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Formulation and Evaluation of Herbal Shampoo containing Red Dragon Fruit Peel Extract (*Hylocereus polyrhizus* (Hook.) Britton & Rose): toward A Potential Anti-Dandruff Application

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Abstract: Dandruff is a common scalp condition influenced by factors such as an oily scalp, hormones, and fungal infections. Red dragon fruit peel (*Hylocereus polyrhizus*) contains flavonoids with natural antimicrobial activity, making it a promising natural ingredient for scalp care. This study aimed to develop and evaluate herbal shampoo containing red dragon fruit peel extract. Four formulations were prepared with different concentrations of extract: F0 (base), F1 (6.25%), F2 (12.5%), and F3 (25%). Each was assessed for organoleptic properties, homogeneity, pH, viscosity, foam height, and foam stability. The stability test was evaluated using a cycling test. Data analysis with One-way ANOVA revealed that extract concentration significantly influenced pH and viscosity stability ($p < 0.05$). F1 showed optimal physical properties and complied with SNI 06-2692-1992 standards. These findings support the potential use of red dragon fruit peel extract in herbal shampoo formulations for future anti-dandruff applications.

Keywords: cosmetic, formulation, hylocereus polyrhizus, physical stability, shampoo

1. INTRODUCTION

Hair plays a vital role in enhancing physical appearance, particularly for women. Excessive activity can lead to scalp moisture, promoting dandruff formation. Dandruff affects 15%–20% of the global population and is more prevalent in Indonesia due to its tropical climate and high humidity levels [1]. Dandruff is characterized by excessive shedding of dead skin cells, usually appearing as white or yellowish flakes, predominantly on the scalp. It is associated with factors such as overactive sebaceous glands and the presence of scalp microorganisms, including *Candida albicans* [2][3].

The use of natural or herbal ingredients in personal care products is increasing due to their minimal side effects compared to synthetic agents [4], [5]. Red dragon fruit (*Hylocereus polyrhizus*) peel, often discarded as waste, contains various bioactive compounds such as flavonoids, alkaloids, and terpenoids [6][7]. These compounds are reported in the literature to exhibit antimicrobial and antifungal properties. Flavonoids, for example, act as natural antimicrobials by inhibiting cell proliferation [8]; alkaloids interfere with bacterial cell wall synthesis [9], and terpenoids interact with ergosterol in fungal cell membranes and walls, impairing their integrity and resulting in cell death [10]. Several studies have reported that red dragon fruit peel extract shows inhibitory activity against *Candida albicans*, a fungus often associated with dandruff. These studies demonstrated inhibition

zones and specific MIC values, indicating the biological potential of the active compounds within the extract [11][12]. Despite its biological potential, this extract has not been widely explored in cosmetic formulations for scalp treatment. Further studies are needed to support its functional claims and to explore its application in stable and well-characterized cosmetic formulations.

One potential way to utilize this extract is by incorporating it into a topical formulation, such as shampoo, for direct application to the scalp. In developing such formulations, excipients like hydroxypropyl methylcellulose (HPMC) are commonly used to achieve the desired viscosity and ensure product homogeneity. HPMC is a widely used gelling agent and stabilizer in cosmetic products and is compatible with aqueous systems and topical applications [13].

To address this research gap, this study aimed to formulate it into a shampoo preparation and evaluate its physical characteristics and stability. This preliminary investigation is intended as a foundation for further development of herbal shampoo formulations toward potential anti-dandruff applications.

2. MATERIALS AND METHODS

2.1. Materials

The simplicia of red dragon fruit peel (*Hylocereus polyrhizus* (Hook.) Britton & Rose) was obtained from PT. Palapa Muda Perkasa in powder form. Other materials used in this study were hydroxypropyl methylcellulose (Brataco), sodium lauryl sulfate (Alpha Chemika), propylene glycol (Brataco), propylparaben (Brataco), methylparaben (Brataco), triethanolamine (Emplura), peppermint oil (Happy Green), $FeCl_3$ (Merck), magnesium powder (Merck), concentrated hydrochloric acid (Merck), buffer solution pH 4.00, 7.00, and 9.00 (Merck), and ethanol 96% (Merck).

2.2. Extraction of Red Dragon Fruit Peel

A total of 500 grams of red dragon fruit peel simplicia powder was soaked for 3x24 hours in 3,750 mL of 96% ethanol [14]. The resulting filtrate was concentrated using a rotary evaporator (IKA) set at 40°C and 50 rpm. It was then further evaporated over a 50°C water bath until a thick extract was formed [15].

