperature-controlled test provides valuable insight into the shelf-life performance and formulation robustness of the product. If degradation is observed under these conditions, it would indicate a need for reformulation (e.g., the incorporation of stabilizers or antioxidants) or improvements in packaging materials to enhance thermal protection.

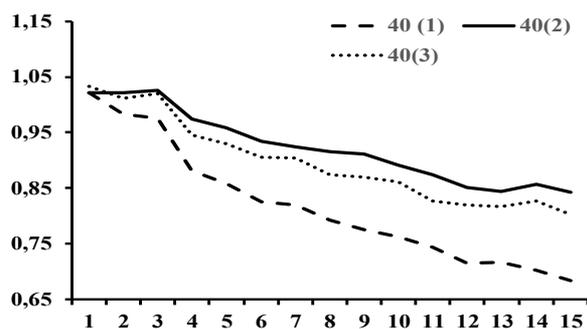


Figure 3. The changes of colour of gambier shampoo bar stored at 40 °C

Based on the hue data presented in Figures 2 and 3, which illustrate the color stability of gambir-based shampoo bars stored at 20°C and 40°C respectively, several assumptions can be made regarding the effect of packaging type and storage temperature on color degradation. The findings indicate that storing the catechin-based solid shampoo bar at 20°C with complete packaging protection—including an aluminum tube, plastic wrap, and silica gel—was most effective in maintaining the product’s color stability over time, as evidenced by minimal changes in hue values. In contrast, samples stored at 40°C, especially those with incomplete or no packaging, exhibited pronounced color degradation, likely due to the accelerated oxidation of catechin compounds under elevated temperatures and increased exposure to oxygen and humidity.

Color Response of Catechin-Based Solid Shampoo during Soaking Test

Table 1 presents the results of the soaking test conducted on catechin-enriched solid shampoo bars, specifically using Sample Code 1 (fully packaged with aluminum tin, plastic wrap, and silica gel). The initial hypothesis posited that prolonged immersion would intensify the brown coloration of the soaking solution due to the reaction between catechins and water, as catechins are known to undergo oxidation and polymerization in aqueous environments, especially under ambient conditions (Yin et al., 2022). Such a color change would typically be observed as a shift in hue values over time. However, the results revealed an unexpected trend: immersion did not consistently lead to browning of the solution. In fact, no significant darkening or visible formation of a brown hue was observed throughout the 217-hour soaking period. Instrumental measurements showed that the hue values fluctuated within a narrow range, alternating between slightly negative and slightly positive values, while the lightness (L*) values also varied but did not indicate consistent darkening. Notably, the highest hue value was recorded at 49 hours (1.47 ± 1.41), whereas the lowest was observed at 24 hours (-1.53 ± 1.41).

Table 1. Results of Color Changes in the Soaking Test of Gambier Shampoo Bar Solution.

Duration	Hue°± SD	L ± SD
4 hours	-0.72 ± 1.41	39.62 ± 7.85
8 hours	-1.01 ± 1.41	73.76 ± 7.85
24 hours	-1.53 ± 1.41	54.07 ± 7.85
28 hours	1.55 ± 1.41	58.14 ± 7.85
32 hours	-1.39 ± 1.41	54.17 ± 7.85
49 hours	1.47 ± 1.41	59.85 ± 7.85
56 hours	-1.44 ± 1.41	51.06 ± 7.85
60 hours	-1.46 ± 1.41	40.11 ± 7.85
64 hours	-1.37 ± 1.41	48.81 ± 7.85
73 hours	1.41 ± 1.41	56.69 ± 7.85
145 hours	1.24 ± 1.41	50.22 ± 7.85
169 hours	1.28 ± 1.41	57.17 ± 7.85
193 hours	1.45 ± 1.41	65.42 ± 7.85
217 hours	1.35 ± 1.41	64.21 ± 7.85

These shifts in hue may reflect structural transformations of catechins under alkaline conditions, as the solid shampoo matrix contains alkaline substances as part of the saponification process. Prior studies have shown that the stability of phenolic compounds, including catechins, is strongly influenced by pH, with alkaline environments capable of altering their oxidation pathways or preventing polymerization into brown pigments (Friedman & Jürgens, 2000; Zhu et al., 2002). It is possible that the basic pH of the shampoo inhibits the expected browning reaction of catechins with water, either by stabilizing their molecular structure or by

diverting them into alternative non-chromophoric degradation pathways. The absence of significant color change, despite extended exposure to water, may also indicate that catechins are either stabilized within the soap matrix or that their release into the aqueous phase is limited. This observation warrants further investigation into the physicochemical behavior of catechins in basic systems. Additional studies involving pH adjustments, antioxidant degradation kinetics, and structural analysis of catechin degradation products could provide deeper insights into the stability and release behavior of catechins in soap-based formulations.

