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Physical stability test of liquid facial soap formulation of citronella extract (*Cymbopogon nardus* L.)

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Abstract

Background: Citronella (*Cymbopogon nardus* L.) is one of Indonesia's most fertile herbal plants. It contains antibacterial compounds and can be used as a liquid facial wash. With the correct formulation, citronella extract facial wash can effectively remove dirt and sebum on the face and prevent the growth of acne-causing bacteria.

Objective: This research aims to obtain the optimal formula for citronella extract liquid facial wash.

Method: Citronella extract was formulated into the liquid facial wash with concentrations of 9, 18, and 24%, and then the physical stability of the preparation was tested for 3 weeks. After that, the results are compared with the standards that have been set.

Results: The citronella extract contained saponins, polyphenols, and flavonoids. The three liquid facial wash formulations made from citronella grass extract with various concentrations had a thick, liquid texture, a unique smell, a brown color, a viscosity range of 948-2,506 cPs, a potential hydrogen range of 4.8–5.97, an adhesive test range of 1.2-2.58 seconds, a foam height range of 0-107 mm, and a spreading power of 5–6.4 cm.

Conclusion: All stability tests for citronella extract liquid facial wash formulations meet the requirements except for the foam height test. Formula 3 is the most optimal formulation, exhibiting a variance in extract concentration of 24%.

Keywords: Citronella grass, liquid facial wash, physical stability test

1. Introduction

Indonesia is situated between the continents of Asia and Australia, bordered by the Pacific Ocean and the Indian Ocean. This geographical location provides Indonesia with a distinct seasonal cycle. The variety of climates, soil types, and environmental factors contributes to Indonesia's diverse ecosystems (Setiawan *et al*., 2022). Indonesia is estimated to possess 25% of the global flora species (Kusmana & Hikmat, 2015). Indonesia's rich biodiversity encompasses 40,000 plant species, with approximately 1,300 utilized for medicinal purposes (Siregar & Ansari, 2020). Medicinal plants are commonly utilized as raw materials for pharmaceuticals or herbal remedies, which can enhance immune function upon consumption. The General Department of Plant Production defines medicinal plants as those utilized for medicinal and cosmetic purposes, derived from various plant parts including fruits, leaves, stems, and rhizomes (Siregar & Ansari, 2020). Tutik *et al*. (2022) demonstrated that the ethanol extract of citronella (*Cymbopogon nardus* L.) inhibits the growth of moderate *Staphylococcus aureus* and *Escherichia coli* bacteria. Another study by Winato *et al*. (2019) demonstrated that citronella (*C. nardus* L.) extract is capable of inhibiting the growth of *Propionibacterium acnes* bacteria. *Staphylococcus aureus*, *S. epidermidis*, and *P. acnes* are commonly implicated in acne infections.

Citronella plants possess the potential to serve as a source of raw materials for the development of essential oils. Citronella oil contains main components such as citronellal, citronellol, and geraniol (Sefriyanti *et al*., 2020). It is often used in the world's soap, perfume, cosmetics, and flavoring industries (Wijayati *et al*., 2023). In addition to its applications in these industries, citronella oil is also recognized for its natural insect-repelling properties, making it a popular choice for outdoor products and repellents. As demand for natural and sustainable ingredients continues to rise, the cultivation and processing of citronella plants present significant opportunities for growth in the essential oils market.

While the research by Rinaldi *et al*. (2021) has established the formulation of citronella ethanol extract liquid soap, this study focuses on an alternative formulation and kind of facial soap. To guarantee the product's quality, purity, and safety, conducting physical stability testing is essential. The analysis assesses the consistency of many factors, such as color, odor, and physical properties, during the storage duration (Sayuti, 2015; Allen & Ansel, 2014). This study will develop a liquid facial wash using citronella extract and assess its physical stability.

2. Method

2.1. Extraction and phytochemical screening

The citronella plants (*C. nardus* L.) were collected from Tlogowungu District, Pati Regency, Central Java. It weighed as much as 2,000 grams in a wet form that has been cleaned, dried, ground with a blender, and sieved using mesh no. 40 for homogenous simplicia powder. The extraction procedure employed the maceration method using 96% ethanol. Prior to extraction, the simplicia was measured for its moisture (water content) to ensure the dryness and avoid microorganism contamination. The phytochemical identification was conducted using several reagents to observe the color change.