2.3. Extract Characterization

2.3.1. Moisture content

Moisture content was determined by weighing 2 g of extract into a porcelain dish and drying it in an oven at 105°C for 5 h until constant weight was obtained. The moisture content was calculated as the percentage loss in weight relative to the initial sample weight [16].

2.3.2. Total ash content

Total ash content was determined by incinerating 5 g of extract in a previously ignited and tared silica crucible at 600 ± 25°C until carbon-free ash was obtained. The residue was cooled, weighed, and expressed as a percentage of the initial sample weight [16].

2.3.3. The water- and ethanol-soluble extractive

For the water-soluble extractive value, 2 g of extract was macerated with 100 mL of chloroform-water (2.5 mL chloroform in 1000 mL distilled water) in a closed flask for 24 h, with shaking during the first 6 h and standing for the remaining 18 h. The mixture was filtered, and 20 mL of the filtrate was evaporated to dryness in a previously tared dish and dried at 105°C to constant

weight. The result was expressed as the percentage of water-soluble constituents relative to the initial extract weight.

The ethanol-soluble extractive value was determined using the same procedure with 100 mL of 95% ethanol as the solvent [16].

2.4. Phytochemical Screening

2.4.1. Flavonoid

One gram of the extract was mixed with 10 mL of hot water, boiled for 5 minutes, and filtered while still hot. A 5 mL portion of the filtrate was added to 0.1 grams of magnesium powder, 1 mL of concentrated hydrochloric acid, and 2 mL of amyl alcohol. The mixture was shaken and allowed to separate. The color in the amyl alcohol layer was observed; the appearance of red, yellow, or orange coloration suggested the presence of flavonoids [17].

2.4.2. Tannin

One gram of the extract was dissolved in 15 mL of distilled water and subsequently filtered. Subsequently, 2 mL of 1% ferric chloride ($FeCl_3$) solution was added. A color change to blue or dark green indicated the presence of tannins [18].

2.4.3. Saponin

Approximately 0.1 grams of the extract was placed in a test tube and mixed with 10 mL of hot water. After cooled, the solution was shaken vigorously for 10 seconds. The formation of foam measuring 1-10 cm within less than 10 minutes indicated the presence of saponins [19].

2.5. Shampoo Formulation

The shampoo formulations developed in this study, consist of a base formulation (F0) and three variations containing red dragon fruit peel extract at different concentrations (F1, F2, and F3). These formulations incorporate common cosmetic excipients such as surfactants, humectants, gelling agents, and preservatives to ensure stability and functionality [20], as presented in Table 1.

Table 1. Shampoo Formulation

Nama Bahan	**Formula (%)**			
	F0	**F1**	**F2**	**F3**
Red Dragon Fruit Peel Extract	0	6.25	12.5	25
Sodium Lauryl Sulfate	2.5	2.5	2.5	2.5
Propylene Glycol	15	15	15	15
HPMC	2	2	2	2
Propyl paraben	0.02	0.02	0.02	0.02
Methyl paraben	0.18	0.18	0.18	0.18
Triethanolamine	1	1	1	1
peppermint oil	0.5	0.5	0.5	0.5
Aquadest	Ad 100 mL	Ad 100 mL	Ad 100 mL	Ad 100 mL

The preparation of the shampoo began by heating aquadest and propylene glycol in separate beakers on a magnetic stirrer (IKA) until the temperature reached 60°C-70°C. Methyl paraben and propyl paraben were dissolved in the hot propylene glycol to form mass 1. Simultaneously, HPMC was dispersed into the hot distilled water to form mucilage. Mass 1 was then gradually added to the

HPMC base, followed by sodium lauryl sulfate and additional distilled water to produce mass 2. The extract, dissolved in distilled water at specific concentrations, was incorporated into mass 2. Finally, triethanolamine and peppermint oil were added, and the mixture was then brought to a final volume of 100 mL using distilled water.

2.6. Physical Evaluation of shampoo preparation)

Physical evaluation of the shampoo formulations was carried out to ensure that each product met the required quality standards and exhibited acceptable performance. The assessments included organoleptic testing, homogeneity testing, pH measurement, viscosity measurement, foam height measurement, foam stability measurement, and evaluation of the physical stability of the shampoo preparations. These tests provided a comprehensive overview of the characteristics and overall quality of the formulations. All evaluations were conducted in triplicate to ensure reliable and reproducible results.

