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Liquid Soap Formulation from Cocoa Pod Husk Extract (*Theobroma Cacao* L.) and Antioxidant Activity

Umi Nafisah[1], Praptanti Sinung Adi Nugroho[2], Wahyu Setyorini[3]

[1,3]Study Pharmacy Program, Politeknik Indonusa Surakarta, Jl. K.H. Samanhudi No. 31 Surakarta

[2]Pharmaceutical and Food Analysis, Poltekkes Kemenkes Surakarta, Jl. Ksatria No.2, Danguran, Klaten Selatan, Klaten

ABSTRACT

Liquid soap is a cleaning agent made from synthetic surfactants, including anionic surfactants combined with amphoteric and nonionic surfactants; it can be supplemented with other additives that do not cause skin irritation. The active ingredient or substance, which can function as an antiseptic, antioxidant, or other significant roles, is an equally important component in soap making. Cocoa pod husk waste is one natural ingredient that can serve as an active substance in soap making. Cocoa husks contain flavonoids, alkaloids, tannins, saponins, and triterpenoids. The cocoa pod husk extract has very strong antioxidant activity and also possesses antibacterial activity. This research aims to determine the formulation and physical stability of liquid soap made from cocoa pod husk extract, as well as its antioxidant activity. The study commenced by extracting cocoa pod husk using the maceration method, which yielded a concentrated extract, and then proceeded with phytochemical analysis. Subsequently, we carried out the formulation of liquid soap, evaluated its homogeneity, organoleptic characteristics, pH, viscosity, specific gravity, foam height, and stability testing, and assessed its antioxidant activity. The evaluation results indicated that the liquid soap with cocoa pod husk extract had good physical properties in terms of homogeneity, organoleptic characteristics, pH, viscosity, specific gravity, and foam height. The results of the antioxidant activity test showed that the higher the concentration of cacao pod extract in the liquid soap, the lower the IC_{50} value. In formula V, the liquid soap with a 4% concentration of cacao pod extract obtained an IC_{50} value of 44.5.

KEYWORDS: cocoa pod husk, liquid soap, evaluation, antioxidant

ARTICLE DETAILS

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INTRODUCTION

The skin is the largest and most visible organ and protects our body (Mazlan & Sharoni, 2024; Michalak et al., 2021). Human skin functions as a physical, chemical, and immune barrier against the external environment while also providing a habitat for its resident microbiota, known as the skin microbiome (Swaney & Kalan, 2021). Significant skin health issues occur worldwide, and developing countries have identified these problems as public health concerns (Gulomovna et al., 2023). Skin health issues will affect the quality of life in the community (AlOtaibi et al., 2021). One way to maintain skin health is by keeping the skin clean using soap (Nafisah et al., 2022).

Soap is a substance humans use to cleanse themselves of dirt caused by bacteria and fungi in human activities, typically possessing a pleasant fragrance. Today, liquid soap is the widely used type of body wash due to its many advantages, such as its practical, hygienic, and portable packaging (Widyasanti et al., 2020). Liquid soap is a cleaning agent made from synthetic surfactants, including anionic surfactants combined with amphoteric and nonionic surfactants. It can be supplemented with other additives that do not cause skin irritation (Borković et al., 2024; Widyasanti et al., 2020). A crucial element in soap production is the active ingredient or substances, which may serve as antiseptics, antioxidants, or other essential compounds. Cocoa pod husk waste is a natural substance that can function as an active ingredient in soap making.

Cocoa pod husk contains flavonoids, alkaloids, tannins, saponins, and triterpenoids (Rahayu et al., 2023). Cocoa husks have outstanding antioxidant activity (Hernández-Hernández et al., 2019; Rahayu et al., 2023). The extract of cocoa fruit husks has antioxidant activity with an IC_{50} value of 10.03 ± 0.43 ppm, which falls into the category of extreme antioxidants (Fahleni et al., 2021). Cocoa husks have antibacterial activity against *Staphylococcus aureus, Escherichia coli, Salmonella sp., Porphyromonas gingivalis,* and *Streptococcus mutans* (Diniardi et al., 2020; Kayaputri et al., 2020; Naufal et al., 2020; Rahayu et al., 2023). Other research findings indicate that cocoa pod husk extract cream has activity as a hair growth stimulator in rabbits (Mustarichie et al., 2022).

