JURNAL SYNTAX TRANSFORMATION

Volume 5, No. 10 October, 2024
p-ISSN 2721-3854 | e-ISSN 2721-2769

Formulation and Stability of Exfoliating Liquid Soap Nanoemulsion from Lerak Extract (*Sapindus rarak DC*) and Coconut Dregs Waste

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ABSTRACT
The use of natural-based skincare products is increasing along with public awareness of the importance of choosing safe and environmentally friendly ingredients. This study aims to develop a nanoemulsion-based exfoliating liquid soap from lerak (*Sapindus rarak* DC) extract and coconut pulp. Considering the environmental challenges due to the use of plastic-based products, this study adopts an eco-friendly approach by using natural raw materials. The Ultrasonic-Assisted Extraction (UAE) method was applied to extract active compounds from lerak fruit, followed by the preparation of *nanoemulsions* using a high-pressure homogenizer. Results showed that the resulting nanoemulsion had good stability, with small droplet size and viscosity suitable for cosmetic products. Organoleptic tests showed that the final product was safe and non-irritating to the skin. The developed exfoliating liquid soap product shows potential as an environmentally friendly alternative to synthetic surfactant-based products. This research contributes to the development of sustainable cosmetic products that utilize local resources, as well as raising awareness of the use of natural ingredients in the beauty industry.

Keywords: Lerak, nanoemulsion, liquid soap, extraction.

INTRODUCTION
In order to achieve the UN Sustainable Development Goals by 2030, green entrepreneurship policies are an important contributor to sustainable development (Wei et al., 2023). Consumer awareness to consider green products has grown rapidly over the past three decades, especially recently. Limited natural resources, waste generation, climate change and air pollution are major ecological challenges that need to be addressed urgently (Liobikienė & Bernatonienė, 2017). Media coverage of sustainable development increases public awareness of environmental issues. This, in turn, motivates the use of more environmentally friendly products (Tengli & Srinivasan, 2022).

Indonesia as a tropical country with abundant flora and fauna has the potential to grow its image as a producer of eco-friendly cosmetics. This country has the opportunity to be the target of pharmaceutical and biotechnology research because it has the second richest diversity of simplisia after the amazon forest (Elfahmi et al., 2014). Indonesia is home to 10% of the world's flowering plant species, 12% of global mammal species, 14% of global coral reefs, 15% of global insects, 16% of global amphibians and reptiles, 17% of total bird species, and 25% of global fish species (Tambunan, 2022). The great potential of natural wealth in Indonesia is the right land for young entrepreneurs to utilize it as the main raw material for environmentally friendly products (Sutarno & Setiawan, 2015).

One of the plants that grows in tropical climates is *Sapindus rarak*, which is often used as a surfactant because of its saponin content in making soap (Wisetkomolmat et al., 2019). *Sapindus rarak* is a group of natural surfactants that can replace synthetic surfactants that are toxic to consumers and the environment. Surfactants play a role in reducing the surface tension between water and oil in making emulsions (Pradhan & Bhattacharyya, 2017). Some synthetic surfactants are considered irritants that damage the epidermis layer of normal skin because they contain toxic substances that are harmful to the skin. The results of a study by Jurek et al., 2021 showed that four synthetic surfactants, namely *Cocomidopropyl Betaine* (CAPB), ammonium lauryl sulfate (ALS), sodium laureth sulfate (SLES), and sodium lauryl sulfate (SLS) showed cytotoxic effects on normal human keratinocytes (HaCaT) with dry mass values ranging above 0.02%, while the dry mass content of soapwort extract 0.6% was not as dangerous as the four synthetic surfactants (Jurek et al., 2021). The results of Lu's research, 2016 proved that synthetic surfactants used in cosmetic products are often above 1% w/v (10000 μg/mL) are considered more toxic compared to the 48-hour test on the skin epidermis model at a concentration of 1000 μg/mL which shows cytotoxicity or irritant effects (B. Lu, 2016). The advantages of bio-surfactants compared to synthetic surfactants include low cytotoxicity, derived from renewable resources, easily biodegradable, very good surface activity, and high specificity and effectiveness in extreme pH and temperature conditions (Vijayakumar & Saravanan, 2015). Compared with synthetic surfactants, bio-surfactants show no toxicity or lower toxicity (Drakontis & Amin, 2020). It can be concluded that bio-surfactants are more recommended for use compared to synthetic surfactant raw materials.

One of the bio-surfactants that is widely used in the pharmaceutical industry is soapnut, for example Lerak. Lerak fruit (Sapindus rarak DC) is included in the genus of fruit that contains other *saponins* such as *Sapindus mukorossi, Sapindus saponaria*, and *Sapindus trifoliatus* (Goyal, 2014). Soap nuts are traditionally used as raw materials for making facial cleansers to treat acne, eczema, and scabies because of the chemical components contained in soap nuts such as 12% surfactants, 0.0365 steroids, 1% alkaloids, and 0.029% triterpenes. Soap nuts have properties that are efficacious as antioxidants, antibacterials, and antifungals (Widowati et al., 2022). The content and benefits of soapberry fruit are used as raw materials for detergent production because they have the effect of cleaning oil and dirt on the body (Handayani et al., 2024).

Environmental concerns such as microplastics are often used as cleaning particles in bath soaps. Microplastics are reported to be a major threat to deep-sea, coastal, and freshwater lake habitats (Vijayaraman et al., 2020). All single-use plastics pose significant water pollution. Nonetheless, this so-called abundant and harmful threat can damage tissue walls or skin cell membranes (Kühn & van Franeker, 2020; Seltenrich, 2015). Therefore, in this study, coconut pulp (Cocos Nucifera L.) will be used as a natural exfoliator to replace synthetic exfoliators that are microplastic. According to Karina et al., 2019, coconut waste is rich in nutrients consisting of 5.78% protein, 38.24% fat, and 15.07% fiber. In addition, coconut waste also contains substances that can inhibit the absorption of nutrients, such as galactomannan (61%), manan (26%), and cellulose (16%). (Karina et al., 2019). The crude fiber contained in coconut pulp has the potential to be a cleaning agent.