2.6.1. Organoleptic Test

The organoleptic evaluation was conducted to assess the physical characteristics of the shampoo formulations, including their appearance, color, and odor. These parameters were observed visually to ensure product consistency and acceptability [21].

2.6.2. Homogeneity Test

To evaluate homogeneity, a small amount of shampoo was placed between two glass slides and examined for the presence of coarse particles or visible clumps. According to SNI 06-2692-1992, a shampoo formulation is considered homogeneous when it displays a smooth and uniform consistency without any visible solid particles [22].

2.6.3. pH Test

One gram of shampoo sample was dissolved in 10 mL of distilled water. The pH meter was calibrated using standard buffer solutions at pH 4.00, 7.00, and 9.00 selected to cover the expected pH range of the samples, following standard calibration principles recommending the use of buffers that bracket the sample pH [23], [24]. The electrode was then immersed in the solution until a stable reading was obtained. The acceptable pH range for shampoo preparations is 5.0–9.0 [22].

2.6.4. Viscosity Test

Viscosity was determined by placing the shampoo sample into a 100 mL beaker and measuring it with a Brookfield viscometer using spindle number 3 at a rotation speed of 30 rpm [25]. The viscosity for shampoo should range from 400 to 4,000 cP [26].

2.6.5. Foam Height Test

A total of 1 gram of shampoo was diluted in 10 mL of distilled water, then shaken for 1 minute. The foam height produced was subsequently measured with a ruler. The acceptable foam height for shampoo products ranges from 1.3 to 22 cm [27].

2.6.6. Foam Stability Test

One gram of shampoo was diluted in 10 mL of distilled water and shaken for 1 minute. The initial foam height was measured immediately after shaking, and a second measurement was taken after 5 minutes to evaluate foam retention over time [28]. Foam stability was calculated using the following formula:

$$\text{Foam stability (\%)} = \frac{Final\ foam\ height}{Initial\ foam\ height}$$

2.7. Physical stability test of shampoo preparation

The shampoo's physical stability was evaluated using the cycling method. Shampoo was stored at low temperature (4°C) for 24 hours, then moved to a high temperature (40°C) for 24 hours, forming one cycle. This process was repeated for six cycles (12 days). Upon completion of the six storage cycles, the formulation was evaluated of its organoleptic properties, pH, homogeneity, viscosity, foam height, and foam stability [29].

2.8. Data Analysis

Statistical evaluation was performed on data obtained after the cycling stability test. Quantitative data from physical evaluations, including pH, viscosity, foam height, and foam stability (n = 3), were expressed as mean ± standard deviation (SD). Prior to analysis, data were assessed for normality and homogeneity as prerequisites for parametric testing. One-way ANOVA was applied to datasets that met these assumptions, with a p-value < 0.05 considered statistically significant, followed by a Post Hoc to identify specific differences between groups. For data that did not meet normality assumptions even after transformation, the Kruskal–Wallis test was used as a non-parametric alternative. All statistical analyses were performed using IBM SPSS Statistics.

3. RESULTS AND DISCUSSION

3.1. Extraction and Characterization of The Extract

The red dragon fruit peel simplicia powder was extracted using maceration with 96% ethanol as the solvent. The use of 96% ethanol in the extraction process was based on its broad solvency properties, allowing efficient separation of a wide range of secondary metabolites, particularly those with semi-polar characteristics such as flavonoids, terpenoids, and alkaloids. Moreover, ethanol is recognized as a safe pharmaceutical-grade solvent with low residual toxicity and rapid evaporation, making it suitable for topical and cosmetic applications.

A total of 138.13 grams of thick extract was obtained, yielding a 27.62% extract, which complied with the required standard of at least 10%. The yield represents the ratio between the mass of the extract obtained and the mass of the simplicia. A higher extract yield indicates a greater efficiency in obtaining bioactive compounds from the simplicia[30]. Subsequently, extract characterization testing was carried out to ensure the quality, efficacy, and safety of the extract. The results of the extract characterization are presented in Table 2.

Table 2. Characterization of the Extract

No	Test	Result (%)	Requirements (%)
1	Moisture content	26.2	5-30 [31]
2	Total ash content	5.22	Not more than 10 [32]
3	Water-soluble extractive	67.5	Not less than 12 [33]
4	Ethanol-soluble extractive	15	Not less than 8 [33]

Based on Table 2, it can be observed that the characterization tests of the red dragon fruit peel extract have met all the specified extract characterization parameters. Moisture content is a critical parameter for assessing residual water following the evaporation process, and the value obtained was within acceptable quality limits. Moisture content is also related to extract purity. Low

moisture content is desirable, as it reflects higher extract purity and reduces the risk of microbial growth, which can compromise extract stability [34].