Considering the various pharmacological actions of cocoa pod husk, we must carry out additional research to formulate it. This research aims to determine the formulation and physical stability of liquid soap from cocoa pod husk extract, as well as to assess its antioxidant activity. Based on the findings of this study, we expect to see more use of cocoa pod husk waste.

MATERIALS AND METHODS

The materials used in this study include sodium lauryl sulfate (KAO), Cocamide DEA, Cocamidopropyl betaine, TEA lauryl sulfate, Sodium Chloride, DMDM hydantoin, Tetrasodium EDTA, Citric Acid, Glycerol, Cocoa pod husk, Aquadest, pH meter (Ohauss®), Viscosimeter (NDJ 8S®), Oven (Memmert®), Spectrophotometer (Genesys®), Vortex.

Table 1. Liquid Soap Formula

Ingredient	FI (%)	F2 (%)	F3 (%)	F4 (%)	F5 (%)
Sodium lauryl sulfat	7	7	7	7	7
Cocamide DEA	2,5	2,5	2,5	2,5	2,5
Cocamidopropyl betaine	2,5	2,5	2,5	2,5	2,5
TEA lauryl sulfate	2,5	2,5	2,5	2,5	2,5
Sodium Chloride	1,5	1,5	1,5	1,5	1,5
DMDM hydantoin	0,1	0,1	0,1	0,1	0,1
Tetrasodium EDTA	0,05	0,05	0,05	0,05	0,05
Cytric Acid	0,05	0,05	0,05	0,05	0,05
Glyserol	1,5	1,5	1,5	1,5	1,5
Cocoa pod husk extract	0	1	2	3	4
Fragrance	0,2	0,2	0,2	0,2	0,2
Aquadest	82,1	81,1	80,1	79,1	78,1
Total	100	100	100	100	100

A liquid soap-making procedure

The soap-making process refers to research Rosmainar, (2021).

1. Process I
   Mix sodium lauryl sulfate, cocamide DEA, cocamidopropyl betaine, and TEA lauryl sulfate into a 1000 ml glass beaker filled with water and stir until dissolved.
2. Process II
   Mix DMDM hydantoin, tetrasodium EDTA, and citric acid into a 250 ml glass beaker filled with water and stir until dissolved.
3. Process III
   Mix sodium chloride in a 100 ml glass beaker filled with water and stir until dissolved.
4. Mix Process II into Process I, stir until dissolved, then add glycerin and stir until mixed.
5. Add cocoa shell extract and stir until evenly mixed.
6. Add fragrance and stir until evenly distributed.
7. Add process III and stir until it is thick and until it becomes soap.

Evaluation of the Physical Stability of Liquid Bath Soap

Homogeneity Test

Measure 0.1 g of liquid bath soap and uniformly apply it in a thin layer to transparent glass. The preparation must exhibit a uniform composition, with no obvious coarse granules.

Organoleptic Test

Observations are conducted about the form, hue, and fragrance of the liquid bath soap formulation (Verspoor et al., 2023).

Verification of Specific Gravity

Verification of specific gravity. The specific gravity of liquid bath soap is determined using a pycnometer.

Evaluation of the viscosity of liquid soap formulations

The viscosity assessment of liquid bath soap formulations is conducted with a viscometer.

Evaluation of the Foaming Efficacy of Liquid Bath Soap

Measure 1g of soap into a tube containing 10 mL of distilled water and seal it to perform the foam height test. After shaking for 20 seconds, assess the height of the resultant foam. We assess the foam height at the 0-minute and 5-minute marks. The Indonesian National Standard establishes parameters for the height of liquid soap foam, ranging from 12 to 220 mm (Situmorang et al., 2023).

pH Test

The pH of liquid soap is assessed using a pH meter (D. Handayani et al., 2024). We conduct the evaluation both before and after the cycling test.

Cycling Test

The cycling test evaluates the occurrence of phase separation in the preparation throughout the storage process. We conduct a stability examination using the freeze and thaw method, which involves placing 2 mL of the preparation into a vial and sealing it securely. We utilize five vials as controls, maintaining them at a temperature of 25°C.

The remaining 15 vials (each formula repeated three times) will follow a freeze-thaw cycle by being stored at 4°C for 24 hours, followed by storage at 40°C for 24 hours, while monitoring for organoleptic changes and homogeneity (one cycle). We conducted up to six cycles, monitoring the organoleptic alterations in each cycle and re-evaluating the pH at the conclusion.

