

## Article

# Unlocking the Potential: A Comprehensive Analysis of the Technological Properties and Consumer Perception of Shampoo Enriched with Patchouli Extract and Allantoin

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**Abstract:** Amidst a growing shift towards eco-friendly choices in personal care products, the challenge of formulating herbal shampoos with efficacy comparable to synthetic counterparts persists. This study investigates the potential of incorporating patchouli extract and allantoin as additives in anti-dandruff shampoo formulations, assessing their impact on the technological properties of the product. With limited research on their efficacy, our investigation contributes valuable insights to the development of effective and consumer-friendly shampoos targeting dandruff concerns. Physicochemical characteristics (pH, surface tension, texture) were evaluated, alongside specific quality assessments such as wetting time, dirt dispersion, foaming, and cleaning action, in in vivo consumer research. Shampoo formulations incorporating 0.5% Patchoul'Up™ and 1% allantoin exhibited acceptable properties. However, the addition of plant-derived ingredients resulted in a beneficial decrease in surface tension (5.87%). Nevertheless, a decrease in cohesiveness (18%) over a 5-month period resulted in rheological changes, indicating potential instability ( $p < 0.05$ ). While the consumer evaluation aligns with laboratory findings, continuous research is essential to ensure stability and validate the anti-dandruff potential of the formulation, both in vitro and in vivo. This involves expanding the number of volunteers, with a specific focus on individuals experiencing dandruff concerns, to assess the shampoo's efficacy and impact on diverse user experiences.

**Keywords:** patchouli extract; allantoin; texture analysis; shampoo; formulation; consumer evaluation



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## 1. Introduction

Shampoos, initially devised for scalp cleansing, have evolved into versatile products that cater to diverse hair types, care preferences, and scalp conditions. Typically formulated as aqueous solutions or emulsions, shampoos comprise a mixture of surfactants (synthetic or natural), cleansing and foaming agents, along with other excipients (viscosity controllers, emollients, preservatives, etc.) and active ingredients [1,2]. The surfactants play a crucial role in both the cleaning and lathering abilities of shampoos and contribute to their skin tolerance [3].

Modern consumers, driven by concerns about synthetic chemicals impacting long-term hair health and the environment, are increasingly inclined toward herbal shampoos [4]. The perception of safety and minimal side effects associated with natural-origin products contributes to the rising popularity of herbal shampoos [1,2]. Numerous medicinal plants, known for their beneficial effects on hair, are commonly used in shampoo formulations [5]. However, creating an herbal shampoo that competes favorably with synthetic ones in terms of foaming and detergency remains a challenging task [6]. Considering the diverse phytochemical composition of plant extracts, it becomes crucial to assess not only their therapeutic effects but also the changes in technological properties resulting from their

incorporation into formulations. This is particularly significant when dealing with active ingredients, where compatibility with the base formulation can be challenging. To address this, various shampoo compositions were prepared and evaluated, encompassing formulations with and without active ingredients.

Patchouli extracts and essential oil, widely used in various industries (pharmaceutical, food, perfume, cosmetic), contain bioactive ingredients, including sesquiterpenes, known for potential health benefits [7]. Despite its traditional uses, patchouli is recognized for its antimicrobial and anti-fungal properties, making it valuable for managing various skin and scalp conditions, including fungal infections, acne, dermatitis, and even dandruff [8,9]. However, these qualities have not been extensively proven in shampoo formulations, with limited available research [7]. To our knowledge, there are no available data on the innovative ingredient Patchoul'Up™—a 100% upcycled active ingredient crafted through green fractionation from distilled patchouli leaves, which was used throughout the research.

Allantoin, a plant-derived substance, stimulates cell proliferation, promoting internal and external wound healing. Allantoin has been incorporated into various cosmetic products, including shampoos, to improve skin health by reducing inflammation and irritation [10]. It dissolves the intercellular cement that holds the corneocytes together, promoting natural desquamation of the stratum corneum, increasing skin smoothness and water binding to the intercellular matrix and keratin [10,11]. These properties make it well suited for an anti-dandruff shampoo.

Lemon essential oil contains primarily monoterpenoids, with D-limonene being a key compound believed to confer anti-inflammatory properties. These beneficial chemical constituents contribute to the significant role of *Citrus limon* in both the food and cosmetics industries [12]. In shampoos, it acts as a natural cleanser suitable for normal-to-oily hair types. Its acidic nature tightens hair follicles, promoting healthier growth and reducing hair fall while enhancing hair shine [13,14]. Research has shown that lemon peel extract exhibits potent antimicrobial activity against *Candida albicans*, *Aspergillus*, and *Aspergillus flavus*, suggesting potential benefits against dandruff. Furthermore, lemon juice and lemon peel powder effectively inhibit the growth of *Malassezia furfur*, a fungus responsible for causing dandruff [15,16].

Dandruff, a common dermatological condition affecting 5% of the population, is characterized by excessive scaling of scalp tissue [1,17]. To address the individuals dealing with dandruff who prefer using natural ingredients rather than opting for medicinal shampoos, this research aims to formulate a stable herbal shampoo containing allantoin, patchoul'Up™, with the potential to benefit individuals dealing with dandruff concerns. Also, this comprehensive evaluation aims not only to shed light on the potential impact of plant extracts on therapeutic efficacy but also the technological characteristics of the shampoo base.

## 2. Materials and Methods

### 2.1. Materials

Lemon fruit essential oil was purchased from “ACappella naturals” (distributor UAB “Aromika”, Kaunas, Lithuania). Allantoin, potassium sorbate, sodium benzoate, and citric acid were procured from “Sigma-Aldrich” (Taufkirchen, Germany). Cocamidopropyl betaine and glyceryl cocoate were purchased from “Dragonspice Naturwaren” (Reutlingen, Germany). Decyl glucoside and sodium cocoyl isethionate were obtained from “Berg + Schmidt” (Hamburg, Germany). Patchoul'Up™ was purchased from “Givaudan Active Beauty” (Jawa Barat, Indonesia). D-panthenol was bought from “ThermoFisher” (Karlsruhe, Germany). Indian ink was purchased from “Daravija” (Ariogala, Lithuania) manufactured by “Koh-In-Noor” (České Budějovice, Czech Republic). Parfum was obtained from the local distributor “ELL” (Vilnius, Lithuania), and purified water was made in the Lithuanian University of Health Sciences laboratory according to Ph. Eur. 01/2008:0008.

## 2.2. Methods

### 2.2.1. Shampoo Formulation

The formulation process started by dissolving D-panthenol in purified water, followed by the addition of allantoin and Patchoul'Up™ if necessary (Table 1). Subsequently, preservatives (potassium sorbate and sodium benzoate) were added and stirred until fully dissolved. The anionic detergent sodium cocoyl isethionate was incorporated and mixed until complete dissolution. Next, the non-ionic detergent decyl glucoside was added, followed by the amphoteric detergent cocamidopropyl betaine. Glycerol cocoate was introduced, followed by the addition of lemon essential oil and parfum. Finally, a few drops of a 50% citric acid solution were added to achieve the desired pH.

