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BETEL LEAF EXTRACT AS AN ANTI-BACTERIAL AGENT IN SOLID SOAP FORMULATION AND CHARACTERISATION

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Abstract: Used cooking oil is a waste oil derived from plants used as cooking oil. Used cooking oil can be reused by a purification process and used in soap making by a saponification process using a base (KOH or NaOH). Based on this, waste cooking oil is handled as a raw material for making solid soap. This study aimed to characterize solid soap from used cooking oil with betel leaf extract and explain the anti-bacterial ability of solid soap from used cooking oil with betel leaf extract. Solid soap is made by purifying used cooking oil by immersion in 15% NaOH and adding bleaching earth, making betel leaf extract by maceration method. The process of making solid soap with the addition of stearic acid, propylene glycol, glycerin, ethanol, and TEA. Characterization of solid soap by testing water content, amount of fatty acid, free alkaline, free fatty acid, mineral oil, foam stability, acidity test (pH), and anti-bacterial test. Testing the water content and the number of fatty acids shows that all samples did not meet the standards required by SNI 06-3532-1994. All samples in the free alkaline test (except sample F0), free fatty acids, mineral oil, and foam stability were tested to meet the standards required by SNI 06-3532-1994, and the anti-bacterial test of sample F4 has the best anti-bacterial properties because it can reduce the growth of bacteria compared to other samples.

Keywords: *Betel Leaf, Used Cooking Oil, Solid Soap*

INTRODUCTION

Clean culture reflects people's attitudes and behavior in maintaining and maintaining personal and environmental hygiene to avoid diseases, bacteria, and germs [1]. Soap is a means to cleanse oneself from dirt, germs, and other things that make the body dirty.

Soap is amphiphilic, with hydrophobic (polar) and hydrophobic (non-polar) groups. Soap can bind dirt and fat molecules and dissolve them in water [2], [3]. Soap making generally uses ingredients in the form of oil and alkali through a saponification process[4], [5]. Saponification reaction (saponification) using alkali is the reaction of triglycerides with alkali (NaOH or KOH), which produces soap and glycerin. Solid soap uses sodium hydroxide (NaOH), while liquid soap uses potassium hydroxide (KOH) as an alkali [6].

One of the ingredients that can be used in soap-making is cooking oil. Used cooking oil is waste oil derived from plants used as cooking oil. Based on BPS data (2019), consumption of palm cooking oil in 2018 reached 10.79 liters/capita/year. Palm cooking oil consumption in 2019 and 2020 increased by 11.09 and 11.38 liters/capita/year, respectively [7].

Betel leaves contain saponins (as anti-microbial), flavonoids, polyphenols, and essential oils. In addition, betel leaves have a distinctive aroma because they contain 1-4.2% essential oil, water, protein, fat, carbohydrates, calcium, phosphorus, vitamins A, B, and C, iodine, sugar, and starch [4] and contain antioxidant activity in the ethanol extract of green betel leaves which has an IC50 of 2.0375 µg/ml which is included in the robust category[5]. Based on this, the best solution for handling used cooking oil waste is to make solid soap using used cooking oil with betel leaf extract.

Based on this, the handling of used cooking oil waste is carried out by using used cooking oil in the process of making solid soap. The manufacture of used cooking solid oil soap is carried out by adding betel leaf extract, which aims to improve the quality of the soap both physically and with benefits.

This study focused on the optimal value of additional betel extract in making solid soap from used cooking oil. This study aims to characterize solid soap from used cooking oil with betel leaf extract based on SNI 06-3532-1994 and to explain the anti-bacterial ability of used cooking solid oil soap with betel leaf extract.

RESEARCH METHODS

The tools used in this project are measuring cups, a beaker, a petri dish, an analytical balance, an electric oven, stirrers, a thermometer, Erlenmeyer, a glass funnel, filter paper, a pipette, a filter, a hot plate, and soap mold. The materials used in this final project are used cooking oil and betel leaves. Besides that, chemicals are also used, namely 30% NaOH, Glycerin, Stearic Acid, Propylene Glycol, TEA, and 96% Ethanol.