The emulsification process affects the stability of the emulsion phase (Ravera et al., 2021). The constituent elements of emulsions have different physiochemical properties so it is necessary to make the right formulation according to the standard of use, as well as the procedure for making emulsions. Nanoemulsion is a technique commonly used in cosmetic manufacturing to obtain good emulsion stability. Emulsion stability is influenced by droplet dimensions, surface tension characteristics (charge, thickness, density, hydrophobic properties), solution conditions (pH, ionic strength, temperature, osmotic pressure), and phase properties (refractive index and dielectric constant) (Hu et al., 2017). The smaller the droplet size of the emulsion, the higher the stability level. The level of emulsion stability affects the shelf life and physical form of the product (Husni et al., 2019).

In this study, high energy processing or dispersion using a high-pressure homogenizer will be used. There are two nanoemulsion methods, namely using high energy and low energy. The advantage of using high energy techniques with high-pressure homogenizers compared to low energy techniques is a simple production method with high nanoemulsion stability (Hidajat et al., 2020), produces a smaller particle size dispersion, and provides flexibility in composition selection (Kumar et al., 2019). To obtain good emulsion stability with a long shelf life, a small emulsion droplet size is required to avoid flocculation, sedimentation, creaming, inversion, as well as coalescent and Ostwald (Suyanto et al., 2019).

In addition to homogenization, another process that affects the stability of the emulsion is the extraction of soapberry fruit. The extraction method plays an important role in the manufacture of emulsions that have a significant impact on the physiochemical characteristics of the emulsion applied in cosmetic products. Elements such as the chemical factors of the emulsifier, the amount of emulsifier used, and the aging test conditions applied contribute to the viscosity and stability of the rheological aspects that play a role in determining the standards and effectiveness of the resulting cosmetic products (Franzol et al., 2021). In addition, sensory evaluation in organoleptic testing includes assessment of texture such as ease of dispersing itself into the skin. These factors highlight the importance of choosing the right extraction method to obtain the desired product properties in cosmetic emulsions.

Considering the environment, extraction methods such as Ultrasonic-Assisted Extraction (UAE) are the right choice to extract soapberry fruit. This method requires only a small amount of solvent compared to traditional extraction methods such as maceration, soxhletation, percolation, digestion, and reflux. Ultrasonic-Assisted Extraction (UAE) extraction technique is an effective procedure that utilizes ultrasonic waves to extract compounds from various plant sources (Freitas de Oliveira et al., 2016). Ultrasonography is a mechanical vibration that has a frequency (20 kHz) exceeding the range of human hearing capacity (20 Hz to 20 kHz). Ultrasonic-Assisted Extraction (UAE) is a fast and efficient extraction technique that has many benefits, such as increasing the effectiveness of extraction, saving energy, and the moderate temperature used can maintain the quality of the extract because it protects compounds that are sensitive to heat (Esclapez et al., 2011). Ultrasonic-Assisted Extraction (UAE) has been used to produce various high-value products from microorganisms, such as color, fat, complex sugars, and proteins. Ultrasonic-Assisted Extraction (UAE) is also influenced by several process variables, such as ultrasonic power, frequency, extraction temperature, reactor characteristics, and solvent-sample relationships. The Ultrasonic-Assisted Extraction (UAE) method is considered superior compared to conventional extraction methods such as maceration or soxhlet which use longer times, use additional solvents, and require higher energy (Martínez Chamás et al., 2023). Therefore, with Ultrasonic-Assisted Extraction (UAE), good extraction can be carried out to maintain the extract content, obtain the desired extract, and be environmentally friendly.

This study is intended to create environmentally friendly products by considering the green chemistry system with environmentally friendly technology and raw materials from natural potential originating from Indonesia. In addition, to determine the effect of high-pressure homogenizers and the selection of Ultrasonic-Assisted Extraction (UAE) extraction methods on emulsion stability. Emulsion stability can be observed from the length of shelf life and the physical form of the product which can be tested with a centrifugator. This study focuses on assessing the stability of the emulsion from droplet size, viscosity, homogeneity, and organoleptic, safety to the skin or irritant effects, and the accuracy of the formulation of the final product of exfoliating liquid soap made from *Sapindus rarak DC* extract and coconut pulp. The results of this study will show the right formulation with organic raw materials and non-toxic effects on the skin using organoleptic tests, liquid soap quality tests according to SNI, emulsion stability tests, and toxicity tests to determine their effects on the skin. The ANOVA method is used as an instrument to produce quantitative data on the results of the study.

RESEARCH METHODS

Tools and materials

Tool

The tools used in the research on making exfoliating liquid soap can be seen in Table 1 below.

Table 1 Tools used in making exfoliating liquid soap

No	Tool	Amount	Function	Acquisition
1.	Homogenizer	1	Reducing the size of droplet particles, creating liquid soap *nanoemulsions*.	UNWAHAS Laboratory
2.	Brookfield Viscometer	1	Obtain the viscosity value of the solution.	UNWAHAS Laboratory
3.	Thermometer	1	Measuring temperature.	UNWAHAS Laboratory
4.	pH meter	1	Measuring pH.	UNWAHAS Laboratory
5.	Centrifugator	1	Knowing the stability of the formula.	UNWAHAS Laboratory
6.	Oven	1	Drying soap nuts and drying coconut pulp.	TRKI Laboratory
7.	Grinder	1	Smoothing coconut dregs	TRKI Laboratory
8.	Sieve	60M and 30M	Produces smaller particle sizes	TRKI Laboratory
9.	UV-VIS Spectrophotometer	1	Measuring the concentration of soapberry fruit extract	UNWAHAS Laboratory
10.	Desiccator	1	Measure the weight until it produces a constant weight.	UNWAHAS Laboratory
11.	Krisbow Ultrasonic Bath	1	Used in the Ultrasonic-Assisted Extraction (UAE) method to obtain soapberry extract.	UNWAHAS Laboratory
12.	Hot Plate	1	Melting solids.	UNWAHAS Laboratory
13.	Digital scales	1	Measuring the weight of the material.	UNWAHAS Laboratory
14.	Beaker glass	5	Formulation container in making exfoliating liquid soap.	UNWAHAS Laboratory
15.	Glass stirrer	1	Stir the exfoliating liquid soap formulation.	UNWAHAS Laboratory
16.	Magnetic stirrer	1	Stir the liquid soap formulation.	UNWAHAS Laboratory
17.	Zetasizer	1	Measuring shelf life	Undip Integrated Laboratory