The total ash content provides an estimate of both endogenous and exogenous mineral residues resulting from the raw material and processing stages [16]. The ash content indicates the amount of mineral elements present in an extract. Excessively high ash content is undesirable, as certain minerals in the ash can cause precipitation in the kidneys, potentially affecting health [35].

The determination of water-soluble and ethanol-soluble extractives serves to estimate the content of polar and semi-polar compounds, respectively [34]. The results showed that red dragon fruit peel extract yielded a higher proportion of water-soluble compounds, suggesting a predominance of polar constituents over nonpolar ones in the extract.

3.2. Phytochemical Screening

Phytochemical analysis was carried out to determine the secondary metabolites contained in the extract of red dragon fruit peel. This testing includes the identification of flavonoids, tannins, and saponins. The results are shown in Table 3.

Table 3. Phytochemical Screening Results

Compound Group	Result	Observation
Flavonoid	+	Orange-red color
Tannin	+	Dark green color
Saponin	+	1 cm foam

The detection of flavonoids, tannins, and alkaloids in this study aligns with previous phytochemical reports on red dragon fruit peel extract. These compounds have been associated with various bioactivities, which may support the rationale for their inclusion in topical formulations [36]. While antifungal activity was not directly evaluated, the detection of key secondary metabolites supports its potential functional role in scalp care formulations [37].

3.3. Physical Evaluation

Physical evaluation was carried out to determine the best physical parameters of formula by comparing the physical parameters of the shampoo formulations with the accepted values. The results of physical parameters including organoleptic, homogeneity, pH, viscosity, foam height, and foam stability tests of each formulation, are presented in Table 4.

Table 4. Physical Characterization of Shampoo Formulations

Parameter	F0	F1	F2	F3	Acceptance
Organoleptic	Clear white, mint scent	Deep brown, mint scent	Deep brown, mint scent	Deep brown, mint scent	-
Homogeneity	Homogeneous				Homogeneous
pH	7.26 ± 0.02	7.28 ± 0.06	7.38 ± 0.01	7.03 ± 0.01	5.0 – 9.0
Viscosity (cP)	3.932 ± 6.93	3.928 ± 6.93	746 ± 2.31	1.208 ± 6.93	400-4.000
Foam height (cm)	2.5 ± 0.42	2.7 ± 0.46	2.3 ± 0.20	3.1 ± 0.42	1.3 – 22
Foam stability (%)	65.78 ± 2.94	65.64 ± 3.39	65.40 ± 4.90	67.05 ± 0.69	60% - 70%

Data are expressed as mean ± standard deviation (SD), n = 3.

The shampoo formulations met general expectations for physical appearance, showing uniform color, homogeneity, and pleasant fragrance, all of which are critical factors influencing

consumer acceptance. The pH values of all formulations were within the acceptable range for scalp products (5.0-9.0), which supports scalp compatibility and reduces the risk of irritation [38]. The physical appearance of the preparations is presented in Figure 1.

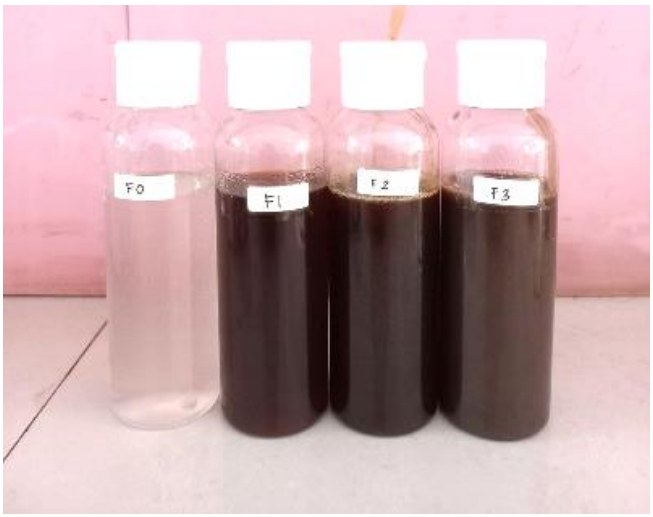

Figure 1. Physical appearance of the shampoo formulations

The viscosity test results show that the formulas exhibit different viscosity levels. F2 and F3 have lower viscosity values, resulting in a slightly thinner texture. This may be attributed to the increased concentration of red dragon fruit peel extract in the formulation. The gel formation mechanism of HPMC involves interactions between the polymer and the solvent (water). A higher extract concentration reduces the available water volume, disrupting these interactions and hindering gel formation [39]. Additionally, another contributing factor is syneresis, a process where trapped liquid escapes from the gel matrix to the surface, further reducing the formulation's viscosity [40]. However, all formulas still meet the viscosity requirements of SNI 06-2692-1992 (400-4,000 cP).