Manufacturing Stage

1. Refining Used Cooking Oil

The first step is to make a NaOH solution with a concentration of 15% (15 grams of NaOH dissolved in 100 ml of distilled water). After that,

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the filtered cooking oil is heated at 40°C, and 15% NaOH solution is added. Then the mixture is stirred using a stirrer for 10 minutes then filtered again with filter paper [6].

The second stage is heating the neutralized cooking oil to a temperature of 70°C, weighing the bleaching earth (bleaching), and putting it into the neutralized cooking oil solution. Bleaching earth (bleaching) removes unwanted dyes in oil using adsorbents (bleaching earth, activated clay, or activated charcoal). The solution was stirred with a stirrer for 10 minutes and then heated at 150°C. Then it is filtered again using filter paper so that the purified used cooking oil is ready to be used in making soap [2].

2. Preparation of Betel Leaf Extract

Betel leaf extract is made by the maceration method (soaking). *Maceration* is a simple extraction method by soaking the material in a solvent for several days at room temperature and protected from light. This process is used to extract betel leaf, which was chosen because the tool used is simple and suitable for compounds that are not heating-resistant [3].

Wash freshly picked betel leaves, air dry until no water sticks, then weigh the betel leaves. The maceration process is carried out for two days; 200 grams of betel leaves are used with 700 mL of 70% alcohol solvent [7].

3. Soap Making Process

Prepare the tools and materials that have been provided, and weigh the ingredients according to a predetermined dosage, first 10 grams of oil are heated to a temperature of 40ºC, then add stearic acid, stir until dissolved, and add NaOH until it clots, then add propylene glycol, stir until it is diluted, then add glycerin. and TEA stirred until it looks foamy then added ethanol and betel leaf extract stirred until frothy, finally printed.

Table 1. The formulation in Solid Soap Making

Materials (g)	F0	F1	F2	F3	F4
Waste Cooking Oil	10	10	10	10	10
Betel leaf extract	-	1,5	3	4,5	6
NaOH30%	13.2	13.2	13.2	13.2	13.2
Stearic acid	4.8	4.8	4.8	4.8	4.8
Propylene glycol	5	5	5	5	5
Glycerin	7.8	7.8	7.8	7.8	7.8
TEA	1	1	1	1	1
Ethanol 96%	9	9	9	9	9

Testing Stage

1. Water Content Test

An empty petri dish was dried at 105ºC for 30 minutes, the cup was cooled in the desiccator for ± 15 minutes, and then the cup was taken from the desiccator and weighed with an analytical balance. Some of the samples were put in a cup and weighed, then put into the oven with a temperature of 105ºC for 2 hours; the cup was then cooled in a desiccator for 15 minutes, then weighed, and recorded the results and calculated the water content using the formula:

$$(\%)\text{Water Content} = \frac{A - B}{C} x\ 100\%$$

Explanation:
A = sample weight before heating (g)
B = sample weight after heating (g)
C = sample weight (g)

2. Fatty Acid Amount Test

10 grams of sample put in a beaker, added with 100 ml of distilled water and heated on a hotplate 2 drops of methine orange and 20% H_2SO_4 until it turns red. The mixture was stirred for a while, then covered using a watch glass. This solution was observed until it formed 2 layers, then 10 grams of paraffin was added and waited until the entire mixture was clear; after that, it cooled over a basin filled with water and given a stirrer to make it easier to take out the solidified results, and finally, the results are weighed. The formula for calculating the number of fatty acids is as follows:

$$\%\ \text{FAN} = \frac{\text{wax cake} - \text{paraffin } weight}{\text{sample weight}}\ x\ 100\%$$

*FAN= Fatty Acid Number

3. Free Alkali

Free alkali examination was carried out by weighing 5 grams of soap sample into an Erlenmeyer. Dissolve it with 100 ml of neutral ethanol and add 0.5 phenolphthalein solution as an indicator; put it in a ball-bottomed tube and add a boiling stone and then condense and change color to pink, then titrate using 0.1 N HCl solution until clear (clear color for 30 seconds) then record the volume of HCl used (V). The free alkaline calculation formula is as follows:

$$\%\ \text{Free Alkali} = \frac{\text{V x N x 0,0561}}{\text{wieght of sample}}\ \text{x}\ 100\%$$