Material

The materials used in the research to make exfoliating liquid soap are:

Table 1. Ingredients Used in Making Exfoliating Liquid Soap

No	Material	Amount	Function	Acquisition
1.	Soap Nut Fruit	3 kg	The main ingredient	shopee store
2.	Glycerin	450 mL	As a humectant	Shopee store/Indrasari chemical store
3.	Hydroxypropyl methyl cellulose (HPMC)	100 grams	Thickening agent, provides a transparent effect	Shopee store/ Indrasari chemical store
4.	Avocado oil	100 mL	As an emollient	Indrasari chemical store/ Shopee store
5.	Shea butter	100 gr	Moisturizing and nourishing skin	Shopee store/ Indrasari chemical store
6.	Liquid germall plus	50 mL	To maintain product stability from microorganism contamination, as a natural preservative in cosmetics	Shopee store/ Indrasari chemical store
7.	Coconut dregs	2 kg	Exfoliating agent	Coconut pulp seller (market)
8.	Citric acid	25 mL	Adjusting pH	Indrasari chemical store/ Shopee store
9.	Aquades	6 L	Universal solvent	Indrasari chemical store/ Shopee store
10.	Tweens 80	25 mL	Emulsifying agent	Indrasari chemical store/ Shopee store
11.	Sunflower oil	200 mL	Vitamin E	Indrasari chemical store/ Shopee store
12.	Peppermint oil	100 mL	Cooling effect	Indrasari chemical store/ Shopee store
13.	Methylene blue	50 mL	Determining the type of emulsion	Indrasari chemical store/ Shopee store
14.	Ethanol	50 mL	Determination of saponin levels	Indrasari chemical store/ Shopee store
15.	KOH	50 mL	Saponification process	Indrasari chemical store/ Shopee store
16.	Litmus Paper	1 box	Checking pH	Indrasari chemical store/ Shopee store
17.	Filter paper	1 sheet	Filtering the supernatant of soapberry extract	Indrasari chemical store/ Shopee store

Research Design

The research design that will be carried out in making exfoliating liquid soap is as follows:

Material Name	Amount (%)	Function of Materials

	Formula I	Formula II	Formula III	Formula IV	
Soap Nut Extract	5 mL	5 mL	5 mL	5 mL	Surfactant
Glycerin	15 mL	15 mL	15 mL	15 mL	Humectant
Hydroxypropyl methyl cellulose (HPMC)	3 grams	4 grams	5 grams	6 grams	Thickening agent
Shea butter	3 grams	3 grams	3 grams	3 grams	Moisturizer
Avocado oil	5 mL	5 mL	5 mL	5 mL	Emollients
Sunflower oil	5 mL	5 mL	5 mL	5 mL	Vitamin E
Peppermint oil	1 mL	1 mL	1 mL	1 mL	Cooling effect
Liquid germall plus	2 mL	2 mL	2 mL	2 mL	Natural preservatives
Tweens 80	2 mL	1.5 mL	1 mL	0.5 mL	Emulsifying agent
Wet coconut dregs	3 grams	4 grams	5 grams	6 grams	Exfoliator
Vanilla fragrance	1 mL	1 mL	1 mL	1 mL	Fragrance
Citric acid	Enough	enough	enough	enough	pH Regulator
KOH	Enough.	enough	enough	enough	Saponification
Aquades	Up to 100 mL	Up to 100 mL	Up to 100 mL	Up to 100 mL	Solvent

Table 2. Exfoliating Liquid Soap Making Formulation

Research Stage Design
The diagram of the research stages can be understood from the following image:

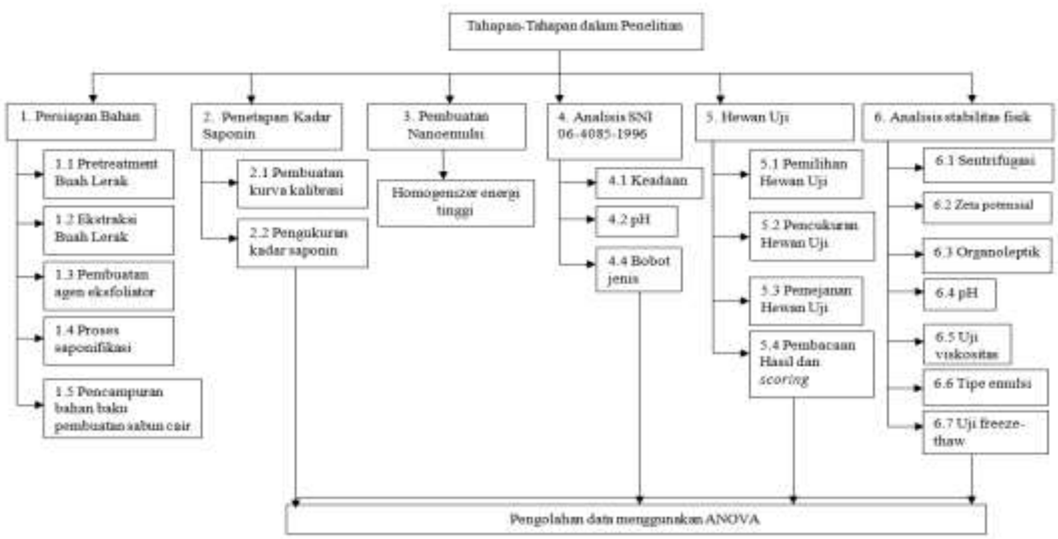


Figure 1. Research Stage Design Chart

Stages in Research
In this study, two stages were examined, namely preparation of materials and making *nanoemulsions*. The following are the stages of research in making exfoliating liquid soap:

Preparation of Materials

The stages of this research are divided into four stages, namely the pretreatment procedure of soapberry fruit, extraction of soapberry fruit, formulation of exfoliating liquid soap, and making *nanoemulsion*. The following are the research procedures that will be carried out:

Research procedure of pretreatment of soapberry fruit

To obtain extract from soapberry fruit, at this stage will operate the Ultrasonic-Assisted Extraction (UAE) method. Before being extracted, the soapberry fruit is first pretreated. Here are the pretreatment steps:

1. Lerak fruit without seeds is dried first using an oven at 100°C for ±12 hours.
2. The dried soap nuts are then crushed using a grinder.
3. The powder obtained was then sieved using a 60 mesh sieve to obtain a uniform fine powder.
4. Sifted dry powder is ready to use.