The results of the foam height test indicate that all formulations complied with the standard range (1.3–22 cm) [41]. Foam volume is often associated with aesthetic perception, as consumers tend to associate abundant foam with higher cleansing efficacy. Nevertheless, foam quantity does not directly reflect the cleansing performance of a shampoo [42].

In terms of foam stability, all formulations also fulfilled the specified requirements (60%–70%) [43]. Foam stability tends to decline when the liquid film surrounding the bubbles becomes too thin. Several factors may influence this parameter, including the testing method itself. In particular, inconsistencies in shaking intensity can affect the reliability of the observed stability.

3.4. Stability Testing (Cycling test)

Stability testing is a critical parameter for evaluating a product's ability to maintain its quality within specified limits throughout storage and use periode. In this study, the cycling test was conducted by subjecting the sample to alternating storage at 4 °C and 40 °C for six cycles. Each testing cycle involved storing the product at 4°C for 24 hours, followed by storage at 40°C for another 24 hours [29]. The detailed values of the stability testing are presented in Table 5. A graphical representation of the changes in physicochemical parameters before and after stability testing is shown in Figure 2.

Table 5. Stability Testing of Shampoo Formulations

Parameter	Cycle	F0	F1	F2	F3
Organoleptic	0	Clear white, mint scent	Deep brown, mint scent	Deep brown, mint scent	Deep brown, mint scent
	6	Clear white, mint scent	Deep brown, mint scent	Deep brown, mint scent	Deep brown, mint scent
Homogeneity	0	Homogeneous	Homogeneous	Homogeneous	Homogeneous
	6	Homogeneous	Homogeneous	Homogeneous	Homogeneous
pH	0	7.26 ± 0.02	7.28 ± 0.06	7.38 ± 0.01	7.03 ± 0.01
	6	7.10 ± 0.01	7.00 ± 0.01	5.92 ± 0.01	5.51 ± 0.01
Viscosity (cP)	0	3932 ± 6.93	3928 ± 6.93	746 ± 2.31	1208 ± 6.93
	6	3932 ± 5.36	3920 ± 4.72	628 ± 4.00	560 ± 4.00
Foam height (cm)	0	2.5 ± 0.42	2.7 ± 0.46	2.3 ± 0.20	3.1 ± 0.42
	6	3.5 ± 0.20	4.2 ± 0.25	3.9 ± 0.82	3.9 ± 0.38
Foam stability (%)	0	65.78 ± 2.94	65.64 ± 3.39	65.40 ± 4.90	67.05 ± 0.69
	6	63.82 ± 1.80	67.97 ± 1.60	63.64 ± 2.91	63.39 ± 4.34

Data are expressed as mean ± standard deviation (SD), n = 3.