4. Free Fatty Acid Test

Free fatty acids were examined by weighing a sample of 5 grams of soap into an Erlenmeyer. Dissolve it with 100 ml of neutral ethanol and add 0.5 phenolphthalein solution as an indicator. This solution was put in a ball-bottomed tube, added a boiling stone, condensed, and changed color to pink, then titrated using 0.1 N KOH solution until a pink color was formed (pink color persists for 30 seconds). Then record the volume of KOH used

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(V). The free fatty acid calculation formula is as follows:

$$\%\ \text{Free Fatty Acids} = \frac{\text{V x N x 0,256}}{\text{w}}\ \text{x}\ 100\%$$

5. Mineral Oil Test

The mineral oil was tested by weighing 5 grams of sample, putting it into an Erlenmeyer, adding 50 ml of water, and heating it to dissolve on a hotplate. An excess of 10% HCl was added, a few drops of red methyl orange were added, and the part that could not possibly be saponified would separate in the top layer, 0.3 mL of the top was taken and pipetted, and 0.5 N KOH added, put into Erlenmeyer and titrated with water if it occurs. Turbidity means it is positive for containing mineral oil. If the solution remains clear, it is negative for not containing mineral oil (SNI 06-3532-1994).

6. Foam Stability Test

A gram of the sample was put in a test tube containing 10 ml of distilled water, then shaken for 1 minute. The foam formed was measured using a ruler (initial foam), Foam height was measured again after 5 minutes (final foam). The foam stability calculation formula is as follows:

$$\%\ \text{Lost Foam} = \frac{\text{Initial Foam Height} - \text{Final Foam Height}}{\text{Final Foam Height}}\ x\ 100\%$$

$$\text{Foam Stability} = 100\% - \%\text{Lost Foam}$$

7. Degree of Acidity (pH)

A gram of sample was put into a glass, added with 10 ml of distilled water, and stirred until dissolved. Measuring acidity (pH) using a pH meter into the liquid and let it stand for a few moments until a constant acidity (pH) was obtained.

8. Bacterial Growth Test

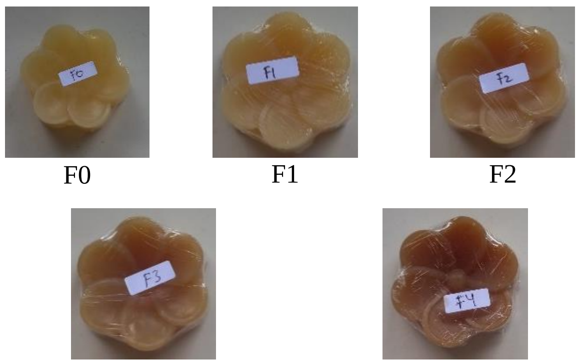
In This study, testing of bacteria was carried out by testing the ability of soap to grow bacteria. Testing is done by preparation of solid media; 23.5 grams of nutrient agar was mixed with 1000 ml of distilled water, then heated until homogeneous and the color changed slightly transparent. Then five pairs of Petri dishes were prepared, wrapped in newspaper, and sterilized with homogeneous nutrient agar using an autoclave at 121ºC for 15 minutes. After 15 minutes, please wait for it to cool slightly, pour enough nutrient agar into a petri dish, and then solidify. Then the etching is done by dividing the petri dish into four sides: quadrants I, II, III, and IV. After that, heat the loop using Bunsen, then take the sample that has been prepared and scratch it in four quadrants. Starting from the top left, took a zig-zag until it reached the middle of the quadrant, continued to quadrant II without taking samples again, and so on until quadrant IV, then left for three days and counted the number of bacteria that grew [8].

RESULTS AND DISCUSSION

Based on the results of the analysis of the water content, total fatty acid, free alkali, free fatty acid, mineral oil, foam stability, pH, and anti-bacterial are shown in Table 2.