Research procedure for soapberry fruit extraction

The method used is Ultrasonic-Assisted Extraction (UAE). The following are the steps used to extract soap nuts:

1. The UAE method uses an ultrasonic bath device (Krisbow Bath Ultrasonic) which has a fixed power of 280 W at a frequency of 50/60 Hz.
2. The extraction process will take place four times with a ratio of soapberry powder: distilled water (S/L) (1:3) mixed in a beaker with an aluminum foil cover.
3. Extraction was carried out in an ultrasonic chamber for 1 hour at a temperature of 40°C.
4. The sample was placed in the ultrasonic bath center at a depth of fifteen centimeters.
5. The second and third treatments were carried out for the subsequent soapberry powder.
6. The sonicated solution was filtered with a filter cloth and then centrifuged for 15 minutes at 4,500 rpm to remove solid particles.(Nafiunisa et al., 2019).
7. The supernatant is carefully taken. The supernatant is the result of the extraction of the soapberry fruit.(Nafiunisa et al., 2019).
8. The concentration of the soapberry fruit extract was then measured using a UV-Vis spectrophotometer to obtain the resulting concentration value.

Saponification process

The working steps of the saponification process are as follows:

1. The ingredients prepared include 2 mL of 20% KOH, soapberry extract, 5 mL of avocado oil, 5 mL of sunflower oil, 3 grams of shea butter.
2. Before the saponification process, the pH of the soapberry extract is checked first using litmus paper. The function of checking the initial pH is to measure the KOH that will be added. The pH size of liquid soap is around 6-8 because it is a deluxe type of liquid soap.
3. Prepare a 100 mL beaker for 3 grams of shea butter. Before making the oil phase, the shea butter is melted first with a hot plate. Add 5 mL of avocado oil, 5 mL of sunflower oil to the beaker containing the melted shea butter.
4. Then add 5 mL of soapberry extract and pour it into a 100 mL beaker. Add enough 20% KOH into the beaker.
5. Mix the three mixtures in a 100 mL beaker, then heat.
6. Boil the solution at a temperature of 50-60 ºC for 30 minutes using a hot plate while stirring using a magnetic stirrer while still paying attention to the pH.

Procedure for making exfoliating liquid soap formulation

The procedure for making the exfoliating liquid soap formulation is as follows:

1. After the saponification process is complete.
2. Prepare a 100 mL beaker on a digital scale. Weigh (3, 4, 5, 6 grams) of HPMC.

3. Then add 15 mL of glycerin and 3 grams of Hydroxypropyl methyl cellulose (HPMC) as a thickening agent. Stir it until evenly mixed using a stirring rod.
4. Then add the previously saponified liquid soap little by little while continuing to stir in the beaker.
5. Then add (2; 1.5; 1; 0.5 mL) tween 80 as an emulsifying agent and mix it with a glass stirrer until evenly mixed.
6. The next step is to add 2 mL of liquid germall plus, 1 mL of fragrance, and 1 mL of peppermint oil into the beaker of the liquid soap formulation.

Nanoemulsion Making

The stages that can be carried out in making *nanoemulsions* are as follows:

1. Place the 100 mL beaker containing the liquid soap mixture on the hot plate before homogenizing.
2. Install the homogenizer so that it does not reach the bottom of the glass, a maximum of 2 cm from the bottom of the glass.
3. The *ultraturrax* homogenizer was turned on at a speed of 5000 rpm with a constant temperature of 50 ºC for 15 minutes to process the liquid soap formulation into a *nanoemulsion* (Zulfa, E., Novianto, D., & Setiawan, 2019).
4. Add distilled water little by little until it reaches 100 mL.
5. Let the liquid soap emulsion stand for 1 day (24 hours) before testing the viscosity and density.

Addition of Exfoliator Agent

Adding dried coconut pulp (3 grams, 4 grams, 5 grams, 6 grams) into the liquid soap formulation as an exfoliator. Then stir it using a glass stirrer until evenly mixed (the addition of coconut pulp as an exfoliator agent is done after the series of tests are completed).

Analysis of SNI 06-4085-1996

SNI liquid detergent checks are used to meet the classification set by the National Standardization Agency (BSN). The following are the work steps to obtain liquid soap according to SNI 06-4085-1996 :

Table 3. Liquid Bath Soap Quality Requirements

No	Test Criteria	Unit	Condition	
			Type S	Type D
1.	Condition:			
	- Form		Fluid	Fluid
			homogeneous	homogeneous
	- Smell		Typical	Typical
	- Color		Typical	Typical
2.	pH 25º		8 – 11	6 – 8
3.	Free alkali (calculated as KOH)	%	Max 0.1	Not required
4.	Active ingredients	%	Min. 15	Min. 10
5.	Specific gravity, 25ºC		1.01 – 1.10	1.01 – 1.10
6.	Microbial contamination: Total plate number		Colony/g	Colony/g

Condition (organoleptic test)

Sample inspection is done visually by observing its smell, color, and shape to determine its quality and condition. The inspection results can also help in identifying possible problems or contamination in the sample (SNI- 06-4085-1996 , 1996).

pH

To test pH, the pH meter is calibrated beforehand with solutions with pH of 4.7 and 10 before testing the sample. Then, the pH of the liquid soap is measured by dissolving 1 gram of soap in 10 mL of distilled water and reading the pH value displayed on the pH meter when the number does not change and shows 'ready' (SNI- 06-4085-1996 , 1996).