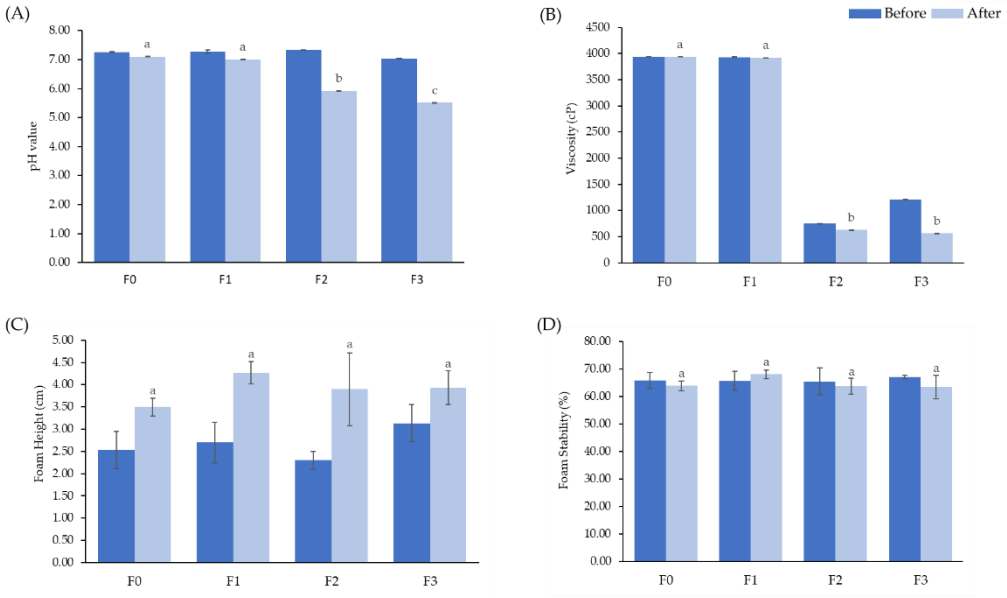

Figure 2. Physical characteristics of shampoo formulations (F0–F3) before and after stability testing: (A) pH, (B) viscosity, (C) foam height, and (D) foam stability. Different letters above the bars indicate statistically significant differences among formulations after stability testing ($p < 0.05$).

The stability evaluation demonstrated that all shampoo formulations maintained acceptable physical characteristics throughout the observation period. Organoleptic assessments confirmed the consistency of color, odor, and texture over six stability cycles. Similarly, all formulations remained homogeneous, with no visible phase separation or coarse particles, indicating good physical stability of the system.

A slight reduction in pH values was observed across all formulations during the cycling test. Nevertheless, the pH remained within the acceptable range of 5.0–9.0 as defined by SNI 06-2692-1992. The decrease in pH may be attributed to temperature fluctuations during the cycling process, which can influence pH stability [44]. Statistical analysis showed that pH values were normally distributed and homogeneous ($p > 0.05$). One-way ANOVA revealed a significant difference among formulations

($p < 0.05$), which was confirmed by post hoc analysis. Formulations marked with different letters above the bars in Figure 2(A) were significantly different, indicating that extract concentration influenced pH values after stability testing.

Viscosity stability was also evaluated. Formulation F0 and F1 maintained consistent viscosity, while F2 and F3 showed a noticeable decrease. This reduction may be attributed to the higher extract concentration, which could interfere with the formation of the HPMC network structure. The presence of bioactive compounds in the extract may disrupt intermolecular interactions, leading to a weaker structural network. Furthermore, temperature fluctuations during the cycling test may accelerate structural rearrangement and reduce the strength of molecular interactions, resulting in decreased viscosity [39], [45]. Statistical analysis confirmed normal and homogeneous data ($p > 0.05$). One-way ANOVA showed significant difference among formulations ($p < 0.05$), which was further supported by post hoc analysis. Formulations marked with different letters in Figure 2(B) indicate statistically significant differences, suggesting that higher extract concentrations may reduce viscosity.

In the foam height and stability test, no significant changes were observed during the storage period. Although slight variations were observed, particularly an increase in foam height, all values were considered acceptable. The data were normally distributed ($p > 0.05$), and One-way ANOVA analysis showed no significant differences among formulation ($p > 0.05$), indicating that extract concentration did not significantly influence foam height or foam stability. However, F2 formulation exhibited relatively larger error bars in foam height as shown in Figure 2 (C), indicating higher variability among replicates compared to other formulations. This variability may be attributed to interactions between the extract and other formulation components, which could influence foam formation and stability. Additionally, foam systems are inherently dynamic and sensitive to experimental conditions, such as air incorporation and measurement timing [46], [47].

Based on the overall evaluation, formulation F1 was selected as the optimal formulation. Although all formulations met the acceptable physicochemical criteria, F1 demonstrated the most balanced performance in terms of stability and physical characteristics. The pH value of F1 remained within the acceptable range after cycling and showed relatively minimal fluctuation compared to higher extract concentrations. In addition, F1 maintained viscosity stability without a significant reduction during storage, unlike F2 and F3, which exhibited a noticeable decrease. Foam height and foam stability of F1 were also within the acceptable range and showed no significant changes throughout the cycling test. Therefore, considering physicochemical stability and overall performance, F1 was determined to be the most stable and suitable formulation.

4. CONCLUSION

Shampoo formulations containing red dragon fruit peel extract at concentrations of 6.25%, 12.5%, and 25% showed significant differences in pH and viscosity characteristics and stability, while foam height and foam stability remained unaffected. Formula F1 demonstrated the most stable and acceptable physical properties based on SNI standards.

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