**Table 1.** Compositions of formulated shampoos.

Ingredients	Compositions (%)			
	1	2	3	4
Sodium cocoyl isethionate	10.0	10.0	10.0	10.0
Cocamidopropyl betaine	10.0	10.0	10.0	10.0
Decyl glucoside	5.0	5.0	5.0	5.0
Glycerol cocoate	5.0	5.0	5.0	5.0
D-panthenol	1.0	1.0	1.0	1.0
Potassium sorbate	0.1	0.1	0.1	0.1
Sodium benzoate	0.1	0.1	0.1	0.1
Parfum	1.0	1.0	1.0	1.0
Patchoul'Up™	-	0.5	0.5	0.5
Lemon essential oil	0.1	0.1	0.1	0.1
Allantoin	-	0.5	1	1.5
50% citric acid solution	*	*	*	*
Purified water	67.7	66.7	66.2	66.2

\* as much as needed to reach the appropriate pH.

### 2.2.2. Determination of pH Value

A 10% aqueous solution of shampoo was made carefully to avoid foam formation and filtered through a paper filter with 20–25 µm pore size (DP 411, Albet-Hahnmuehle S.L., Barcelona, Spain) [18]. The pH was measured at room temperature ( $22 \pm 2$  °C) using a pH meter (inoLab® pH/ION 7320, Berlin, Germany).

### 2.2.3. Determination of Dirt Dispersion

To initiate the experiment, two drops of herbal shampoo were introduced into a wide-mouthed Falcon tube filled with 10 mL of distilled water. Following this, a single drop of India ink was incorporated. The Falcon tube was securely covered and subjected to ten vigorous shakes. The discernible presence of ink within the resulting foam was assessed using qualitative descriptors: None, Light, Moderate, or Heavy [19].

### 2.2.4. Determination of Wetting Time

The wetting time was determined by observing the duration it took for a canvas paper to fully submerge into 1% aqueous solution of shampoo. The size of the canvas paper was 0.44 g, with a diameter of approx. 2.54 cm. The canvas paper disc was placed on the surface of the sample solution and the stopwatch was used to measure the time it took for the paper to submerge entirely [19].

### 2.2.5. Determination of Surface Tension

Throughout the experiment, 10% aqueous shampoo solutions were consistently utilized. The experiment was conducted at room temperature, employing a Traube stalagmometer with a diameter of 0.62 mm. Equal volumes of test samples were filled into the stalagmometer, secured in a stand, and allowed to descend gradually until reaching the

mark on the tube [18]. The number of drops was counted and the surface tension was calculated using Equation (1).

$$R2 = \frac{(W3 - W1)N1}{(W2 - W1)N2} \times R1, \quad (1)$$

where  $W_1$ —weight of empty beaker (g);  $W_2$ —weight of beaker with distilled water (g);  $W_3$ —weight of beaker with test solution (g);  $N_1$ —no. of drops of distilled water;  $N_2$ —no. of drops of shampoo solution;  $R_1$ —surface tension of distilled water at room temperature (72.75 mN/m);  $R_2$ —surface tension of shampoo solution (mN/m).

#### 2.2.6. Determination of Foaming Ability and Foam Stability

The foaming ability was assessed using a modified cylinder shake method. In total, 50 mL of the 1% aqueous solution of shampoo was poured into a 250 mL graduated measuring cylinder, covered with parafilm, and inverted ten times. The total volume of the foam content was then recorded. The recordings were taken again after 1, 2, 3, and 4 min as well to account foam stability [19].

#### 2.2.7. Detergency Test

The cleansing efficacy of the developed shampoo was assessed through the slightly modified washing-of-wool-yarn technique [18]. Initially, 5 g of wool yarn, pre-moistened with sunflower oil, was added into a cylinder containing 200 mL 35 °C purified water along with 1 g of the test sample (shampoo or control solution). The cylinder was subjected to shaking for approximately 4 min, with around 50 shakes per minute. Following the shaking process, the water was drained from the cylinder, and the yarn threads were dried and weighed; 35 °C purified water was used as the control solution. The cleansing action was determined using Equation (2).

$$DP = 100 \left( 1 - \left( \frac{T}{C} \right) \right) \quad (2)$$

where DP—detergency power (%); T—weight of yarn after shaking with shampoo (g); C—weight of yarn after shaking with water (g).

#### 2.2.8. Texture Analysis

It was performed using the back extrusion test by the Ta.Xtplus Texture Analyzer (Stable Micro Systems, Vienna, Austria). During this test, four parameters were determined: firmness (g), consistency (g·s), cohesiveness (g), and viscosity index (g·s). The back extrusion rig, denoted as A/BE, consisted of a sample container filled with the developed shampoo, positioned on the analyzer platform beneath a disc plunger. The compression test was initiated by lowering the test probe into the sample at a speed of 3 mm/s, causing the product to extrude up and around the disc's edge. Once a trigger force of 5 g was reached, the disc plunger initiated the deformation of the sample to a predetermined distance (5 mm), following which the probe returned to its initial position [20].

#### 2.2.9. Consumer Evaluation

The research was granted approval (No. BEC-FF-52) by the Lithuanian University of Health Sciences Center for Bioethics after evaluating the protocol. Ten students from the Lithuanian University of Health Sciences, serving as volunteers, participated in the study. The inclusion criteria comprised being healthy, proficient in Lithuanian and English, aged above 18, without a medical history of scalp conditions or diseases (except for dandruff), and no known allergic reactions to the shampoo ingredients. Informed consent was obtained from all subjects involved in the study, acknowledging the objectives and methods of the research. Participants refrained from using any hair or scalp care products 12 h before and during the study. Each participant received 30 g of shampoo for hair washing. To

assess the quality of the formulated shampoo, all volunteers responded to specific questions outlined in the questionnaire (Appendix A).

#### 2.2.10. Microscopic Evaluation

The microscopic analysis was carried out using an optical microscope (BMS 739960) equipped with a digital USB camera (Breukhoven, Netherlands) for processing the image (400× magnification fold). The diameter of droplets in each sample was measured using previously calibrated BMS 2.0 software.

#### 2.2.11. Statistical Analysis

Data are presented as mean  $\pm$  SD. Statistical analysis was performed by one-way and two-way analysis of variance (ANOVA), followed by Dunnett's post-test using the software package Prism 6.0 (GraphPad Software Inc., La Jolla, CA, USA). A value of  $p < 0.05$  was taken as the level of significance.