Specific gravity

Specific gravity is measured by running a *pycnometer*. The steps are to fill the *pycnometer* with distilled water and liquid soap alternately with the same volume and weigh each sample. Then, the specific gravity is calculated using the following formula:

Information:

W = Sample weight

W1 = Water weight

(SNI- 06-4085-1996 , 1996).

Skin Irritation Analysis Using Rabbits

Toxicity analysis using rabbits aims to assess potential irritation, determine the level of safety for consumers, and assist in product development. Rabbits are used as experimental animals because they have sensitive skin, are readily available, and are large enough to be used as a testing area. According to Zulfa et al., 2018, the following are the stages for irritation testing using rabbits:

Selection of Test Animals

The rabbits to be used are albino rabbits with inclusion characteristics, namely: local white rabbits, male, healthy, 2-4 months old, and no wounds on their back skin. While the exclusion characteristics are: sick rabbits (diarrhea, fever, have fungus or fleas), rabbits with sensitive skin, and rabbits with wounds on their skin.

Shaving of Test Animals

The part to be shaved is the rabbit's back. Rabbit shearing is done carefully to obtain the body parts that will be tested for primary irritation. After the rabbit's fur is shaved, it is left for 24 hours before applying the test compound. The rabbit skin that will be smeared with the test material is marked with a marker to form a small box measuring 2.54 cm x 2.54 cm (F. Lu, 1995; OECD, 2015).

Exposure of Test Animals

Before applying the product, the shaved rabbit skin was cleaned slowly using clean cotton that had been moistened with distilled water. Then apply 0.5 grams of exfoliating liquid soap to the rabbit's skin while rubbing it on its body (to test the effect of adding coconut pulp as an exfoliator agent) and then cover it with sterile gauze and plaster for 24 hours. After that, the rabbit was put in its cage and the next day at the same time, the plaster was removed and the rabbit's skin was cleaned with distilled water from the remaining test compound that was attached. The symptoms observed were primary irritation in the form of erythema and edema for 24, 48, and 72 hours (OECD, 2015).

Reading Results and Scoring

When observing the symptoms of primary irritation toxicity on the skin of guinea pigs, the things that can be observed are the presence of erythema and edema. Then from the level of irritation that arises, a score is given according to the table below. Then calculate the primary irritation index with the formula:

Eri 24 hours + eri 48 hours + eri 72 hours + ede 24 hours + ede 48 hours + ede 72 hours.

Information:

Eri = erythema

Edema = edema(Zulfa et al., 2018).

Table 4. Skin Reaction Evaluation (F. Lu, 1995).

1.	*Erythema*and crust formation	Score
	Without Erythema	0
	*Erythema*very little (almost none)	1
	*Erythema*clearly defined	2
	*Erythema*moderate to severe	3
	*Erythema*heavy (beet red) to slightly crusty (sore)	4
	Total possible erythema score	4
2.	Formation of edema	Score
	Without edema	0
	*Edema*very little (almost invisible)	1
	*Edema*little (clearly defined area edges)	2
	*Edema*moderate (rising edge approx. 1 mm)	3
	*Edema*severe (rises more than 1 mm and extends beyond the extrusion area)	4
	Total possible edema score	4

Table 5. Irritation property category based on the combined average of the primary irritation index of chemical compounds (F. Lu, 1995).

Primary Irritation Index	Compound Groups
< 2	Just a little stimulating
2 – 5	Moderate irritant
> 6	Severe irritation

RESULTS AND DISCUSSION

SNI 06-4085-1996 Test Analysis

Organoleptic Properties

Organoleptic testing is an evaluation process that involves human senses, such as sight, smell, and taste, to assess the sensory characteristics of a product.(Rosmainar, 2021). Organoleptic testing is an important test that needs to be done in evaluating product quality including liquid soap. In SNI liquid soap, this test aims to assess the overall quality of the product starting from appearance (color), aroma, and texture. The following are the results of the organoleptic test of the exfoliating liquid soap product of soap nut extract and coconut dregs:

The following are the values that can be given in the organoleptic test as a sample.

Table 7. Results of Organoleptic Properties Testing of Liquid Soap Products

Formula	Observation	Results	
		Before the Cycling test	**After Cycling Test**
F1R1	a. Form	Thick liquid	Thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown with white foam	Light brown with white foam
F1R2	a. Form	Thick liquid	Thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown with white foam	Light brown with white foam
F1R3	a. Form	Thick liquid	Thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown with white foam	Light brown with white foam
F2R1	a. Form	Thick liquid	Thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown with white foam	Light brown with white foam

Formula	Observation	Results	
		Before the Cycling test	**After Cycling Test**
F2R2	a. Form	Thick liquid	Thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown with white foam	Light brown with white foam
F2R3	a. Form	Thick liquid	Thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown with white foam	Light brown with white foam
F3R1	a. Form	Very thick liquid	Very thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown	Light brown
F3R2	a. Form	Very thick liquid	Very thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown	Light brown
F3R3	a. Form	Very thick liquid	Very thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown	Light brown
F4R1	a. Form	Very thick liquid	Very thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown	Light brown
F4R2	a. Form	Very thick liquid	Very thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown	Light brown
F4R3	a. Form	Very thick liquid	Very thick liquid
	b. Smell	Typical peppermint and has a sweet vanilla scent	Typical peppermint and has a sweet vanilla scent
	c. Color	Light brown	Light brown

(a) (b)

Figure 2. Liquid Soap (a) Before and (b) After Cycling Test

Based on the observation results that have been obtained, it is concluded that there is no significant difference before or after the cycling test. Formulas 1 to 4 do not show significant differences in terms of physical form, aroma, and color. If there is a difference, it is strongly suspected that this is caused by variations in viscosity caused by the addition of HPMC and Tween 80 in varying amounts in each formula. The higher the viscosity of the liquid soap, the greater the value of the organoleptic test obtained. Soap with low viscosity has a thinner consistency, which allows air to mix more easily with the liquid, resulting in foam. Conversely, soap with high viscosity is thicker and more resistant to mixing with air, resulting in less foam.(Mendes Cangussú et al., 2016; Nazdrajic & Bratovcic, 2019). The observation results in Table 4.1 show that the four soap formulas (F1, F2, F3, and F4) remain homogeneous before and after the cycling test. This proves that the formulas are stable and meet the homogeneity quality requirements according to

SNI 06-4085-1966 and the Indonesian Pharmacopoeia Edition IV(Ministry of Health of the Republic of Indonesia, 1995).

pH

The soap we use must be safe for the skin. To ensure this, we need to test the acidity level (pH) of the soap. pH testing is carried out to ensure the safety of soap for human skin. Through pH testing, it is expected to obtain a pH value that is balanced with the natural condition of the skin. This aims to prevent irritation, dryness, or skin damage due to pH imbalance. In addition, pH testing is also important to measure the effectiveness of soap cleaning. A pH value that is too low (acidic) can damage the skin's protective layer.