Assessment of Antioxidant Activity

DPPH Method Antioxidant Assessment

Preparation of DPPH Stock Solution To prepare a DPPH solution, dissolve 2 mg of methanol in a volumetric flask to a final volume of 100 mL. Agitate the solution until it attains homogeneity and uniformity, obtaining a concentration of 0.002%. Subsequently, preserve the DPPH solution in a container lined with aluminum foil. Preparation of blank solution combine 2 mL of the DPPH stock solution (0.002%) with 2 mL of methanol. Vortex the mixture until it attains homogeneity and uniformity. Subsequently, we incubate the mixture for 30 minutes at ambient temperature in a dark environment. Furthermore, we employ UV-Vis spectrophotometry to quantify the absorbance of the solution until a maximum wavelength of 515 nm is attained.

Antioxidant Activity

We introduced a 2 mL sample into a test tube and added 2 mL of DPPH at a concentration of 0.002%. We vortexed the mixture until homogeneous and subsequently incubated it at room temperature in a dark environment. We quantified the absorption at approximately 515 nm utilizing a UV-Vis spectrophotometer. The measurements were conducted twice. We determined the absorption value of the DPPH solution as the percentage (%) inhibition before and after the addition of the sample, employing the formula: % inhibition = (blank absorbance - sample absorbance) / (blank absorbance) x 100%.

RESULT

Phytochemical Screening

Table 2. Results of Phytochemical Screening

Test	Result
Flavonoid	+
Alkaloid	+
Saponin	+
Tanin	+

The extraction process of cocoa husk uses the maceration method with 96% ethanol solvent. The ethanol extract of cocoa husk contains flavonoids, alkaloids, saponins, and tannins. Previous research has shown that cocoa husk contains alkaloids, flavonoids, saponins, tannins, and triterpenoids (Dian et al., 2024; Pandjo et al., 2021).

Table 3. Results of Homogeneity and Organoleptic

Formula	Homogeneity	Organoleptis		
		Shape	Color	Smell
I	Homogen	Liquid	Clear	Signature chocolate
II	Homogen	Liquid	Brick brown	Signature chocolate
III	Homogen	Liquid	Brick brown	Signature chocolate
IV	Homogen	Liquid	Brick brown	Signature chocolate
V	Homogen	Liquid	Brown	Signature chocolate

According to Table 3, the result liquid bath soap formulation is homogenous. The lack of coarse particles when the preparation is put to transparent glass is a sign of this. The composition of a good liquid soap formulation should be uniform and free of coarse particles (Nafisah et al., 2022).

Determining the liquid soap preparation's shape, color, and scent is the aim of organoleptic evaluation (Martihandini et al., 2024). The organoleptic test results suggest that the soap generated is in liquid form and possesses a distinct brown odor. The soap in formula I, which does not contain cocoa pod husk extract, is transparent or translucent, whereas formula II, III, and IV are brick brown, and Formula V is brown. Different extract concentrations will have an impact on the liquid soap's properties.

Table 4. Result of Specific Weight and pH

Formula	Specific Weight (g/mL)	pH Before Cycling Test	After Cycling Test
I	1.027 ± 0.001	8.48 ± 0.006	9.04 ± 0.015
II	1.034 ± 0.003	8.32 ± 0.021	9.02 ± 0.023
III	1.038 ± 0.001	8.38 ± 0.020	9.02 ± 0.015
IV	1.038 ± 0.001	8.30 ± 0.015	9.01 ± 0.015
V	1.039 ±0.000	8.30 ± 0.035	9.21 ± 0.023

Specific gravity is the ratio of a substance's density to the weight of an equivalent volume of water at the same temperature (25°C). The specific gravity of a substance is determined by the type and concentration of the components incorporated into the solution. The specific gravity of liquid soap is determined using a pycnometer. The Indonesian National Standard requires that the specific gravity of liquid bath soap ranges from 1.01 to 1.10 (Nafisah et al., 2022). According to table 4, the specific gravity of the formulated liquid soap is 1.027 ± 0.001 to 1.039 ± 0.000. An increase in the specific gravity of the liquid soap correlates with an increase in viscosity and a thicker texture (P. A. Handayani et al., 2023). Increased concentration of the added extract correlates with an elevated specific gravity of the liquid soap.