### 3. Results and Discussion

Given the diverse phytochemical composition of plant extracts and the complexities associated with integrating various active ingredients, it is imperative to assess not only their therapeutic effects but also the resulting changes in technological properties. This becomes particularly crucial when dealing with active ingredients or unknown composition plant extracts, where compatibility with the base formulation can pose challenges due to the lack of scientific research data. As part of our investigation, we meticulously prepared several shampoo compositions, including the shampoo base and various formulations containing investigated active ingredients (Table 1).

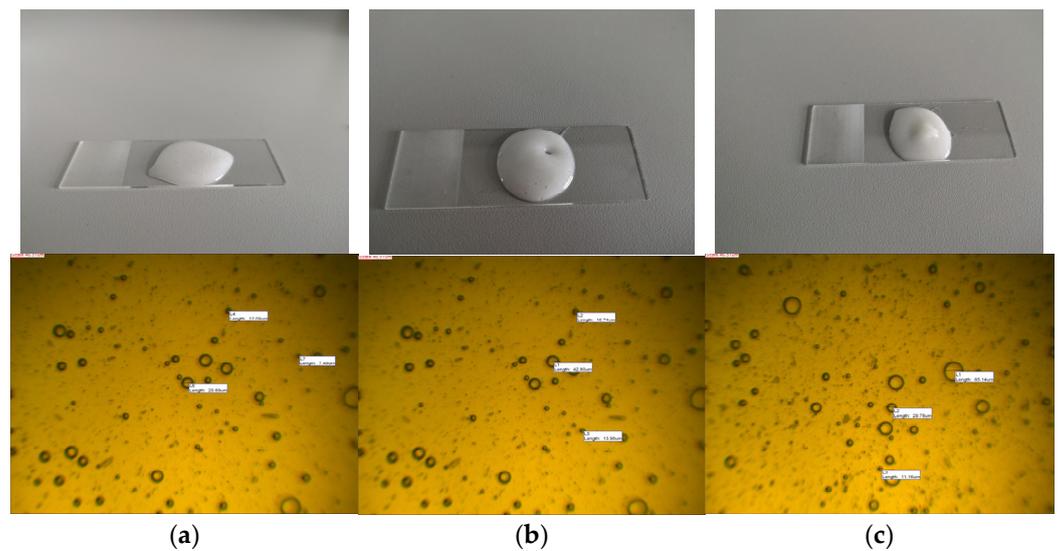
#### 3.1. Physical Appearance and Microscopic Evaluation

In the assessment of the physical appearance, the developed formulation underwent evaluation for clarity, color, and odor. Shampoo formulas were crafted with varying concentrations of allantoin powder, specifically 0.5%, 1%, and 1.5%. Becker et al determined that it is safe to use up to 2% allantoin in cosmeceuticals [10]. Visual examination revealed that the addition of 1.5% allantoin resulted in a less homogeneous shampoo, while 0.5% and 1% concentrations produced a more uniform and consistent product.

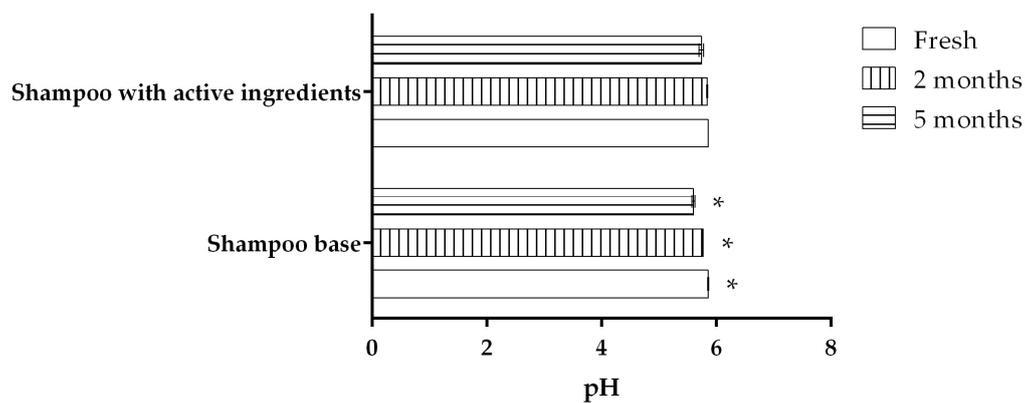
In Figure 1, microscopic evaluation confirmed that the droplet size of shampoo with 1.5% allantoin concentration was over 10–12% higher compared to shampoos with lower allantoin concentrations ( $p < 0.05$ ). Consequently, a shampoo formula containing 1% allantoin was selected for further evaluation, as it is anticipated to showcase superior moisturizing and keratolytic benefits.

#### 3.2. Evaluation of pH

Continuing with the evaluation, the pH levels of both the shampoo base and the formulated shampoo containing allantoin and patchouli extract were assessed. Although there is not a universally defined recommendation for the pH of shampoo, it is crucial to ensure it is suitable for use on the scalp. The scalp's pH is generally around 5.5, and the hair shaft's pH is approximately 3.67. Research by M. Dias et al. suggests that an alkaline pH may increase the negative electrical net charge on the hair fiber surface, leading to increased friction between fibers, potential cuticle damage, and fiber breakage. It is a well-established fact that lower pH in shampoos can contribute to reduced frizz by minimizing negative static electricity on the fiber surface. However, it is imperative that the shampoo ingredients collectively result in a final pH of around 5.5 to prevent any damage to the scalp [21]. On the other hand, AlQuadeib B. et al. suggest that the acceptable pH range for hair shampoo should be 5.0–7.0 [22]. In our research, we attempted to achieve a pH of approximately 5.8 by adding a 50% citric acid solution. The pH levels of the developed shampoos remained mildly acidic and exhibited insignificant variation throughout a span of 5 months (Figure 2).



**Figure 1.** Physical appearance and microscopic evaluation (400× fold) of shampoos containing various concentrations of allantoin: (a) 0.5%; (b) 1.0%; (c) 1.5%.



**Figure 2.** pH variation in shampoo formulations over time. \*  $p > 0.05$  vs. shampoo with active ingredients.

Similar investigations on herbal shampoos, which adjusted the pH to be below 7, also reported stable pH levels over time [18,19].