Table 8. Results of pH Testing of Liquid Soap Products

Formula	pH Percentage (%)	
	Before the Cycling Test	After Cycling Test
F1R1	6.78	6.73
F1R2	6.81	6.79
F1R3	6.80	6.78
F2R1	6.71	6.66
F2R2	6.73	6.72
F2R3	6.69	6.60
F3R1	6.70	6.67
F3R2	6.75	6.73
F3R3	6.84	6.70
F4R1	6.80	6.76
F4R2	6.81	6.82
F4R3	6.83	6.69

The pH measurement was carried out before and after the cycling test using a pH meter. Based on the tests that have been carried out, the results obtained were that the pH before the cycling test and after the cycling test were not much different. Based on the test results, all four showed acidity levels (pH) that were acidic, but these results were still within the permitted limits based on the Indonesian National Standard (SNI) 06-4085-1966 for deluxe liquid soap products with a pH standard of 6 - 8(Cahyaningsih et al., 2019). Such acidity levels are expected not to cause irritation to the skin when applied. Cycling tests were carried out for 2 days with stages of 24 hours at a temperature of -1ºC then 24 hours at a temperature of 40 ºC. The result is that the pH of the liquid soap tends to be stable(Usman & Baharuddin, 2023).

Skin Irritation Analysis

Using Test Animals

The acute dermal irritation test is one of a series of toxicity test processes carried out on test animals such as albino rabbits, which functions to determine the toxic or irritating effects that are visible after exposure to the test animal formulation.(BPOM RI, 2014). The quantitative results obtained are a source of information regarding the degree of danger if the formulation is exposed to humans, so that the right dose can be determined for use for human safety. The purpose of this acute dermal test is to determine whether or not there is an irritation effect on the skin of test animals and to assess and evaluate the characteristics of a substance if exposed to the skin.(BPOM RI, 2014; OECD, 2015).

Erythema is a redness reaction that occurs on the skin that appears due to the side effects of using topical preparations. This redness is accompanied by the appearance of raised spots that are spread symmetrically. The symptoms that arise are followed by vesiculation (watery) accompanied by itching and heat. While edema is a swelling reaction that occurs on the skin caused by the side effects of using topical preparations. Edema can occur due to an increase in the volume of fluid outside the cells (extracellular) and outside the blood vessels (extravascular) that accumulates in the body's tissues.

The test animals used in skin irritation testing are rabbits. Rabbits are test animals that are generally used in the world of pharmacology in irritation testing. Rabbits are used as irritation testing because rabbit skin is quite sensitive skin. The rabbits used are male and albino rabbits. The variables used in this study are HPMC (Hydroxypropyl Methylcellulose) and tween 80. Different concentrations in the use of HPMC and tween 80 in the soapberry extract liquid soap formulation can affect the results of irritation on rabbit skin. The results of the irritation test obtained will show erythema and edema on rabbit skin. Testing is carried out with a time span of 24, 48, and 72 hours.

Table 9. Skin Reaction Evaluation Table based on US EPA (United States Environmental Protection Agency, 2002).

1.	*Erythema*and crust formation	Score
	Without Erythema	0
	*Erythema*very little (almost none)	1
	*Erythema*clearly defined	2
	*Erythema*moderate to severe	3
	*Erythema*heavy (beet red) to slightly crusty (sore)	4
	Total possible erythema score	4
2.	Formation of edema	Score
	Without edema	0
	*Edema*very little (almost invisible)	1
	*Edema*little (clearly defined area edges)	2
	*Edema*moderate (rising edge approx. 1 mm)	3
	*Edema*severe (rises more than 1 mm and extends beyond the extrusion area)	4
	Total possible edema score	4

Table 10 Dermal Irritation Index PDII for Classifying Irritation Potential in Test Animals (Derelanko et al., 1993).

WWII	Classification
0.0	Non-irritating
0.1-0.5	Negligible irritation
0.6-2.0	Mild irritation
2.1-5.0	Moderate irritation
5.1-8.0	Severe irritation

Table 11. Table of Toxicity Category Criteria for Skin Corrosion or Irritation based on US EPA (United State Environmental Protection Agency, 2002)**.**

Toxicity Category	Criteria
I	Corrosive (anytime)
II	Severe irritation at 72 hours
III	Moderate irritation at 72 hours
IV	No irritation to mild irritation at 72 hours

The PII index can be obtained for each test animal by adding all erythema and edema scores for 24, 48, and 72 hours and then dividing the sum by 4 to obtain the individual irritation score.
Information:
Er = erythema score
Ed = edema score
JO = number of observations
24 hours, 48 hours, and 72 hours are the observation times

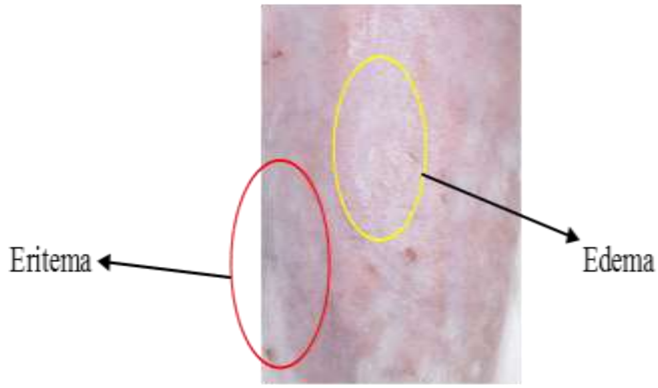


Figure 3. Erythema and Edema on Rabbit Skin

(a) (b) (c)

Figure 4. Irritation Test to Rabbit Skin for (a) 24 hours, (b) 48 hours, and (c) 72 hours.