2.2. Formulation of liquid facial soap loaded with citronella extract

Facial soap preparations are cleansing soaps specially formulated for healthy facial skin to safely remove oil and dirt from the surface of the facial skin (Renata & Soeyono, 2017). The formula used in this study is derived from the formulation by Yuniarsih *et al*. (2020), which creates a facial wash gel using *Hylocereus polyrhizus* extract and the gelling agent carbopol, along with an evaluation of its physical properties. In this research, citronella extract becomes the main active ingredient with various concentrations as follows: The concentration of citronella extract is 9% in the first formula (F1), 18% in the second formula (F2), and 24% in the third formula (F3).

Other materials to produce facial wash are ethylene diamine tetra acetic acid as a chelating agent, glycerin as a wetting agent, sodium lauryl sulfate as a foaming agent, propylene glycol as a solvent, nipagin as a preservative, carbopol as a gelling agent, triethanolamine as an alkali-forming agent, citric acid as a buffer-forming agent, and distilled water as a solvent. The initial step of liquid facial wash formulation involves the mixing of the solution, achieved through stirring, which consists of a blend of ethylenediamine tetra acetic acid and glycerin with distilled water. Subsequently, nipagin is incorporated, dissolved, and combined with propylene glycol. Once the mixture is deemed to be adequately formed, it is subjected to heating until it reaches a temperature of 40 degrees. Sodium lauryl sulfate was introduced gradually until a uniform mixture was achieved. Afterwards, citronella extract and citric acid were incorporated into the combination. The mixture was agitated until a uniform solution is attained. Carbopol is introduced gradually until a homogeneous mixture is achieved. Then, triethanolamine was incrementally introduced, maintaining the stirring process until a uniform mixture is attained (Yuniarsih *et al*., 2020). The formulation of liquid facial wash was then tested for its physical stability including organoleptic, viscocity, pH, foam height, adhesion, and spreadibility.

2.3. Data analysis

The SPSS software for Windows was applied in order to process the data in accordance with statistical principles. The procedure of analysis included the normality test (Shapiro-Wilk), which regarded the data to be normal if the *p*-value was more than 0.05, and the homogeneity test (Levene), which considered the data to be typical if the *p*-value was greater than 0.05: both of these tests were performed. A correlation analysis was performed on the outcomes of each test in this study to determine how well they correlated with each formula.

3. Result and discussion

3.1. Plant determination

The citronella plants (*C. nardus* L.) was determined at the Biology Laboratory of Ahmad Dahlan University with letter number 084/Lab.Bio/B/II/2024. Determination was carried out to determine the truth of the plant based on its morphological characteristics with the results as follows:

1b – 2b – 3b – 4b – 12b – 13b – 14b – 17b – 18b – 19b – 20b – 21b – 22b – 23b – 24b – 25b – 26b – 27b – 799b – 800b – 801b – 802a – 803b – 804b – 805c – 806b – 807a – 808a *Poaceae* 1b – 10b – 11b – 12b – 13b – 19a – 20a – 21b – 57b – 72b – 74b – 75b – 80a – 81b *Cymbopogon* 1b – 3b – 5a *Cymbopogon nardus* (L.) Rendle.

3.2. Citronella extract

The maceration process of citronella plant resulting an extra thick and greenish brown extract. **Table 1** displays the weight measurements of the citronella plant (*C. nardus* L.). **Table 2** shows the weight of the extract and the yield of the citronella (*C. nardus* L.) ethanol extract.

Table 1. Weight extraction simplicia citronella plant (*C. nardus* L.)

Sample	Weight before drying (g)	Weight after drying (g)
Citronella plant (*C. nardus* L.)	2000	800

Table 2. Weighting results extract and yield extracts citronella plant (*C. nardus* L.)