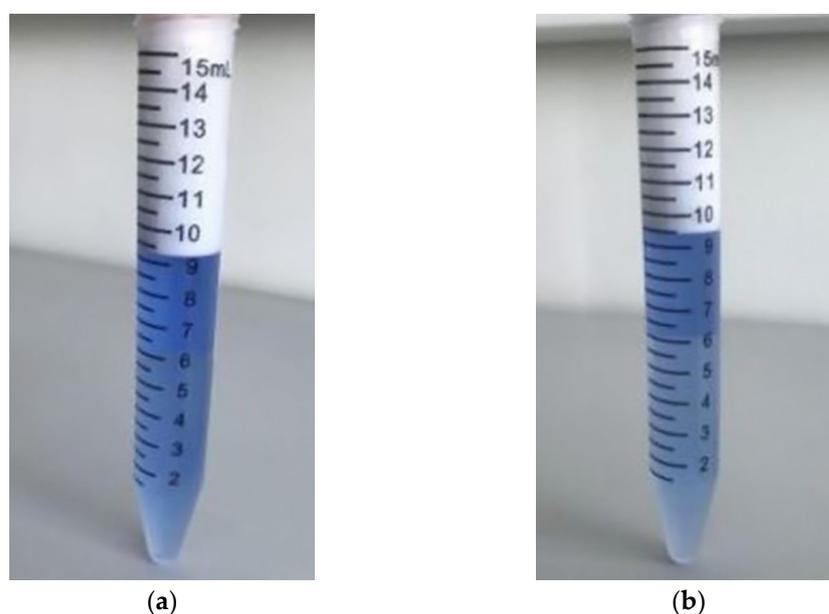
### 3.3. Evaluation of Wetting Time

In the context of our results, we evaluated the wetting time, a critical parameter hinging on surfactant concentration, commonly employed to assess its efficacy. The wetting process involves intricate interactions influenced by factors like surface tension, diffusion, concentration, and the nature of the surface being wetted [6,19,23]. Specifically, the wetting time for the shampoo with active ingredients was  $74.5 \pm 1.025$  s, while the comparison shampoo exhibited a wetting time of  $75.32 \pm 1.459$  s. Despite a slight increase in wetting time, statistical analysis deemed this change to be insignificant ( $p > 0.05$ ). Consequently, the detergency difference between the two samples was considered inconsequential.

Comparatively, existing research indicates that some commercial shampoo brands, assessed using Drave's test, exhibit wetting times ranging from  $12.7 \pm 5.0$  s to  $18.9 \pm 8.2$  s [22]. On the other hand, compositions with natural ingredients, evaluated using a paper canvas test, showed wetting times extending up to  $120\text{--}187 \pm 4$  s [22,24]. These variations underscore the impact not only of the shampoo's chemical composition but also the subtle nuances in the wetting test technique, which can lead to significant changes in wetting time.

### 3.4. Evaluation of Dirt Dispersion

In evaluating shampoo effectiveness, an essential parameter is its ability to disperse dirt. A premium-quality shampoo should efficiently eliminate dirt without allowing its redeposition on the hair. Shampoos that lead to ink accumulation in the foam are generally deemed of poor quality, as it becomes challenging to rinse away and may result in dirt accumulation on the hair [19]. To determine the dirt dispersion capability, both the shampoo with active ingredients and the comparison shampoo underwent testing. Remarkably, no ink was detected in the foam for either shampoo (Figure 3). This observation remained consistent when the test was repeated after 2 and 5 months. The enduring absence of ink in the foam suggests that the formulated shampoos effectively remove dirt without leaving any residue on the hair, aligning with the anticipated standards of high-quality shampoo. In comparing the dirt dispersion of the formulated shampoo with the findings from other researchers, it is evident that all marketed shampoos exhibit no dirt dispersion as well [22,25]. However, according to the data from studies conducted by A. Pradhan and A. Bhattacharyya or T. Malpani, experimental shampoos, particularly those incorporating herbal extracts, often leave light or moderate dirt residue in the foam. This may signal the need for formula refinement before marketing [4,19].



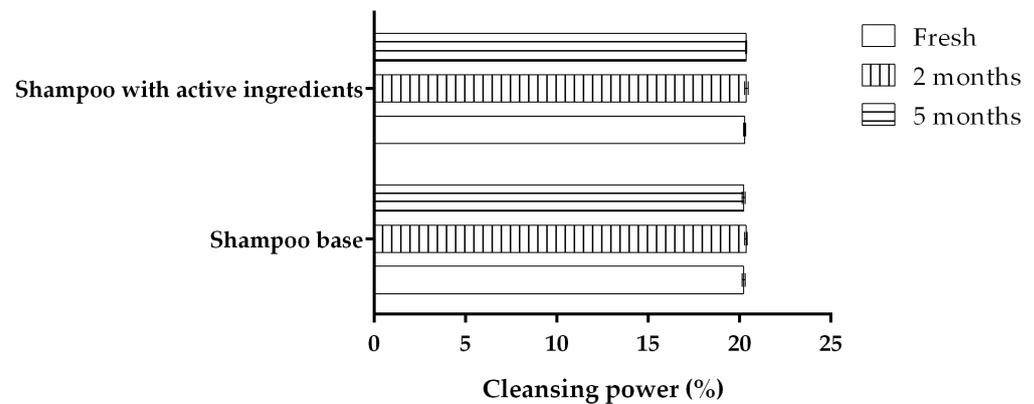
**Figure 3.** View of dispersion test tubes: (a) shampoo base; (b) shampoo with active ingredients.

### 3.5. Evaluation of Detergency Test Results

Moving on to the detergency test, the evaluation focused on one of the most crucial properties: the cleansing power of the shampoo. There is a consensus among cosmetic chemists that a shampoo should not be so powerful that it strips all natural secretions from the hair and scalp. The detergency ability of a normal hair shampoo is expected to be higher than that of dry-hair shampoos but lower than greasy-hair shampoos [3].

Upon analyzing the results of the investigated shampoo samples, it can be concluded that both shampoos exhibit a similar cleansing power, showing no statistically significant difference (Figure 4). The addition of allantoin and Patchoul'Up did not impact the cleansing power of the shampoo. Furthermore, the cleansing power remained statistically unchanged even after periods of 2 and 5 months ( $p > 0.05$ ). In comparison, Dhayanithi et al. found that the cleansing power of some marketed shampoos ranged from 18 to 33% [26], indicating that our samples have a suitable cleaning action. However, in contrast to some other detergency studies, the investigated shampoos showed up to four times lower detergency ability when compared with the formulations assessed in those studies [3,25] ( $p < 0.05$ ). This discrepancy might arise due to the lack of standardization in

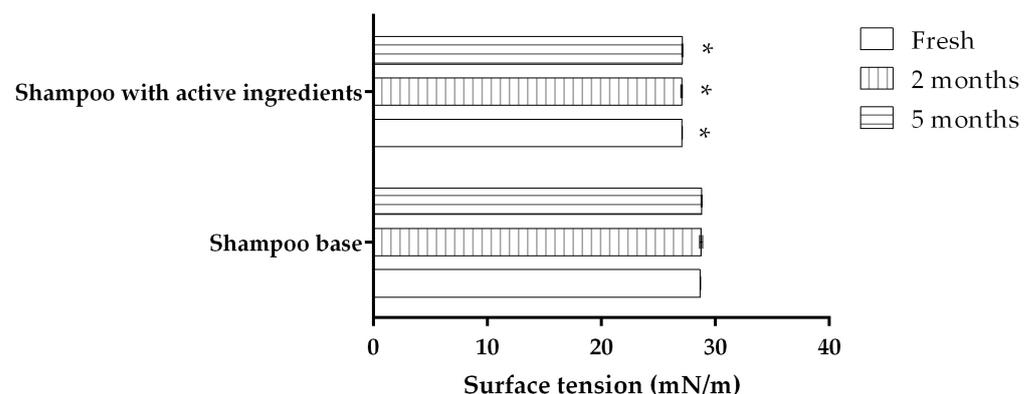
experimental detergency evaluation. Standardizing this process has proven difficult as there is no unanimous agreement on a standard soil, a reproducible soiling process, or the ideal amount of soil a shampoo should remove [3,26].