The acute dermal irritation test data were analyzed quantitatively using an erythema and edema assessment scale. The scores obtained at each observation time (24, 48, and 72 hours) were then averaged to obtain a primary irritation index, which represents the severity of the overall skin inflammatory reaction. The following are the results of irritation testing using rabbits as test animals:

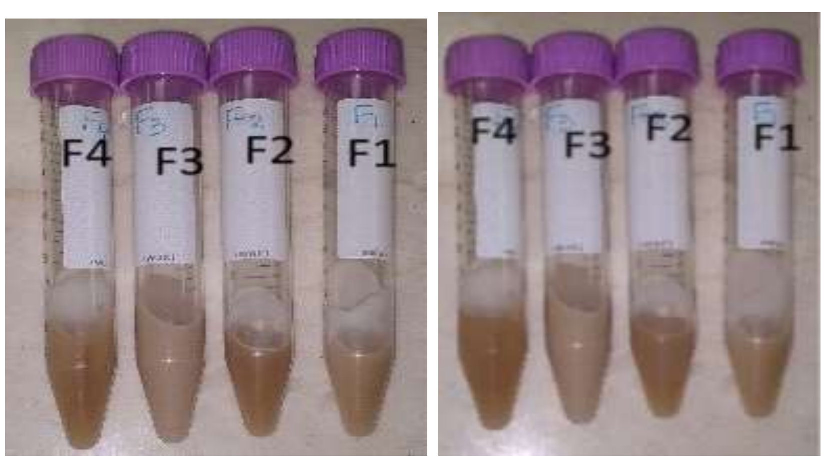
Table 12. Results of Irritation Test of Soap Nut Extract Liquid Soap

Formula	**24 Hour Treatment**	
	Erythema	**Edema**
F1	2	1
F2	2	0
F3	1	0
F4	0	0
Neutral F1	3	2
Formula	48 Hour Treatment	
	Erythema	Edema
F1	2	1
F2	1	0
F3	1	0
F4	0	0
Neutral F1	3	2
Formula	72 Hour Treatment	
	Erythema	Edema
F1	1	0
F2	0	0
F3	0	0
F4	0	0
Neutral F1	1	1

Based on quantitative observations obtained from the assessment of a veterinarian, namely Drh. Deasy Andini Ersya Putri using the erythema and edema assessment scale (Table 4.3), the highest score obtained was 3. This result indicates a redness and swelling reaction on the skin after applying the formulation. When compared to normal skin, all formulas showed a higher level of irritation. Then the result of the PII Index calculation was 4.8, meaning that there was irritation but only slightly stimulating.
Based on the results obtained above, it can be concluded that the treatment of giving soap soap extract to five male rabbits at 24, 48, 72 hours, namely the longer the time, the better the rabbit's skin, meaning that soap soap extract does not cause irritation to the rabbit's skin.

In the first 24-hour treatment, it was found that formula 1 and neutral obtained high erythema and edema values, it can be concluded that the administration of tween 80 affects irritation to the rabbit's skin. In formula 1 and neutral with the addition of tween 80 as much as 2 ml, this resulted in quite high erythema and edema. While in formula 4 with the addition of tween 80 as much as 0.5 ml did not cause erythema and edema on the rabbit's skin. The addition of tween 80 shows that the higher the concentration used, the more it will cause irritation to the skin(Belgium, 1998).

Physical Stability Analysis of Liquid Soap Product Nanoemulsion
Centrifugation Method
Centrifugation aims to accelerate the phase separation process that may occur during long-term storage. In emulsion stability, centrifugation plays a role in early detection of phase separation, evaluating kinetic stability, formulation comparison, the influence of formulation factors, and predicting long-term stability.(Iskandar et al., 2021). Centrifugation testing was carried out before and after the cycling test at a speed of 3000 rpm for 30 minutes.(Iskandar et al., 2021). From the analysis results obtained through the centrifugation process showed stability. The stability of the soapberry extract liquid soap was maintained well before and after the cycling test. This indicates that the four soapberry extract liquid soap formulations have a good stable emulsion.



(A) (B)

Figure 6. Centrifugation (a) Before and (b) After Cycling Test

Zeta Potential Testing
Zeta potential is commonly used to describe the surface charge properties of very small particles, related to the electrical attraction between very small particles. This electrical attraction will determine the tendency of aggregation and repulsion. Zeta potential is a measure of the surface charge of particles distributed in a carrier medium. Ideally, the zeta potential of the particles should be higher than that of the carrier medium to prevent aggregation. The zeta potential should be controllable(Abdassah, 2017).

Table 13. Results of Zeta Potential Testing of Liquid Soap Products

Formula	After Cycling test
1	-19.04 mV
2	-18.17 mV
3	-22.15 mV
4	-22.1 mV

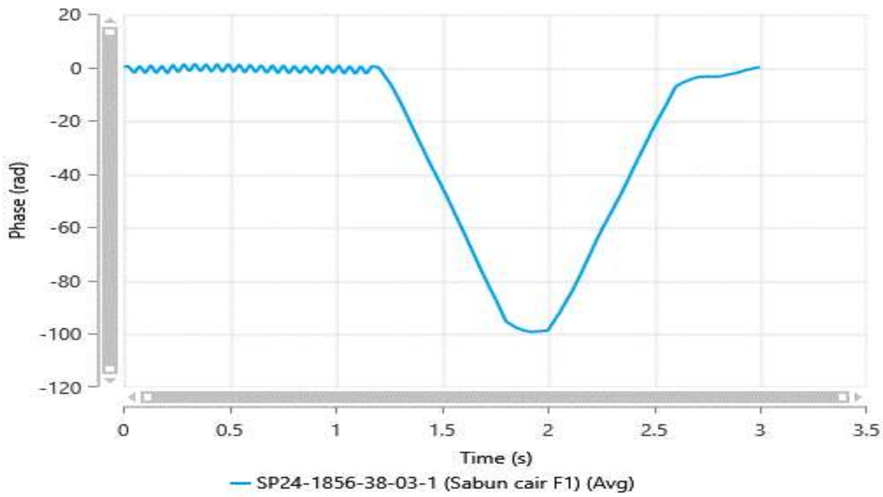


Figure 7. Distribution of Zeta Potential in Formula 1 of Soap Liquid Soap Extract

