Formulation and Evaluation of the Physical Stability of Liquid Soap Preparations with Sonneratia caseolaris Leaf Extract

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ABSTRACT

Red pidada leaf (Sonneratia caseolaris L.) is a plant that has benefits as an antioxidant with active compounds such as alkaloids, flavonoids, tannins, terpenoids, and saponins. Usually the local community uses this plant as a wet pupur. The purpose of this study was to determine whether red pidada leaves can be formulated into a liquid soap preparation that meets the requirements of physical properties and stability evaluation. This type of research is true experimental laboratory. Extraction of red pidada leaves is carried out by maceration using ethanol as a solvent. The formulation used three extract concentrations, namely F1 (2%), F2 (4%), and F3 (8%). Parameters carried out in the form of physical properties testing, including organoleptic test, pH, specific gravity, viscosity, foam resistance and stability testing with the cycling test method. The results of organoleptic physical properties testing on the three formulations showed that the F1 preparation was light brown, F2 brown, and F3 dark brown, had a distinctive aroma and had a liquid consistency in F1 and slightly thick in F2 and F3. Testing of pH, specific gravity, viscosity and foam resistance in the three formulations has met the requirements, except for the specific gravity test in the F3 preparation. Testing with the cycling test method has affected the evaluation of physical properties in the three formulations which causes a very thick consistency in the preparation. So that the tests that meet the requirements are only the pH and foam resistance tests, while the specific gravity is only in the F1 preparation.

Keywords: *Formulation, Sonneratia caseolaris Leaf, Liquid Soap, Physical Properties, Stability*

INTRODUCTION

Soap is one form of cosmetic product. Soap varies widely in type, color, scent, and the benefits it offers, including liquid bath soap. It is often preferred because it is easy to use and suits common habits. In addition to cleansing, soap can also help maintain skin health better than other forms like ointments or creams, which may require more effort to apply. However, natural-based soaps are still rare, as most soaps use synthetic ingredients that can cause irritation, especially for sensitive skin.

Sonneratia caseolaris (pidada red) is a natural ingredient with potential for skincare. Its leaves are commonly used by the people in Pagatan, South Kalimantan, as a cold powder to treat chickenpox, acne scars, and brighten the skin. Literature reveals that the leaves contain alkaloids, flavonoids, tannins, terpenoids, and saponins, which offer antioxidant, antimicrobial, and anti-inflammatory benefits. Other research even suggests that red pidada leaves can be developed as a sunscreen.

Despite its potential, no study has yet developed red pidada leaves into a soap product. Therefore, the authors aim to formulate liquid bath soap from red pidada leaf extract and evaluate its physical stability.

METHODS

This study was conducted through laboratory experiments, including the collection of red mangrove (pidada merah) leaves, extract preparation, formulation of liquid bath soap, and testing of the physical properties of the preparation such as organoleptic tests, pH test, specific gravity test, viscosity test, foam stability test, and stability test using the cycling test method. The test results were processed into descriptive tables and analyzed using one-way ANOVA.

Tools and Materials

The tools used in this study included a blender, sieve (mesh no. 40), analytical balance, filter paper, porcelain dish, water bath, wooden clamps, aluminum foil, stirring rod, metal/ wooden spoon, pipette/ filler/ measuring pipette, hotplate, mixer, pH stick, pycnometer, stand, thermometer, Brookfield viscometer, refrigerator, oven, and standard laboratory glassware (Iwaki).

The materials used in this study were 96% ethanol, red mangrove leaf extract, ice cubes, olive oil, 30%

KOH, sodium carboxymethylcellulose (Na-CMC), sodium lauryl sulfate (SLS), glycerin, stearic acid, citric acid, and distilled water.

Preparation of Red Mangrove Leaf Simplicia

A total of 2 kg of red mangrove leaves were collected and subjected to wet sorting by removing dirt and unnecessary parts, followed by washing with running water and draining. The leaves were then cut into pieces and dried under direct sunlight covered with black cloth [8, 9].

Preparation of Red Mangrove Leaf Extract

The extract was prepared using the maceration method. Maceration was carried out using 96% ethanol as a solvent in a 1:5 ratio. About 488–500 grams of powdered simplicia were placed in a glass jar, mixed with 2.5 liters of solvent, and soaked for 3 days with occasional stirring, then filtered. The resulting macerate was filtered and concentrated using a water bath until a thick extract was obtained.

Formulation of Liquid Bath Soap

The liquid bath soap was prepared in several stages. First, a mixture of olive oil and 30% KOH was heated on a hotplate while being stirred with a mixer until it formed a homogeneous paste-like mixture, which was then left to stand for approximately 3 days (mixture 1). After 3 days, melted stearic acid (preheated in a water bath at 90℃) was added and stirred until homogeneous (mixture 2). Next, Na-CMC dissolved in distilled water was added, followed by glycerin, and stirred until homogeneous. The Na-CMC and glycerin mixture was then added to mixture 2 and stirred to homogenize (mixture 3). SLS was dissolved in distilled water, citric acid was added, and the mixture stirred until homogeneous. Finally, the SLS and citric acid mixture was added to mixture 3 and stirred to a uniform consistency. The red mangrove leaf extract was added at the final step according to the specified concentrations, then stirred until homogeneous and transferred into suitable containers. The formulation of the red mangrove leaf extract liquid bath soap is shown in Table 1.