Moisture content is the amount of water in a product or material, aiming to determine the amount of water in the solid soap itself. Based on the results of the water content test presented in Table 2, it can be seen that the water content of the sample F0 is 37%, F1 is 40.63%, F2 is 43.63%, F3 is 47.88%, and F4 is 49.12%. The water content of the soap samples (F0, F1, F2, F3, and F4) did not meet the water content quality standards. These samples did not meet the standards according to SNI 06-3532-1994, having a water content of at most 15% [9-10]. The addition of betel leaf extract influences the high-water content in soap. The more betel leaf extract, the higher the water content in the soap sample. This result aligns with another research that the water content is directly proportional to the extract added[11]. The more extract added, the higher the water content in solid soap. Besides that, adding distilled water to the processing soap can affect the water content of the preparation [12].

The test for the number of fatty acids aims to determine the total amount of all fatty acids in soap that have or have not reacted with alkali. Based on the results of testing the number of fatty acids presented in Table 2, it can be seen that the free alkali value of the sample F0 is 33.65%, F1 is 34.30%, F2 is 35.05%, F3 is 29.05%, and F4 is 32, 70%. Based on the standard according to SNI 06-3532-1994 [13], the total fatty acids have total fatty acids with a value greater than 70%. The total fatty acids from the soap samples (F0, F1, F2, F3, and F4) do not meet the quality standards for total fatty acids; this is because all of these samples have low values below the standard, perhaps due to the material used as a transparent former for the soap and the oil used is oil that has been neutralized as if it had been neutralized.



F0 F1 F2 F3 F4

Figure 1. Soap with some formulations

Table 2. Solid Soap Testing Result

Testing	F0	F1	F2	F3	F4	SNI
Water Content (%)	37.00	40.63	43.63	47.88	49.12	15
Fatty Acid number (%)	31.65	34.30	35.05	29.05	32.70	>70
Free Alkali (%)	0.16	0.13	0.11	0.09	0.07	Maks. 0,14
FFA (%)	2.05	2.05	1.54	1.54	1.54	<2.5
Mineral Oil	-	-	-	-	-	-
Foam Stability (%)	51.43	52.26	66.54	66.88	69.70	60-70
pH	10.89	10.30	10.24	10.82	10.93	8-11
Bacterial growth	~	9	7	5	1	-

~: infinite

The free alkali test aims to show the amount of alkali in soap that is not bound as a compound. The presence of alkali in soap can increase the ability to produce saponification of soap [14]. Based on the results of the free alkali test presented in Table 2, it can be seen that the sample free alkali value F0 is 0.16%, F1 is 0.13%, F2 is 0.11%, F3 is 0.09%, and F4 is 0.07 %. Based on the standard according to SNI 06-3532-1994, the maximum free alkali content is 0.14%. The highest free alkali from the F0 soap sample was 0.16% and did not meet the standard because if the alkali in the soap exceeds 0.14% for HCl, then this indicates that this alkali has harsh properties and can cause irritation to the skin and cause the skin to dry. The free fatty acid test results on F1, F2, F3, and F4 met the standards, and the alkali in soap does not have a harsh nature, so it does not cause irritation to the skin. The more betel leaf extract added, the lower the free alkaline value produced. Because betel leaf extract contains phenol, phenol is an alcoholic compound that functions as a natural antioxidant that dissolves in acetone, alcohol, glacial acetic acid, and alkaline hydroxide. Phenol dissolves in alkaline hydroxide, which causes the addition of green betel leaf extract to the manufacture of solid soap, which can cause a reduction in alkali because it dissolves well with alkali in soap [6].

The fatty acid test aims to determine the free fatty acids in the soap but not bound as sodium or triglyceride compounds (mineral fat). Based on the free fatty acid test results presented in Table 2, it can be seen that the free fatty acid value of the sample F0 is 2.05%, F1 is 2.05%, F2 is 1.54%, F3 is 1.54%, and F4 is 1. .54%. SNI 06- 3532-1994 shows that the maximum free fatty acid content is <2.5. The highest free fatty acids from the soap samples were F0 and F1, namely 2.05%. The free fatty acid content in the soap could be affected by mixing too little or too much NaOH so that in the saponification process, the number of fatty acids bound to NaOH is small and large, Exists in free form. If the free fatty acid content is high in the soap, the tendency for the soap to smell rancid will be even greater [15]. The free fatty acid test results at F0, F1, F2, F3, and F4 met the standard, which is below 2.5%, which means that the fatty acid content is not high and does not cause the soap to smell rancid and is not bound with a little or too much NaOH.