Sample	Weight of thick extract (g)	Yield (%)
Citronella plant (*C. nardus* L.)	53.20	10.64

A higher yield value indicates more effective and efficient extraction, as well as an increased concentration of bioactive components in the extract. In accordance with the current standard, the yield extract exceeds 10% (Kemenkes RI, 2017).

3.3. Water content test results

The moisturizer analyzer tool was used to determine the water content of citronella simplicia as shown on **Table 3** for the result. This outcome signifies that the simplicia was of high quality and adequately desiccated, as its moisture content was below 10% (Utami *et al.*, 2020). Controlling the water content is important because insufficient water in the simplicia does not provide a suitable medium for microorganism growth.

Table 3. Water content test results of citronella extract (*C. nardus* L.)

Simplicia	Water content (%)
Citronella (*C. nardus* L.)	6.51

3.4. Phytochemicals screening

The phytochemical analysis of citronella extract (*C. nardus* L.) indicated the presence of flavonoids, polyphenols, and saponins, as outlined in **Table 4**. The saponin assay on the citronella plant extract produced positive outcomes, evidenced by the formation of stable foam in the extract solution. The appearance of foam is attributable to the saponin content, which is soluble in polar solvents, and the presence of chemicals that dissolve in non-polar solvents. Compounds with polar or non-polar groups exhibit surface-active properties. Saponin can form a micelle when concurrently dissolved with the solvent. A micelle structure forms as polar groups orient towards the outside. Simultaneously, the non-polar group orients towards the interior. This can be noticed as foam (Triwahyuni *et al.*, 2019).

Analysis of the polyphenols in citronella extract yielded favorable results, characteristic of a blackish-green solution formation. In a polyphenol assay with $FeCl_3$ reagent, polyphenols can liberate

H^+ ions and generate phenoxy ion compounds, which subsequently interact with $FeCl_3$ to produce an iron (III) hexaphenolate complex (Rismawati *et al.*, 2018).

Table 4. The result of phytochemical screening of citronella extract

Compound	Reagent	Discoloration		Conclusion
		Beginning	End	
Flavonoid	Mg + HCl powder	Brown	Brick red	Positive
Polyphenols	$FeCl_3$	Brown	Blackish green	Positive
Saponins	Aquadest	Brown	Foamy brown	Positive

3.5. Stability test of liquid facial soap with citronella extract

The physical stability of the liquid facial soap formulation was assessed over a period of three weeks, with observations conducted weekly. The tests comprised organoleptic properties, adhesion, foam height, viscosity, spreadability, and pH level. The preparation can be deemed stable if there is no substantial variation in the observed findings of the parameters each week, with a reference based on a *p*-value greater than 0.05.

3.5.1. Organoleptic test

The organoleptic testing aims to determine whether the liquid facial soap preparation meets the desired criteria. The parameters assessed in this test include odor, color, and shape. Organoleptic testing itself is carried out using the five human senses. The results of the organoleptic test of the liquid facial soap preparation with citronella extract can be seen in **Table 5**.

Table 5. Organoleptic test results of liquid facial soap with citronella extract (*C. nardus* L.)

Formulation	Parameter	Results
Basis control	The colors that emerge The emergence smell Form object	Transparent white There isn't any thick
F1	The colors that emerge The emergence smell Form object	chocolate Special extract Thick
F2	The colors that emerge The emergence smell Form object	Chocolate Special extract Thick
F3	The colors that emerge The emergence smell Form object	Chocolate Special extract Thick

The results obtained from this organoleptic test vary. The citronella extract at the basis control is colorless or transparent, viscous, and devoid of scent. The F1-F3 formulations exhibit a brown color, possess a dense texture, and emit a characteristic fragrance; as the color intensifies and becomes more concentrated, the aroma correspondingly strengthens. The intensity of the extract's color and scent increases with greater usage (Doloksaribu & Fitri, 2019). The Indonesian national standard mandates that liquid soap possess a liquid consistency, as well as a unique fragrance and color. The results of the preparations conform to the specifications, specifically liquid consistency, unique aroma, and characteristic color of citronella extract.