Tabel 1. Formulation of Liquid Soap

Formula	Function	Consentration (%)			
		F0	F1	F2	F3
Sonneratia caseolaris Extract	Active Ingredient	-	2	4	8
Olive Oil	Oil Phase	15	15	15	15

Potassium Hidroxide 30%	Alkalizing agent	8	8	8	8
CMC-Na	Gelling agent	0.4	0.4	0.4	0.4
SLS	Emulisifer	3	3	3	3
Glyserin	Humectan	3	3	3	3
Stearic Acid	Emulsifier	0.5	0.5	0.5	0.5
Citrate Acid	Preservative	0.3	0.3	0.3	0.3
Aquadest	Solvent	ad 100 ml	ad 100 ml	ad 100 ml	ad 100 ml

Evaluation of the Preparation

1. Organoleptic Test

The organoleptic test was conducted using human senses to observe the appearance, color, and odor of the preparation.

2. pH Test

The pH test was performed to determine the pH of the liquid bath soap by fully dipping pH indicator paper into the sample, observing the resulting color change, and matching it with the scale on the pH meter [10].

3. Specific Gravity Test

The pycnometer was cleaned by rinsing with acetone, then dried and weighed. The sample was cooled in the pycnometer submerged in ice water and allowed to reach 25℃, then filled to the calibration line. The pycnometer was then removed from the ice bath, allowed to reach room temperature, and weighed again [11].

4. Viscosity Test

The viscosity of the liquid soap formulation was measured using a Brookfield viscometer with spindle no. 4 at a speed of 30 rpm [12].

5. Foam Stability Test

Foam stability testing was performed by placing 1 ml of the preparation into a measuring cylinder and adding distilled water until the volume reached 10 ml. The cylinder was shaken by inverting it, and the foam height formed was measured. The cylinder was then left to stand for 5 minutes, and the foam height was measured again after the interval [11].

6. Cycling Test

The cycling test is an accelerated stability test conducted by storing the preparation at a cold temperature (4–8℃) for 12 hours, then transferring it to an oven at 40℃ for another 12 hours. This process counts as one cycle. The test was carried out for six cycles, and the preparation was evaluated at each cycle [12].

RESULTS

From 2 kg of fresh red pidada leaves, 488 grams of simplicia were obtained. Using the maceration method, 80 grams of thick extract were produced, yielding 16%. Evaluation of physical properties and stability using the cycling test method was performed on organoleptic properties, pH, specific

gravity, viscosity, and foam stability.

a. Organoleptic tests

The results of the organoleptic test of the liquid bath soap show differences in shape and color as the concentration of the extract increases.The color becomes more intense with increasing concentration. Soap without extract (blank) and with 2% extract has a liquid consistency, while 4% and 8% extracts are thicker.The soap with extract has a distinctive aroma of red pidada leaves, while the base has a base aroma. Although there were organoleptic changes in each extract variation, the liquid bath soap formulation before the cycling test still met the requirements.This is in line with Muthmainnah's (2014) research on basil oil liquid soap, which also showed an increase in viscosity in accordance with the concentration of active ingredients [14].

The results of the organoleptic test after the cycling test on the liquid soap showed changes in shape and color over the cycles.In the 3rd cycle, the soap with the extract became very thick, so other physical property tests such as pH test, specific gravity test, viscosity test, and foam stability test were stopped at the 2nd cycle.On the other hand, the soap without extract did not undergo significant changes.The aroma of the soap remains the same until cycle 3.This is in line with Taufiq's (2019) research on liquid soap with cherry leaf extract, which also showed changes in viscosity with variations in storage temperatures, both cold and hot [15].

pH Test Based on the observation results of pH measurement on the four liquid soap preparations before and after the cycling test, they have the same pH values, namely the blank preparation (without extract) has a pH value of 10, 2% extract has a pH of 9, and 4% and 8% extracts have a pH of 8.This has met the requirements for the pH test, as according to SNI (2017), the good pH value for liquid bath soap falls within the range of 4-10 [16]. The decreasing pH value in line with the increasing concentration of the extract is consistent with the research results by Mahdi (2022) on antiseptic liquid soap with kapul fruit extract [17]. From the pH test results, it can be concluded that the pH of the liquid bath soap formulation, whether using extract or not, produces a soap that is alkaline in nature. This is due to the strong alkaline nature of KOH, which

affects the pH of the soap, making it alkaline.The pH value indicates the acidity level of a substance, and this pH can affect the skin's absorption capacity, potentially causing irritation. Therefore, the liquid soap that is made must be adjusted to the skin's pH.