Mineral oil is a substance or permanent ingredient as oil, but when water is added, an emulsion between water and oil will occur [16], characterized by turbidity. Based on the results of the mineral oil testing presented in Table 2, the mineral oil values of the samples (F0, F1, F2, F3, and F4) are negative. Based on SNI 06-3532-1994, mineral oil in solid body soap should not be more than 0.05%, indicated by the absence of turbidity when titrated with water. A good result is a negative one. The mineral oil values in the samples (F0, F1, F2, F3, and F4) met the standards because they were under the applicable mineral oil standards, namely no turbidity or clarity.

Foam stability is the ability to retain gas for a certain time. Foaming ability can be seen from the increase in volume after the gas is fed into the solution. The foam stability test aims to determine the stability of the foam produced by solid soap [14]. Based on the results of the foam stability test presented in Table 2, it can be seen that the stability value of sample F0 is 51.43%, F1 is 52.26%, F2 is 66.54 %, F3 is 66.88%, and F4 is 69.70%. Based on the standard provisions for good foam stability, which ranges from 60-70%. The foam stability values in the samples (F0 and F1) did not meet the standards, while the samples (F2, F3, and F4) did meet the standards because they complied with the applicable foam stability standards. The high foam stability is directly proportional to the amount of extract added; the more betel leaf extract added, the higher the foam stability value produced.

The acidity or power of hydrogen (pH) is the degree of acidity used to express the level of acidity or alkalinity of a solution. Testing the degree of acidity (pH) aims to determine the pH value of soap so that it complies with standards and does not irritate the skin (between 8-11) [17-18].

Based on the pH test results in Table 2, the pH value of the sample F0 is 10.89, F1 is 10.30, F2 is 10.24, F3 is 10.82, and F4 is 10.93. The acidity (pH) values in the samples (F0, F1, F2, F3, and F4) have met the quality standards required by SNI 06-3532-2016 [19]. It is because the standard pH value has been set, which is between 8-11 [10].

Anti-bacterial are compounds used to control the growth of harmful bacteria [18]. Controlling the growth of microorganisms aims to prevent the spread of disease and infection and prevent spoilage and destruction of materials by microorganisms. Based on the results of the anti-bacterial test presented in Table 2, it can be seen that the value of the number of bacteria growing in the F0 sample is infinite, F1 is 9 colonies, F2 is 7 colonies, F3 is 5 colonies, and F4 is single/one colony. Based on these results, the highest value for bacterial growth was obtained, F0, because there was no addition of betel leaf extract, so it was very easy for bacteria to grow. The lowest value for bacterial growth, F4, was obtained, namely singular, which means that the more betel leaf extract used, the fewer bacteria will grow, so the characteristic resulting anti-bacterial is stronger. The ability to inhibit anti-bacterial growth in used cooking oil soap was influenced by the addition of betel leaf extract. It is because the content of phenolic compounds in betel leaves can promote bacterial growth [21-23].

CONCLUSION

Characterization of solid soap with the addition of betel leaf extract is obtained by testing the water content and number of fatty acids. All samples did not meet the standards required by SNI 06-3532-1994. The free alkali testing of samples F1, F2, F3, and F4 had met the standards set required by SNI 06-3532-1994, testing for free fatty acids, mineral oil, and foam stability, all samples met the standards required by SNI 06-3532-1994, and the anti-bacterial test for sample F4 had the best anti-bacterial properties because it was able to minimize the growth of bacteria compared to another sample. Based on this, the soap with the F4 sample has the best formulation. The best anti-bacterial ability of solid soap is sample F4. This sample contains the highest betel leaf extract and can inhibit bacterial growth the best compared to other samples. Phenol content in betel leaf extract inhibits the growth of bacteria.

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