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FORMULATION AND EVALUATION OF NUTMEG LEAF EXTRACT (Myristica fragrans Houtt.) TRANSPARENT SOLID SOAP

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A hstract

Introduction: Soap is a cosmetic that can act as a means of skin beauty, by keeping the skin supple, smooth and not dry. Soap is also known as a body cleansing tool to minimize the onset of symptoms or diseases that can grow on the skin area. The purpose of this study was to formulate and evaluate the transparent soap of nutmeg leaf extract with three variations of the formula, namely the concentration of nutmeg leaf extract 0,5%, 1%, and 1,5%. **Method:** The evaluation test treatment for the preparation was in the form of organoleptic tests, pH, foam strength, water content, free alkali and free fatty acids to show soap with the best evaluation test results in table data according to SNI standards. **Results:** The results showed that each formulation produced stable dosage forms with various extract concentrations of 0,5%, 1%, and 1,5%. **Conclusion:** So, the best results were obtained in formulation 1, with the pH test getting the most stable results with an average of 9.6, the highest foam height with an average of 127 mm, the value of water content does not exceed the requirements, namely on an average of 12,6%, free alkali with the lowest value with an average of 0,08%, and free fatty acid test with an average of 0%, which means that free fatty acid content is not detected.

Key words: Extract, Myristica fragrans Houtt., Nutmeg Leaf, Transparent Solid Soap

INTRODUCTION

Soap is a cosmetic product that can play a role in maintaining skin beauty and skin health. This is starting to become a very important aspect with the increasing market demand for beauty and skin health products, one of which is transparent solid soap products (Ainiyah & Utami, 2020). Transparent soap as a charming and transparent appearance, with a softer density, making the skin more moist, and made using natural ingredients to nourish the skin and can prevent premature aging. The use of transparent solid soap is very suitable for use among teenagers and adults, especially for skin beauty use (Ramadian et al., 2019). Factors that affect the transparency of soap lies in the content of alcohol, sugar, and glycerin which have a role to clarify soap (Widyasanti et al., 2016). Transparent solid soap is made with the help of active substances from safe natural ingredients, the natural ingredients used are nutmeg leaves which contain secondary metabolites in the form of alkaloids, flavonoids, triterpeneoids and tannins (Anggriani et al., 2018). Nutmeg leaves can act as an antibacterial in the content of flavonoid and terpenoid compounds. Antibacterial can make soap as a cleaning tool that can minimize the onset of symptoms or diseases that grow on the skin area (Arrizqiyani et al., 2018). Pratiwi, 2019 say in the manufacture of liquid soap using the active substance of nutmeg leaf extract with a concentration of 0,5% found the inhibition zone of S. aureus bacteria on agar media of 10-20 mm. It is stated that nutmeg leaf extract can function in inhibiting bacteria. The greater the concentration of nutmeg leaf extract, the better it will be in inhibiting bacteria (Moningka et al., 2020). Based on several previous studies and seeing the advantages of transparent solid soap preparations, researchers are interested in conducting research on the formulation and evaluation of transparent solid soap from 96% ethanol extract of nutmeg leaves (Myristica fragrans Houtt.) using various extract concentrations of 0,5%, 1%, and 1,5%.

METHOD

A.Qualitative Test of Nutmeg Leaf Extract

1. Alkaloid Test

The extract was taken as much as 0,1 g and added 5 ml of chloroform and 3 drops of ammonia. Divided into 3 soaps, each added with *Mayer*, *Wargner*, and *Dragendorf* reagents. The alkaloids in the

sample will be characterized by the presence of a white precipitate in the *Mayer* reagent, an orange precipitate in the *Dragendorf* reagent, and a brown precipitate in the *Wagner* reagent (Pratiwi *et al.*, 2019).

2. Flavonoid Test

0,1 g of extract was taken and put into a test tube, 0,1 g of magnesium powder and 0,4 ml of amyl alcohol were added to the test tube. Add 4 ml of alcohol and mix until homogeneous. A positive reaction is indicated by the formation of a red, yellow or orange color (Pratiwi *et al.*, 2019).

3. Tanin Test

The extract was put into a test tube. Add FeCl₃ until a green-blue color is formed. The color is a marker of the presence of tannin compounds in the sample (Pratiwi *et al.*, 2019).

4. Triterpenoid Test

0.1 g of the extract sample was taken, then 3 drops of concentrated HCl and 1 drop of concentrated H₂SO₄ were added. A positive trirepenoid is indicated by the formation of a red color (Pratiwi *et al.*, 2019).