b. Density Test

Based on the results of the specific gravity test on the liquid soap formulation, namely in the blank preparation (without extract), the 2% and 4% extract concentrations have met the specific gravity test requirements, which are not less than or exceeding 1.01-1.10 g/ml [18].In the preparation with an 8% extract concentration, it does not meet the requirements because it has a value of 0.97 g/ml, which is less than 1.01 g/ml.Therefore, the researchers can assume that the high concentration of red pidada leaf extract affects the formulation of liquid bath soap, causing a decrease in specific gravity.This is in line with Hutaruk's (2020) research on liquid soap with celery herb extract, where the higher the concentration of the extract, the more it will affect the specific gravity value [19].The specific gravity value of a substance can be influenced by its constituent components and physical properties.When a substance is dissolved in water and forms a solution, its density will change. Generally, substances like sugar and salt increase density, but density can also decrease if there is fat or ethanol in the solution [20]. Based on the results of the specific gravity test after the cycling test on the liquid soap formulation with a 4% extract concentration, there has been a gradual decrease in specific gravity values, reaching up to cycle 2, which caused the soap preparation with that extract concentration to not meet the requirements because it was less than 1.01 g/ml, while the preparation had a value of 1.00 g/ml.This can occur due to the increase in viscosity during the cycling test cycle, which causes a decrease in the specific gravity value of the liquid soap preparation with that concentration.

c. Viscosity Test

Based on the results of the viscosity test on the four liquid soap formulations, it can be observed that there was an increase in thickness with the higher concentration of red mangrove leaf extract. Nevertheless, the viscosity test results still met the

requirements, which range between 400–4000 cP [21]. This is in line with the study by Rasyadi (2019) on liquid soap made with cardamom fruit extract, which stated that the higher the extract concentration, the higher the viscosity [22]. Viscosity testing is a measurement of the thickness of a liquid (fluid), indicating the level of internal friction. The viscosity level of liquid soap can affect consumer preferences as well as the choice of appropriate packaging [21].

Based on the results of the viscosity test after conducting the cycling test on the liquid soap formulations containing all concentrations of red mangrove leaf extract, it was found that there was a very drastic increase in viscosity, particularly up to cycle 2. In cycle 1, the 8% extract concentration did not meet the requirement as it exceeded the limit of 4000 cP. However, other formulations such as the base (without extract), 2%, and 4% extract concentrations still met the requirements. Although there was an increase in viscosity, it was not significant. In cycle 2, a drastic increase in viscosity occurred in all liquid soap formulations containing red mangrove leaf extract, exceeding the viscosity limit of 4000 cP. However, the base formulation (without extract) still met the requirement and did not show a significant increase in viscosity. The factor that may influence this increase in viscosity could be the addition of a high concentration of plant extract, which may increase the cohesion force in the liquid soap [23]. The increase in viscosity is also related to particle size during storage. During storage, particles tend to aggregate to reduce their surface area, forming larger particles with smaller surface area, which causes an increase in viscosity [24]. Viscosity of the product also increases with higher KOH concentration, longer processing time, and higher operating temperatures [25]. The viscosity value of each vegetable oil used can also influence the overall viscosity, as each oil has different characteristics, especially in terms of saturated fat content. The higher the saturated fat content, the lower the viscosity value, whereas lower saturated fat content results in a higher viscosity value. In VCO, the saturated fat content consists of lauric acid at 44.0–52.0%, while in olive oil, the saturated fat content consists of

palmitic acid at 7.5–10.5% [26].

d. Foam Stability Test

Based on the results of the foam stability test of the four formulations before and after the cycling test—namely, the base formulation (without extract), and formulations with 2%, 4%, and 8% extract—the requirements were met, and no significant changes occurred. The requirement for foam stability or foam weight loss during testing should not be less than 60–70%, while the liquid soap formulations tested had a range of 80–90%. Unlike the previous tests that were influenced by certain factors, this test did not result in changes caused by specific factors such as increased extract concentration or temperature changes. The purpose of this stability test is to evaluate foam stability by measuring foam height in a test tube at specific time intervals, as well as the surfactant's ability to produce foam. The reduction in the volume of liquid flowing from the foam after a certain period, when the foam breaks and disappears, is expressed as a percentage. Foam stability refers to the ability of bubbles to remain intact, where after five minutes, the foam is expected to retain 60–70% of its initial volume [27]. The foam characteristics of soap are influenced by various factors, including the presence of surfactants, foam stabilizers, and other ingredients used in the liquid soap. Long-lasting foam is preferred as it helps with cleansing, but excessive use of foaming agents can cause skin irritation [28].

CONCLUSION

The physical characteristics of the liquid soap formulations containing red pidada leaf extract met all evaluation criteria except for the F3 (8%) formula in the specific gravity test.

The stability test using the cycling method affected the physical properties of all extract-containing formulas, resulting in very thick consistency. Only the pH and foam stability tests met the requirements post-cycling, while the specific gravity test met the criteria only in the F1 (2%) formula.

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