**Figure 4.** Cleansing power of investigated shampoo samples over time.

### 3.6. Evaluation of Surface Tension

Continued investigation focused on the surface tension of the shampoo samples, and the findings are illustrated in Figure 5. Detergents, which are integral components of shampoos, play a key role in reducing surface tension, thereby enhancing the cleaning efficacy of the shampoo [17]. Numerous studies suggest that an effective shampoo should be capable of decreasing the surface tension of pure water to approximately 32–40 mN/m [4,17]. The surface tension of the shampoo containing active ingredients was found to be 5.87% lower than that of the shampoo base ( $p < 0.05$ ). This difference may be attributed to the inclusion of plant extracts and allantoin in the formulation. It is worth noting that the impact of additives on surface tension can vary; for instance, Farah et al. reported a significant increase in surface tension with the addition of patchouli oil (5%) to a fuel mixture [27]. Conversely, the incorporation of allantoin (0.2%) in bioadhesive gels resulted in a decrease in surface tension of 4.05 to 7.03% [16]. Regarding stability, there was no statistically significant difference in surface tension observed between the shampoos produced immediately and those tested after 2 and 5 months ( $p > 0.05$ ). This suggests that the formulated shampoos maintained consistent surface tension values over the specified time frame, indicating stability in this particular aspect.

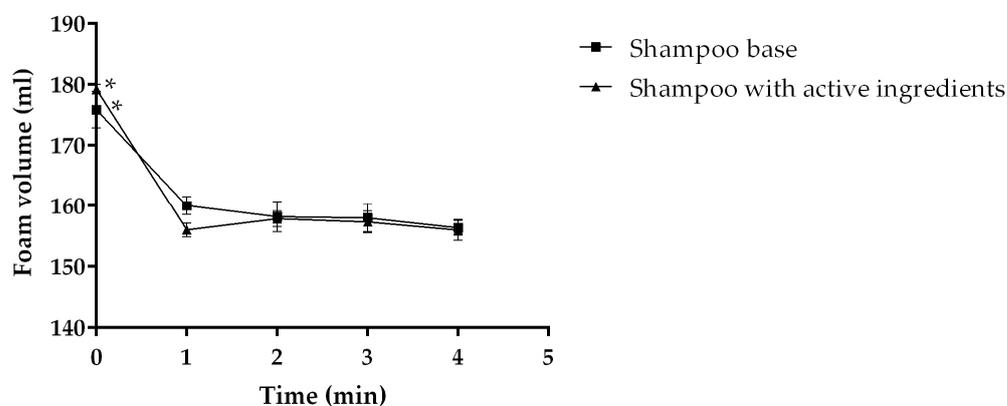


**Figure 5.** Surface tension of investigated shampoo samples over time. \*  $p < 0.05$  vs. shampoo base.

### 3.7. Evaluation of Foaming Ability and Foam Stability

In this section, this study delves into the investigation of foam volume and stability, recognizing the consumer's perception of foam as a crucial factor in the success of a shampoo, even though it does not have a direct correlation with cleansing efficacy [28]. It is expected for the shampoo to lather well and keep it stable for a certain period of time. Also,

foaming ability strongly correlates with the content and type of surface active ingredients added to clean the sebum. The results of the formulated shampoo's foam volume and stability are presented in Figure 6.



**Figure 6.** Foam volume of investigated shampoo samples over time. \*  $p < 0.05$  vs. 1, 2, 3, and 4 min.

The foam volume of the shampoo exhibited a slight 1.9% increase with the addition of active ingredients, which was deemed statistically insignificant ( $p > 0.05$ ). Comparatively, a study involving shikakai and soapnut in shampoo formulation demonstrated enhanced foam characteristics, producing uniform, small-sized, compact, denser, and stable foam in comparison to the control, emphasizing the role of natural saponins found in these plant extracts [24]. A decline in foam volume was observed over a 4 min interval for both the shampoo without active ingredients (up to 11.9%, with a Pearson correlation coefficient of  $-0.8078$ ) and the shampoo with allantoin and patchouli extract (up to 13%, with Pearson correlation coefficients determined to be  $-0.7094$ ). Previous evaluations of marketed shampoos suggested no significant difference in foam volume after 5 min [22]. A. Pradhan and A. Bhattacharyya emphasized that foaming abilities are influenced by factors such as soaking time, temperature, and surfactant properties, contributing to variations in foam volume and stability among different compositions [4].