The objective of performing a pH assessment is to ascertain the stability and safety of the manufactured liquid soap. A low pH in liquid soap can result in skin irritation, while a high pH can lead to dryness. Prolonged exposure may produce symptoms such as itching, rashes, redness, and scaling (Situmorang et al., 2023). The liquid soap's pH evaluation revealed that as the concentration of the added extract increased, the preparation's pH dropped. Furthermore, it was noted that the pH of the liquid soap formulation elevated following the execution of the cycle test. The pH of the liquid soap complies with the Indonesian National Standard, which requires a range of 8 to 11.

Table 5. Results of Viscosity and Foam Height Tests

Formula	Viscosity (cps)	Foam Height (mm)		Foam Stability (%)
		T0	T5’	
I	463.40 ± 19.674	77.7 ± 7.51	66.0 ± 7.94	85.71
II	607.23 ± 50.030	76.0 ± 6.56	64.7 ± 4.62	85.13
III	733.40 ± 66.751	71.7 ± 2.89	63.3 ± 5.77	88.28
IV	826.00 ± 10.550	70.3 ± 0.58	63.0 ± 0.00	89.61
V	865.00 ± 20.564	72.7 ± 2.52	61.7 ± 2.89	84.87

The viscosity test was conducted using a viscometer with spindle number 4 at a speed of 60 rpm. The viscosity test results indicate that the higher the concentration of the extract, the higher the viscosity. A viscosity that is too high will make the liquid soap difficult to pour, while a viscosity that is too low will make the liquid soap easier to pour and will run out quickly. The general standard for the viscosity of liquid soap products is 400 – 4000 cps. (Nurhayati et al., 2022).

The foam height test of liquid soap is conducted to determine the foaming properties produced during use. Most consumers expect soap formulations to produce foam as a sign of effective cleaning. The measure of foam formation ability can be seen from the height of the foam in the container immediately after the foam is formed. (Nafisah et al., 2022). Based on the literature review, the foam height of liquid soap ranges from 13 to 220 mm, with foam stability after 5 minutes being more than 60% (Nurhayati et al., 2022; Sukro Muhab et al., 2024). According to the table 5 the evaluation results of foam height indicate that the foam height at minute 0 ranges from 70.3 ± 0.58 to 77.7 ± 7.51, and after 5 minutes, there is a decrease ranging from 61.7 ± 2.89 to 66.0 ± 7.94, with foam stability between 84.87% and 89.61%.

Determination of Antioxidant Activity of Cocoa Extract Liquid Soap (Theobroma cacao L.)

Determination of the antioxidant activity of cocoa extract liquid soap (*Theobroma cacao* L.) was carried out using the DPPH method using a UV-Vis spectrophotometer. The principle of the DPPH method is H atom in the antioxidant compound interacts with free electrons in the radical compound. This results in the conversion of free radicals (diphenyl picryl hydrazyl) into non-radicals (diphenyl picryl hydrazine). The presence of antioxidants reduces free radicals, then the color of the solution changes from purple to yellow (Apriyandi, 2022).

Calculation of %inhibition is used to determine antioxidant activity. The smaller the concentration is the smaller % the inhibition. Each soap formulation is plotted with the relationship between concentration (X-axis) and %inhibition (Y-axis) resulting in a line equation y = bx + a. Formulas F1, F2, F3, F4, and F5 have IC_{50} values shown in Table 6.

Table 6. IC_{50} Value of Cocoa Extract Liquid Soap (*Theobroma cacao* L.)

Formulation	IC_{50}
1	84,36
2	73,71
3	66,29
4	63,28
5	44,56

Antioxidant activity is determined by the IC_{50} value. The smaller value IC_{50} made the antioxidant more stronger. From the Table 6 can be seen if in the manufacture of liquid soap with the addition of cocoa extract (*Theobroma cacao* L.) which is increasingly causing stronger antioxidant properties. The strongest antioxidant is shown in formula F5 (addition of cocoa extract as much as 4%), which has an IC_{50} value of 44.5. Antioxidants found in the body can help from free radicals which are the cause of damage to body cells. Antioxidants can also prevent anti aging by regenerating cells that have died or been damaged. The skin which is the outermost part of the body is very susceptible to free radicals so it requires antioxidants packaged in cosmetic products (Hardiyanthi, 2015).

CONCLUSIONS

The liquid soap produced from cocoa pod husk extract has good physical properties in terms of homogeneity, organoleptic characteristics, pH, viscosity, specific gravity, and foam height. The liquid soap made from cocoa pod husk exhibits antioxidant activity, with the IC_{50} value decreasing as the extract concentration increases. We can further develop liquid soap from cocoa pod husk extract into a formulation with added value.

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