3.5.2. Viscosity test results

This test aims to assess the prepared liquid face wash. The viscosity of a formula has stringent terms and conditions, and it is recommended to be neither too thick nor too liquid. The requirements for a good viscosity value range from 500 to 20,000 cPs (Gunarti, 2018). The viscosity test was performed using an assisted Brookfield viscometer with adjustments to the spindle and speed. Viscosity is strongly correlated with the concentration of the gelling agent, and in this study, carbopol was chosen since it can act as a gel base with limited safety when applied topically, as it will not trigger a reaction like hypersensitivity in the skin area (Tambunan & Sulaiman, 2018).

Table 6. Viscosity test results of citronella extract liquid facial soap *(Cymbopogon nardus* L.)

Liquid facial soap	Viscocity (week)			Mean ± SD
	1	2	3	
Basis control	1,556	948	905.3	1,136.44 ± 315.8
F1	2,427.3	1,143.3	1,098.67	1,556.44 ± 654
F2	2,234.6	2,312	1,133.3	1,893.33 ± 571.04
F3	2,506	2,410	2,354.6	2,423±70.85

Note: Viscosity meets the requirements with the value between 500-20,000 cPs (Gunarti, 2018)

Results from the viscosity test conducted over three weeks on the four formulations indicate, as presented in **Table 6**, that the facial soap preparation exhibited a reduction in viscosity value. Environmental factors in gel storage can lead to a decrease in viscosity at varying temperatures, thereby impacting the viscosity of the preparation. The viscosity measurements obtained meet the standard requirements for satisfactory preparations.

The analysis of viscosity test data indicates that the normality test yields a *p*-value greater than 0.05. The homogeneity test indicates results with $p < 0.05$, suggesting that the condition for normality is not met, as a *p*-value greater than 0.05 is required for normality. The one-way ANOVA test necessitates homogeneity of data, as indicated by Palupi & Prasetya (Palupi & Prasetya, 2022). The Kruskal-Wallis test serves as an alternative when the parametric test does not meet normality assumptions. The analysis results indicate a *p*-value of less than 0.05, suggesting significant differences in the data.

3.5.3. pH test results

The measurement of pH (hydrogen potential) seeks to evaluate the level of acidity. Products that disrupt pH equilibrium may result in skin irritation, manifesting as dryness (when pH is excessively alkaline) or redness and inflammation (when pH is excessively acidic). The potential hydrogen in liquid facial soap can markedly elevate the hydrogen potential on the skin, particularly on the face. Consequently, it can facilitate the proliferation of bacteria that instigate the onset of acne. To avert skin irritation, the production of facial soap must establish a pH comparable to its natural acidic state, approximately 4.5 to 6.5(Marhaba *et al*., 2021).

Table 7. pH test results of citronella extract liquid facial soap (*Cymbopogon nardus* L.)

Liquid facial soap	pH (week)			Mean ± SD
	1	2	3	
Basis control	5.97	5.66	5.63	5.76 ± 0.16
F1	5.67	5.42	5.30	5.47 ± 0.16
F2	5.27	5.04	5.02	5.11 ± 0.12
F3	5.09	4.96	4.8	4.96 ± 0.12

Note: pH meets the requirements with hydrogen potential value of 4.5-6.5 (Marhaba *et al*., 2021)

According to the pH test results of the liquid facial soap formulation including citronella extract (**Table 7**) indicates that the findings conform to the hydrogen potential standards for liquid facial soap, which range from 4.5 to 6.5.

3.5.4. Foam height test results

The purpose of the foam height test is to determine the preparation's ability to produce foam. The Indonesian National Standard sets a liquid soap foam height of between 12-220 mm. According to Yuniarsih *et al*. (2020), there is no specific regulation regarding the foam height limit for facial cleansing preparations; instead, the aesthetic appeal derived from the product's foaming ability can capture consumers' attention.

Table 8. Results of the foam height test of citronella extract liquid facial soap (*Cymbopogon nardus* L.)