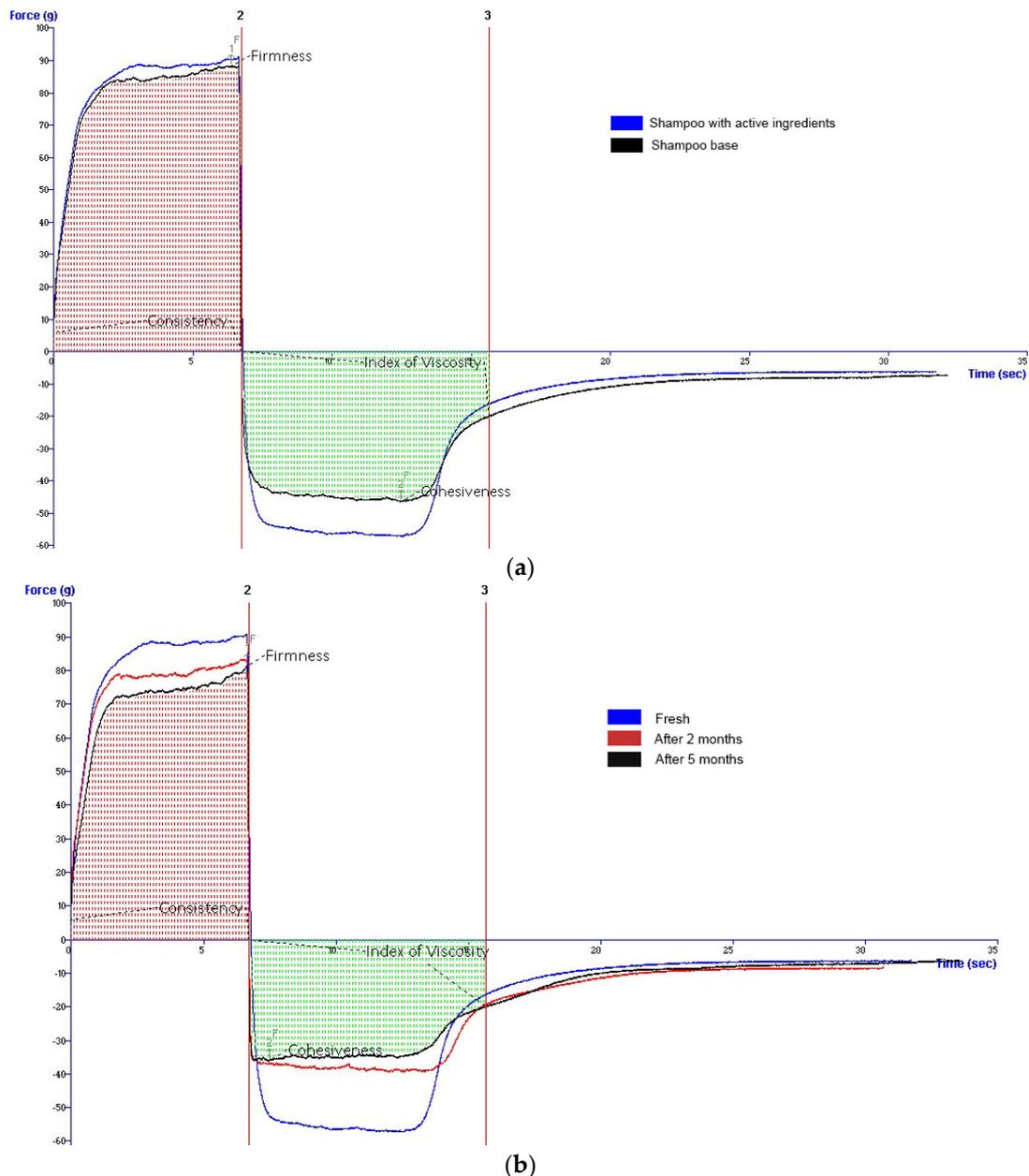
### 3.8. Evaluation of Texture Analysis Results

In this section, we have investigated the rheological properties of the shampoo samples. Texture analysis, a crucial aspect, not only aids in formulating a product that aligns with consumer expectations but also characterizes rheology, a key determinant when assessing the stability of semi-solid preparations. The back extrusion test was chosen, yielding four parameters: firmness (g), consistency (g·s), cohesiveness (g), and viscosity index (g·s). The software of the equipment facilitated graphical data presentation (Figure 7).

For the most part, parameters describing the texture of the shampoo with active ingredients did not exhibit statistically significant differences ( $p > 0.05$ ) when compared to the shampoo without active ingredients. Notably, a more substantial difference was observed in cohesiveness: the addition of active ingredients led to an 18% reduction in cohesiveness, suggesting an enhanced likelihood of breakage ( $p < 0.05$ ). These findings align with our previous studies, where plant materials were shown to not only enhance product quality but also impact technological parameters, including rheology [29].

A comparative analysis of the shampoo's texture with active ingredients over a 5-month period revealed statistically significant differences ( $p < 0.05$ ) in consistency. The formulation immediately tested showed lower consistency after 2 months and 5 months, with reductions of 6.97% and 13.19%, respectively, indicating decreased resistance to flow or deformation and suggesting a less solid and cohesive structure. Concurrent changes were observed in cohesiveness, which increased from  $-56.13 \pm 2.45$  g to  $-36.80 \pm 0.40$  g over the 5-month period ( $p < 0.05$ ). In contrast, firmness exhibited minimal variation over time. A noteworthy 24.25% increase in viscosity index was observed, indicating that the sample's viscosity changed less with temperature variations, a favorable outcome ( $p < 0.05$ ). Com-

paring our results with texture analyses conducted by other researchers, such as L. Silva et al., the incorporation of botanical extracts similarly resulted in shampoos with decreased hardness, firmness, and shear. According to the researchers, this outcome enhances user experience and comfort during shampoo use, attributed to easier spreadability [30]. During the texture analysis, the non-Newtonian pseudoplastic behavior of the investigated samples was observed. This is a desired property of shampoo formulations because it makes it easier to spread and massage the product into the hair and scalp during application. This property enhances the spreadability and sensory experience of the shampoo, providing a smoother and more luxurious feel during use [31].



**Figure 7.** Graphical view of shampoo sample texture analysis: (a) comparison of texture properties between shampoo base and shampoo with active ingredients; (b) changes in the texture properties of shampoo with active ingredients over time.

### 3.9. Evaluation of Consumer Research Results

After formulating and rigorously assessing the shampoo's quality, consumer research was conducted with ten volunteers to evaluate whether it possessed the desired anti-

dandruff properties and was acceptable for the consumers. The research protocol, subjected to the Center of Bioethics, received approval. Volunteers, aged 18 and above, without known allergic reactions to any shampoo ingredients, proficient in English and Lithuanian, were selected. Each participant refrained from using any hair care products 12 h before the experiment, receiving 30 g of a shampoo sample and a questionnaire (Appendix A). Respondents evaluated the formulated shampoo's quality by answering questionnaire questions, and the survey results are described below and presented in Figures 8 and 9.

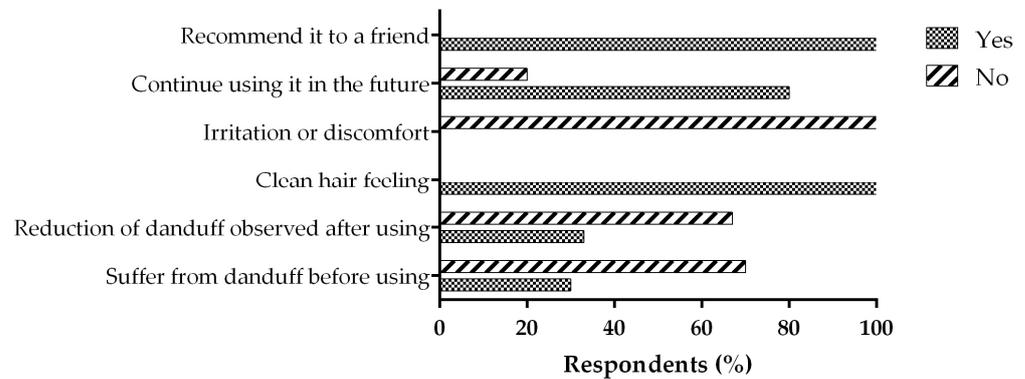


Figure 8. Shampoo evaluation by consumer: graphical questionnaire representation.