The visual observation of shampoo bar samples after 24 hours of immersion is presented in Figure 4. The test aimed to evaluate the physical stability and potential color changes that may indicate chemical degradation, such as oxidation of catechins or other natural compounds present in the gambir-based formulation. As shown in the image, the samples did not exhibit significant brown discoloration, which often signals oxidative degradation or browning reactions associated with polyphenolic compounds (Nanjo et al., 1999). The color of the shampoo base remained relatively stable, with only a slight turbidity observed in the surrounding medium. This result suggests that the shampoo formulation, including the encapsulated gambir catechins, demonstrates adequate oxidative stability under aqueous exposure for a short-term period (24 hours). The absence of visible brown color development also indicates that the interaction between the shampoo matrix and water did not trigger notable degradation of the active components, especially when no transition metals or extreme pH conditions were present to catalyze oxidation (Coltelli et al., 2025). This reinforces the potential of the gambir-based shampoo bar to maintain visual and functional quality under typical user exposure scenarios.



Figure 4. Visual appearance of solid shampoo soak water after 24 hours of immersion

Dip Test

The dip test was conducted to evaluate the potential discoloration or leaching effect of solid shampoo when repeatedly exposed to water across different time intervals (1 to 4 days). This test was based on the same hypothesis as the soaking test, i.e., that the catechin-rich gambir-based formulation exhibits satisfactory oxidative and color stability when in contact with water. As presented in Table 3, the Hue° values remained relatively consistent across the 1st, 3rd, and 4th days, ranging between 0.84 to 0.85, with a slight increase to 1.05 on the 2nd day. A higher hue value typically indicates a shift toward yellow-red tones, possibly due to mild catechin oxidation. However, the changes were minor and statistically within a narrow margin (± 0.10), suggesting limited oxidative degradation of tannins and flavonoids over time (Hou et al., 2025).

The L* (lightness) values ranged from 30.24 to 44.14, with the highest brightness observed on day 2. This spike may indicate transient release of non-colored surfactant components or dispersion artifacts rather than actual pigment leaching. On subsequent days, L* values returned to the ~ 31 -range, similar to day 1, confirming that discoloration was not progressive. These findings are in line with observations that catechins are notably unstable in strongly alkaline solutions (pH > 8), with rapid degradation reported under such conditions, whereas neutral to mildly alkaline environments may allow for limited stability over short periods (Yan et al., 2022; Zare et al., 2025). Overall, these results reinforce the oxidative stability of the solid shampoo, even under simulated repeated-use conditions. The color remained relatively unchanged and did not exhibit significant darkening or browning, which is a favorable indicator for product shelf life and user acceptability.

Table 2. Results of Color Changes in the Dip Test of Gambier Shampoo Bar Solution.

Duration	Hue° \pm SD	L \pm SD
1day	0.84 \pm 0.10	30.24 \pm 6.47
2day	1.05 \pm 0.10	44.14 \pm 6.47
3day	0.85 \pm 0.10	31.78 \pm 6.47
4day	0.84 \pm 0.10	31.81 \pm 6.47

Figure 4 visually illustrates the color change of soaking water from the dip test of gambir-based solid shampoo, conducted over four consecutive days. Each container reflects the water appearance after the shampoo was briefly soaked, mimicking intermittent use conditions. Visually, no drastic color differences are observed between days, indicating the product's stability in terms of pigment or active compound leaching. The slight turbidity and brownish hue

observed, especially on Day 2, are likely caused by the partial release of surface-level catechins or surfactant traces, rather than deep-seated oxidation or breakdown. Notably, by day 3 and day 4, the water's color intensity appears to return to the tone observed on day 1, supporting the data that no cumulative leaching or destabilization occurred. This visual confirmation complements the instrumental data shown in Table 3, reinforcing that the gambir-based formulation maintains its integrity during repeated water contact. The consistency in visual appearance across four cycles of dipping indicates good retention of color and structural stability, which is important for consumer perception and product reusability.

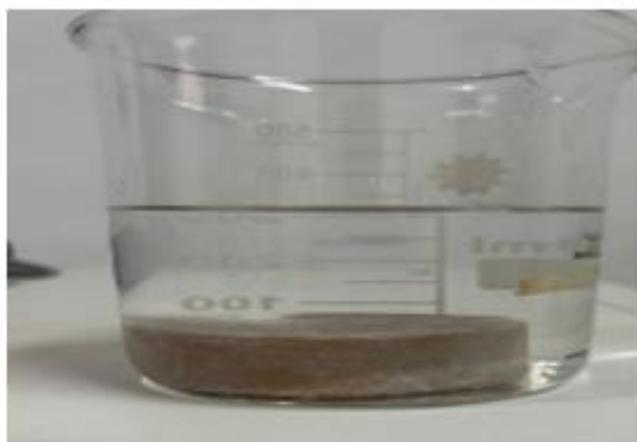


Figure 5. Visual of dipping test of solid shampoo

Comparative Analysis of Catechin Behavior in Water and Alkaline Media

To further investigate the behavior of catechins under different pH environments, an extended analysis was conducted by dissolving pure catechin powder into two types of solvents: demineralized water (neutral) and a 2% sodium hydroxide (NaOH) solution (alkaline). Both solutions were stored under ambient conditions for eight days. Visual observations revealed two distinct reaction pathways depending on the solvent environment (Lee et al., 2023; Lin et al., 2025).