Liquid facial soap	Week	Beginning	End	Stability (%)
Basis control	1	0.4	0.3	74.6
	2	0.2	0.1	49.8
	3	0.2	0.1	49.8
	Average	0.26	0.17	62.6
F1	1	0.5	0.4	80
	2	0.3	0.2	66
	3	0.3	0.2	66
	Average	0.37	0.27	73
F2	1	1.3	1.2	91
	2	1.1	1	89
	3	0.6	0.5	83%
	Average	1	0.9	89%
F3	1	1.4	1.3	91.4%
	2	1.3	1.2	91%
	3	0.8	0.7	86.7%
	Average	1.17	1.07	90.2%

Note: The Indonesian national standard for height of liquid soap foam is between 12-220 mm (Maharani *et al*., 2021).

Upon analyzing the outcomes derived from the three formulations, it is evident that only F3 fulfills the criteria for an acceptable foam height. This demonstrates that an increased concentration of extract is associated with a greater foam height, given that each formula contains a consistent amount of sodium lauryl sulfate, which is incapable of emulsifying citronella extract (Maharani *et al*., 2021). In the F1 and F2, the value fails to satisfy the necessary criteria. The variation factors that may influence foam height include the composition of materials, stirring speed, temperature, and the duration of mixing, as well as the type of active substance involved.

3.5.5. Adhesion test results

The adhesion test is carried out to determine how much the preparation can adhere to the skin within a specific time. Gel is considered good if it exhibits high adhesion. The higher the adhesion, the better, because the active substance can be absorbed evenly and diffuse maximally. The data from the adhesive power test indicated that the gel form had an influence on the adhesive power. The thicker the dosage form, the greater the adhesive power value. All results obtained meet the standard requirements of the preparation, which exceeds 1 second.

Table 9. Adhesion test of liquid facial soap with citronella extract (*Cymbopogon nardus* L.)

Liquid facial soap	Adhesion (week)			Mean ± SD
	1	2	3	
Basis control	1.77	1.66	1.2	1.55 ± 0.27
F1	2.02	1.75	1.25	1.68 ± 0.34
F2	2.15	1.85	1.32	1.78 ± 0.37
F3	2.58	2.31	1.81	2.23 ± 0.34

Note: The adhesion test meets the requirements of the adhesion time is > 1 second (Rosari *et al.*, 2021)

The results of data analysis using the Kruskal-Wallis test showed results in the first week of 0.044 ($p < 0.05$), the second week showed results of 0.027 ($p < 0.05$), and the third week showed results of 0.027 ($p < 0.05$), which means that there is a difference in the data every week.

3.5.6. Spreadability test results

The spreadability test is performed to assess the distribution ability of the formulation during application, adhering to a standard of 5 to 7 cm. This characteristic is associated with viscosity; increased viscosity results in less spreadability. Increased spreadability facilitates the preparation's penetration into the skin, enhancing its efficacy (Rosari *et al.*, 2021).

Table 10. The spreadability test results of citronella extract liquid soap (*Cymbopogon nardus* L.)

Liquid facial soap	Spreadibility (week)			Average
	1	2	3	
Basis control	5.2	5.5	6.0	5.63 ± 0.35
F1	5.0	5.3	5.6	5.34 ± 0.29
F2	5.5	5.9	6.4	5.98 ± 0.38
F3	5.1	5.3	5.4	5.28 ± 0.15

Note: The spreadability test meets the requirements ranging from 5-7 cm (Rosari *et al.*, 2021).

The outcomes of the spreadability assessment for the citronella extract liquid facial soap formulation improved weekly, however it consistently remained within the appropriate spreadability range. The weekly increase may be attributed to inconsistent storage temperature and agitation, leading to a reduction in viscosity (Rosari *et al*., 2021). The findings of the data analysis employing the Kruskal-

Wallis test indicated that each week's results were < 0.05, signifying a difference in the data on a weekly basis.

4. Conclusion

The stability test of the physicochemical properties of soap formulations loaded with liquid citronella extract (*Cymbopogon nardus* L.) accross three formulations meets the following criterias: organoleptic properties, viscosity, pH value, stickiness, and spreadability. In the foam height test, only formula 3 (F3) meets the standard requirement for good foam height, which is between 12 and 220 mm.

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