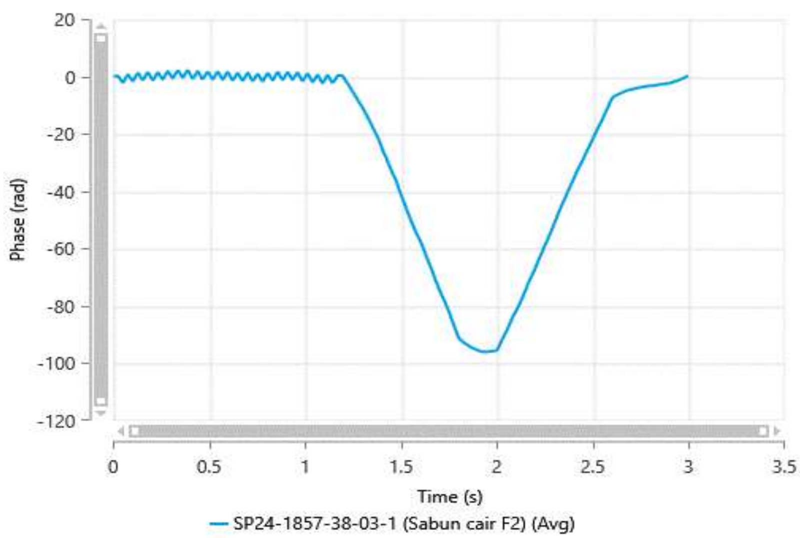


Figure 8. Distribution of Zeta Potential in Formula 2 Soap Liquid Soap Extract

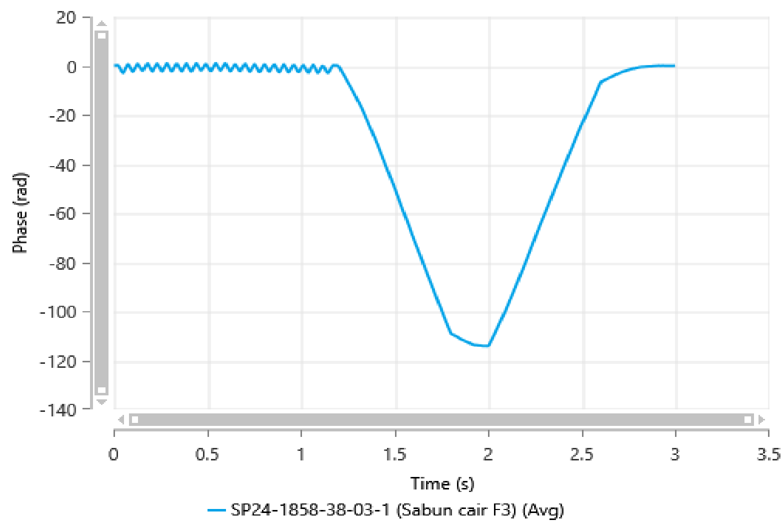


Figure 9. Distribution of Zeta Potential in Formula 3 Soap Liquid Soap Extract

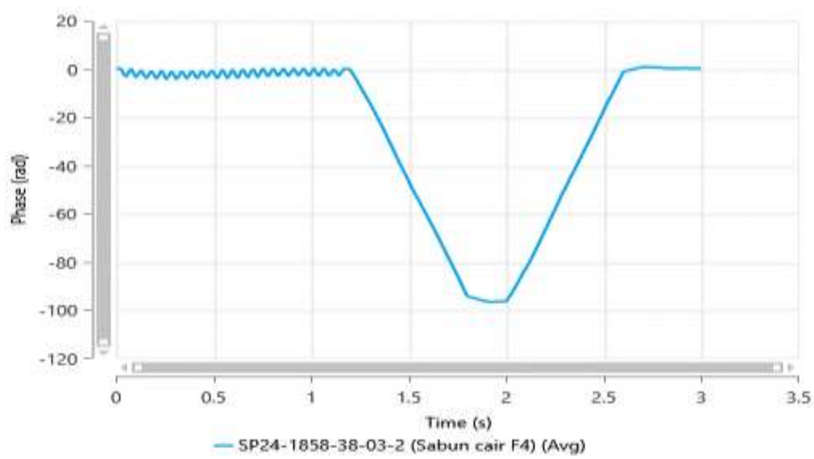


Figure 10. Distribution of Zeta Potential in Formula 3 Soap Liquid Soap Extract

Based on the results of the zeta potential test of the four liquid soaps, all four obtained quite high negative zeta potentials, this indicates that the dispersion stability of the four formulas is quite good. The negative sign indicates that the surface of the soap particles tends to be negatively charged. A fairly high and negative zeta potential value indicates that this liquid dispersion tends to be stable. Negatively charged soap particles will repel each other, making it difficult to clump or settle. The addition of soapberry extract also contributes to the zeta potential value obtained.

The zeta potential value is greater than +30 MV or less than -30 MV, at that value it has a high degree of stability. Lower values can cause aggregation because there is a van der waals force between particles (attraction force between weak electrically charged particles) during the shelf life.(Wulansari et al., 2019). It can be concluded that when viewed directly from the values obtained from the zeta potential test, F3 and F4 have the highest zeta potential values among the four, namely F3 of -22.15 mV and F4 of -22.10 mV. Therefore, F3 and F4 are the formulas with the best stability.

CONCLUSION

Based on the research that has been conducted, it can be concluded that all liquid soap formulations of soapberry extract containing coconut pulp exfoliation are considered moderately irritating, which is obtained based on a reaction score of 4.8. In addition, the formulation shows a good level of stability as a liquid soap, which is tested through zeta potential measurements with a value of -22.15 mV.

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