In the neutral water-based solution, the catechin was initially colorless on the first day but gradually developed a yellow-brown hue over time—indicative of slow oxidation and polymerization of catechins, consistent with documented aqueous oxidative browning of catechins (Tan et al., 2025). In contrast, the alkaline solution exhibited a deep brown color immediately upon addition of catechin, which progressively faded to a lighter yellow over the storage period. This color transformation suggests degradation of chromophoric structures, possibly due to base-catalyzed oxidative breakdown or structural

rearrangements in the catechin molecule a behavior aligned with findings on flavanol degradation in strong alkaline environments involving ring cleavage and formation of simpler aromatic compounds (Dussling et al., 2024; Rogozinska & Biesaga, 2020). These findings highlight a marked difference in the reactivity and stability of catechins under neutral versus alkaline conditions.

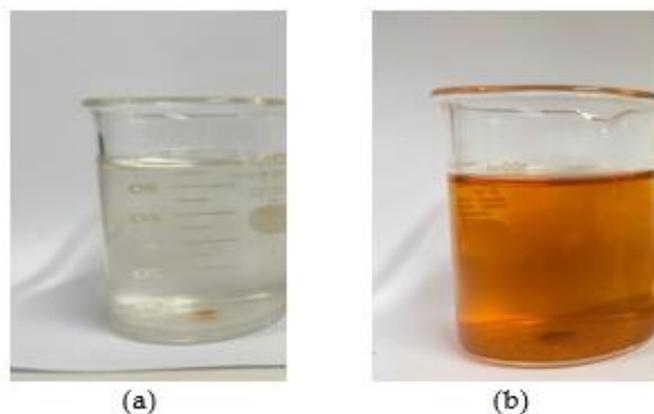


Figure 6. Comparison of 100 ppm catechin in water and alkaline solutions. (a) Observation on Day 1: water (left) and alkaline solution (right); (b) Observation on Day 8: water (left) and alkaline solution (right).

The immediate browning followed by fading in NaOH contrasts with the delayed but persistent browning in neutral water, suggesting divergent oxidative and degradation pathways. In the context of gambir-based product development—particularly soap and shampoo formulations which typically exhibit alkaline pH—this behavior provides a critical clue: structural transformations in alkaline media may alter typical oxidation pathways, thus influencing both visual and functional stability of the final product (Liu et al., 2025; Wijsekara & Xu, 2024). Further research is warranted to clarify the underlying chemistry, including kinetic modeling, structural identification of degradation products, and the influence of environmental factors (e.g., temperature, light, and metal ions). A deeper understanding of these mechanisms will support rational formulation design, packaging decisions, and storage strategies for catechin-enriched gambir-based personal care products (Dong et al., 2025).

Conclusion

This study highlights the crucial role of storage conditions and packaging in preserving the visual and functional stability of catechin-based solid shampoo derived from *Uncaria gambir* Roxb. The findings demonstrate that complete multilayer packaging

(aluminum tube, plastic wrap, and silica gel) significantly improves the product's resistance to discoloration, especially under elevated temperatures. In contrast, partial or no packaging leads to accelerated visual degradation, likely due to catechin oxidation. Simulated usage tests—including soaking and dip experiments—confirmed the shampoo's high stability in aqueous conditions, with minimal color change observed over time. Furthermore, the catechin stability test revealed that alkaline conditions can influence oxidation behavior, potentially contributing to improved stability in the shampoo matrix. These results suggest that formulation design, packaging strategy, and pH management are key to extending the shelf life and marketability of gambir-based personal care products. The insights gained from this study provide a strong foundation for further development and commercialization of eco-friendly, functional cosmetics using locally sourced bioactive compounds from Indonesia.

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Author Contributions

R. V wrote the manuscript, D. S designed the research, performed data analysis, and wrote the manuscript. R. T. A and R. R supervised the laboratory analysis. A. A contributed industrial perspective and insight. I. H. W developed the shampoo bar formulation. D. A. Z, N. R. ah and D. A. N assisted with laboratory experiments.

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Conflicts of Interest

The authors declare no conflict of interest.

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