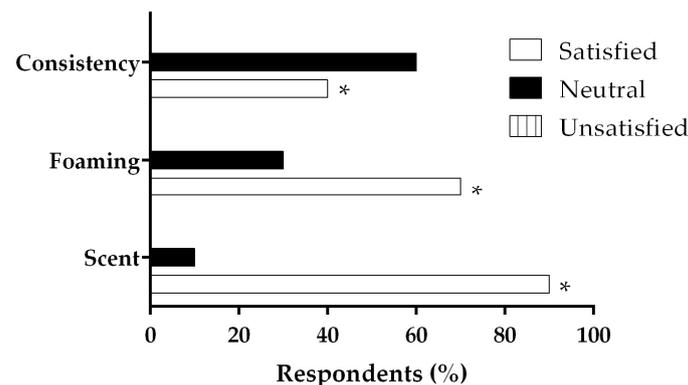


Figure 9. Shampoo sensory properties evaluation by consumer. \*  $p < 0.05$  vs. neutral.

The survey involved 10 participants, comprising 3 males and 7 females. Among the respondents, 30% reported experiencing dandruff, while 70% did not face this issue. Of the three participants with dandruff, only one observed a reduction in dandruff after using the shampoo, while the remaining two did not provide specific feedback on any reduction. As illustrated in Figure 8, an overwhelming 100% of the respondents reported that the shampoo left their hair feeling clean, and they would recommend it to a friend. In a consumer behavior study, it was found that 40% of individuals do not recommend the use of shampoos to others unless they find it genuinely useful [32]. This positive feedback suggests that the shampoo was well received among the surveyed individuals. None of the respondents experienced any irritation from using the shampoo. According to J.S Pahmar, every second respondent believes herbal shampoos are safer than non-herbal ones [32]. Overall, 80% of participants expressed their intention to continue using the shampoo, emphasizing their satisfaction. However, two individuals indicated that they would not continue using it.

In the survey, participants were provided with response options, including 'satisfied', 'neutral', and 'unsatisfied', to assess the scent, foaming, and texture of the shampoo. Figure 9 reveals that a significantly higher number of respondents expressed satisfaction with the scent of the shampoo compared to those who were neutral ( $p < 0.05$ ), with none expressing dissatisfaction with the aroma.

Concerning the lather or foaming of the shampoo, 40% more respondents were satisfied ( $p < 0.05$  vs. neutral), and none reported dissatisfaction. Additionally, 20% more participants adopted a neutral stance on the thickness of the shampoo ( $p < 0.05$  vs. satisfied). Consumer preferences for hair shampoo are influenced by various factors, including usage experience, ethical and eco-friendly attributes, brand preference, product quality, and problem-solving ability [33].

#### 4. Conclusions

Based on the comprehensive analysis of the obtained results, it can be deduced that the shampoo formulation incorporating 0.5% Patchoul'Up™ and 1% allantoin exhibits satisfactory physicochemical properties, including pH, texture, and surface tension. The product also demonstrates favorable characteristics in terms of wetting time, dirt dispersion, foam volume, and cleansing action. The consumer evaluation aligns with the laboratory findings, indicating that the shampoo was well received without causing irritations. Acknowledging the wide phytochemical composition of plant extracts, it is advisable to not only concentrate on their bioactivity but also evaluate the alterations in technological properties that arise upon their addition into the shampoo base. Continuous research is essential to enhance rheological and foam stability while adjusting consistency based on consumer evaluations. Further investigation is warranted to substantiate the anti-dandruff potential by expanding the study to include volunteers specifically experiencing scalp problems.

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**Informed Consent Statement:** Informed consent was obtained from all subjects involved in the study.

**Data Availability Statement:** All data are available upon request.

**Conflicts of Interest:** The authors declare no conflicts of interest.

#### Appendix A

##### QUESTIONNAIRE

The purpose of this survey is to gather feedback on the effectiveness of herbal shampoo targeted for antidandruff. Your participation is voluntary, and all information collected will remain confidential. Your response will be analyzed in aggregate and used only for research purposes. If you agree to participate, please answer the questions honestly to the best of your ability.

By signing below, I acknowledge that I have read and understand the above information.

Signature      Date

1. Gender
  - i. Male
  - ii. Female
2. Do you suffer from dandruff? (If no, go to question 4)
  - i. Yes
  - ii. No

3. Did you observe any reduction in dandruff after using herbal shampoo?
  - i. Yes
  - ii. No
4. How would you rate the scent of the herbal shampoo?
  - i. Satisfied
  - ii. Neutral
  - iii. Unsatisfied
5. How would you rate the lather/foaming of the herbal shampoo?
  - i. Satisfied
  - ii. Neutral
  - iii. Unsatisfied
6. How would you rate the thickness or consistency of the herbal shampoo?
  - i. Satisfied
  - ii. Neutral
  - iii. Unsatisfied
7. Did the herbal shampoo leave your hair feeling clean?
  - i. Yes
  - ii. No
8. Did you experience any irritation or discomfort while using the herbal shampoo?
  - i. Yes
  - ii. No
9. Would you continue to use this shampoo in the future?
  - i. Yes
  - ii. No
10. Would you recommend the herbal shampoo to a friend?
  - i. Yes
  - ii. No

## References

1. Pierard-Franchimont, C.; Xhaufaire-Uhoda, E.; Pierard, G. Revisiting dandruff. *Int. J. Cosmet. Sci.* **2006**, *28*, 311–318. [[CrossRef](#)] [[PubMed](#)]
2. Mainkar, A.; Jolly, C. Formulation of natural shampoos. *Int. J. Cosmet. Sci.* **2001**, *23*, 59–62. [[CrossRef](#)]
3. Fazlolahzadeh, O.; Masoudi, A. Cosmetic evaluation of some Iranian commercial normal hair shampoos and comparison with new developed formulation. *Int. J. Pharmacogn.* **2015**, *2*, 259–265.
4. Pradhan, A.; Bhattacharyya, A. Shampoos then and now: Synthetic versus natural. *J. Surf. Sci. Technol.* **2014**, *30*, 59–76.
5. Halith, S.M.; Abirami, A.; Jayaprakash, S.; Karthikeyini, C.; Pillai, K.K.; Firthouse, P.M. Effect of *Ocimum sanctum* and *Azadiracta indica* on the formulation of antidandruff herbal shampoo powder. *Pharm. Lett.* **2009**, *1*, 68–76.
6. Al Badi, K.; Khan, S.A. Formulation, evaluation and comparison of the herbal shampoo with the commercial shampoos. *Beni-Suef Univ. J. Basic Appl. Sci.* **2014**, *3*, 301–305. [[CrossRef](#)]
7. Muhammad, S.; Utari, R.S.D.; Rahmatullah, M.; Fadhlurrahman, H.; Arie, F.M.; Amanda, T.; Erwan, F.; Lufika, R.D.; Ernawati, E. Innovation of Shampoo Bar From Natural Herbal Essential Oil of Aceh. *J. Patchouli Essent. Oil Prod.* **2022**, *1*, 18–21. [[CrossRef](#)]
8. Pandey, S.K.; Bhandari, S.; Sarma, N.; Begum, T.; Munda, S.; Baruah, J.; Gogoi, R.; Haldar, S.; Lal, M. Essential oil compositions, pharmacological importance and agro technological practices of Patchouli (*Pogostemon cablin* Benth.): A review. *J. Essent. Oil Bear. Plants* **2021**, *24*, 1212–1226. [[CrossRef](#)]
9. Ramya, H.; Palanimuthu, V.; Rachna, S. An introduction to patchouli (*Pogostemon cablin* Benth.)—A medicinal and aromatic plant: It's importance to mankind. *Agric. Eng. Int. CIGR J.* **2013**, *15*, 243–250.
10. Becker, L.C.; Bergfeld, W.F.; Belsito, D.V.; Klaassen, C.D.; Marks, J.G.; Shank, R.C.; Slaga, T.J.; Snyder, P.W.; Andersen, F.A. Final report of the safety assessment of allantoin and its related complexes. *Int. J. Toxicol.* **2010**, *29*, 84S–97S. [[CrossRef](#)]
11. Savić, V.L.; Nikolić, V.D.; Arsić, I.A.; Stanojević, L.P.; Najman, S.J.; Stojanović, S.; Mladenović-Ranisavljević, I.I. Comparative study of the biological activity of allantoin and aqueous extract of the comfrey root. *Phytother. Res.* **2015**, *29*, 1117–1122. [[CrossRef](#)] [[PubMed](#)]

12. Klimek-Szczykutowicz, M.; Szopa, A.; Ekiert, H. *Citrus limon* (Lemon) phenomenon—A review of the chemistry, pharmacological properties, applications in the modern pharmaceutical, food, and cosmetics industries, and biotechnological studies. *Plants* **2020**, *9*, 119. [[CrossRef](#)] [[PubMed](#)]
13. Madhusudhan, M.; Rao, M.K.; Radha, G.; Ganapathy, S. Use of traditional herbs for the formulation of herbal powdered shampoos and their evaluation. *Plant Arch.* **2021**, *21*, 845–856. [[CrossRef](#)]
14. Arora, P.; Nanda, A.; Karan, M. Shampoos based on synthetic ingredients vis-a-vis shampoos based on herbal ingredients: A review. *Int. J. Pharm. Sci. Rev. Res.* **2011**, *7*, 41–46.
15. Olakunle, O.; Joy, B.; Irene, O. Antifungal activity and phytochemical analysis of selected fruit peels. *J. Biol. Med.* **2019**, *3*, 040–043. [[CrossRef](#)]
16. Rathi, S.; Murarkar, K.; Chandak, A. Antimicrobial activity of natural herbal products against dandruff causing fungus and bacteria. *World J. Pharm. Res.* **2019**, *8*, 1460–1467.
17. Singh, A.; Saxena, A. Formulation and Evaluation of Herbal Anti-Dandruff Shampoo from Bhringraj Leaves. *Pharm. Pract. Res.* **2018**, *1*, 5–11.
18. Revansiddappa, M.; Sharadha, R.; Abbulu, K. Formulation and evaluation of herbal Anti-dandruff shampoo. *J. Pharmacogn. Phytochem.* **2018**, *7*, 764–767.
19. Malpani, T.; Jeithliya, M.; Pal, N.; Puri, P. Formulation and evaluation of Pomegranate based herbal shampoo. *J. Pharmacogn. Phytochem.* **2020**, *9*, 1439–1444.
20. Kasparaviciene, G.; Kalveniene, Z.; Pavilonis, A.; Marksiene, R.; Dauksiene, J.; Bernatoniene, J. Formulation and characterization of potential antifungal oleogel with essential oil of thyme. *Evid. Based Complement. Altern. Med.* **2018**, *2018*, 9431819. [[CrossRef](#)]
21. Dias, M.F.R.G.; de Almeida, A.M.; Cecato, P.M.R.; Adriano, A.R.; Pichler, J. The shampoo pH can affect the hair: Myth or reality? *Int. J. Trichol.* **2014**, *6*, 95. [[CrossRef](#)]
22. AlQuadeib, B.T.; Eltahir, E.K.; Banafa, R.A.; Al-Hadhairi, L.A. Pharmaceutical evaluation of different shampoo brands in local Saudi market. *Saudi Pharm. J.* **2018**, *26*, 98–106. [[CrossRef](#)] [[PubMed](#)]
23. Jolly, M. Evaluation of commercial herbal shampoos. *Int. J. Cosmet. Sci.* **2000**, *22*, 385–391. [[CrossRef](#)] [[PubMed](#)]
24. Vijayalakshmi, A.; Sangeetha, S.; Ranjith, N. Formulation and evaluation of herbal shampoo. *Asian J. Pharm. Clin. Res.* **2018**, *11*, 121–124.
25. Krunali, T.; Dhara, P.; Meshram, D.; Mitesh, P. Evaluation of standards of some selected shampoo preparation. *World J. Pharm. Pharm. Sci.* **2013**, *2*, 3622–3630.
26. Dhayanithi, S.; Hoque, E.; Pharm, B.; Pallavi, N.; Pn, K. Formulation and evaluation of herbal shampoo. *Natl. J. Pharm. Sci.* **2021**, *1*, 88–93.
27. Farhan, M.; Gamayel, A. An Addition of Ethanol and Patchouli Oil on Pertamina: The Case Study of Their Effect on Exhaust Gas Emission for 4-stroke Engine. *J. Tek. Mesin Mech. Xplore* **2023**, *3*, 87–95. [[CrossRef](#)]
28. D'Souza, P.; Rathi, S.K. Shampoo and Conditioners: What a Dermatologist Should Know? *Indian J. Dermatol.* **2015**, *60*, 248–254. [[CrossRef](#)] [[PubMed](#)]
29. Cizauskaite, U.; Marksa, M.; Bernatoniene, J. The optimization of technological processes, stability and microbiological evaluation of innovative natural ingredients-based multiple emulsion. *Pharm. Dev. Technol.* **2018**, *23*, 636–645. [[CrossRef](#)]
30. Silva, L.N.; Leite, M.G.A.; Costa, G.M.D.A.; Campos, P.M.B.G.M. Influence of botanical extracts in the texture profile of shampoo formulations. *Int. J. Phytocosmetics Nat. Ingred.* **2020**, *7*, 6. [[CrossRef](#)]
31. Kumar, A.; Mali, R.R. Evaluation of prepared shampoo formulations and to compare formulated shampoo with marketed shampoos. *Evaluation* **2010**, *3*, 025.
32. Parmar, J.S. Customer Behavior on the Purchase Decision and Brand Preference for Shampoos. *Apeejay J. Manag. Technol.* **2013**, *8*, 10.
33. Rawal, N.; Singh, G.S. A Study on Consumer Preference and Satisfaction Towards Various Brands of Hair Shampoo in Ahmedabad City. In *Data-Driven Decision Making for Long-Term Business Success*; IGI Global: Hershey, PA, USA, 2023; pp. 356–368.

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