B.Transparent Solid Soap Preparation Formula

Table 1. Transparent Solid Soap Formulation of Nutmeg Leaf Extract (Widyasanti et al., 2016)

	Material –	Formula			
No	Composition	I(g)	$\Pi(\mathbf{g})$	III(g)	Function
1.	Nutmeg Leaf Extract	1	2	3	Active Subtance
2.	Coconut Oil	40	40	40	Oil Phase
3.	Stearic Acid	14	14	14	Emulsifisier
4.	NaCl	0,4	0,4	0,4	Tonicity Agent
5.	NaOH 30%	40,6	40,6	40,6	Buffer Agent
6.	Glycerin	26	26	26	Preservative
7.	Coco-DEA	2	2	2	Foam Stability
8.	Fragrance Oil	0.2	0.2	0.2	Fragrance
9.	Sugar	30	30	30	Humectans
10.	Ethanol 96%	30	30	30	Solvent
11.	Aquadest	15,8	14,8	13,8	Solvent

C.Preparation of Transparent Solid Soap

The process of making soap using water bath media with the hot method. Pure coconut oil that has been placed in a glass beaker is heated through a water bath. Add stearic acid, then stir until homogeneous. After homogenization, the 30% NaOH solution was added and other supporting materials such as 96% ethanol, glycerin, sugar syrup (sugar + liquefied aquadest), coco-DEA, NaCl and fragrance oil. Stirring is done until the dough looks mixed evenly. The addition of nutmeg leaf extract with the temperature of the dough lowered first until it reaches 70°C. After that, the mixture was stirred until the extract was completely mixed. Then it is poured into the prepared silicone soap mold. Let the dough rest at room temperature for 24 hours (Widyasanti et al., 2016).

D.Transparent Solid Soap Characteristic Test

1. Organoleptic Observations

Organoleptic tests include observations of shape, color changes, and odor changes (Susanti & Guterres, 2018).

2. pH Test

The pH test was tested by weighing 1 g of soft soap dissolved in 9 ml of distilled water. Then the pH marker is dipped and the pH value of soft soap is observed, the pH of soft soap is 9-11(Susanti & Guterres, 2018).

3. Foam Strength Test

The foam strength test was tested by weighing 10 g of soft soap dissolved in aquadest. After that, shake vigorously and measure the amount of foam that is formed after shaking for 30 seconds and 60 seconds (Susanti & Guterres, 2018).

4. Water Content Test

The water content test was carried out using a moisture analyzer. The tool is first turned on, then press the mode button and select the mode you want to use. Open the cover on the device so that the display status will change. Insert it into an empty aluminum pan that has been cleaned, making sure the pan is in the correct position. The cover on the tool is closed again until the towering is done automatically. When it is tare, open it again and add 1g of soap sample then close the tool again. The appliance will start heating the soap sample until drying is complete. If the display indicator is green, the sign has shown constant results to determine the weight of the sample after drying (Febriani et al., 2021).

5. Free Alkali Content Test

5g of solid soap was dissolved using 10 ml of distilled water, then bromthymol blue was added and titration was carried out using 0,1 N NaOH until the color changed from blue to yellow. The test was carried out 3 times replication. Determination of free fatty acid content can be calculated using the formula:

$$\frac{V \times N(BS) \times BM}{W \times 1000} \times 100\%$$

Information:

V : Volume of NaOH during titration

N : Normality of NaOH

BM : Molecular weight of free fatty acids

Weight of soap preparation (Susanti & Guterres, 2018).

6. Free Fatty Acid Content Test

5g of solid soap is weighed into a measuring cup. Then 10 ml of distilled water was added and phenolphthalein marker was added. After the solution shows a pink color, it is titrated using 0.1N HCl solution until the red color change disappears. Determination of free fatty acid content can be calculated using the formula:

$$\frac{V \times N(BS) \times BM}{W \times 1000} \times 100\%$$

Information:

V : Volume of HCl during titration

N : Normality of HCl

BM : Molecular weight of free fatty acids

Weight of soap preparation (Susanti & Guterres, 2018).

RESULTS

A.Qualitative Test of Nutmeg Leaf Extract

Phytochemical screening carried out in this study was in the form of tests such as alkaloids, flavonoids, tannins and triterpenoids. The results of some of these tests yielded positive results in all four tests. The results of phytochemical screening can be seen in full in table 2.

Table 2. Qualitative Test Results of Nutmeg Leaf Extract

Table 2. Qualitative less results of realing Bear Extract						
Sample Name	Keadaan Sampel	Parameter		Hasil	Teknik Analisi	
			Mayer	Positive	White Precipitate	
		Alkaloid	Wagner	Positive	Brown Precipitate	
Nutmeg	Thick		Dragendroff	Positive	Orange Precipitate	
Leaf	Lear Extract	Flavonoid		Positive	Make Orange Color	
Extract		Tanin	FeCl ₃ 1%	Positive	Make Blackish Green Color	
		-	Triterpenoid		Positive	Brownish Red Precipitate

1. Transparent Solid Soap Evaluation Test Results

Table 3. Results of Organoleptic Observations of Transparent Solid Soap

Formula	Replication	Color	Odor	Appearance
	1	Light Brown	Aromatic	Solid
F1	2	Light Brown	Aromatic	Solid
	3	Light Brown	Aromatic	Solid
	1	Light Brown	Aromatic	Solid
F2	2	Light Brown	Aromatic	Solid
	3	Light Brown	Aromatic	Solid
	1	Dark Brown	Aromatic	Solid
F3	2	Dark Brown	Aromatic	Solid
	3	Dark Brown	Aromatic	Solid

Information:

F1 : Transparent solid soap with 0,5% nutmeg leaf extract concentration

F2 : Transparent solid soap with 1% nutmeg leaf extract concentration

F3 : Transparent solid soap with 1,5% nutmeg leaf extract concentration

If seen in table 3, the results obtained from organoleptic tests of transparent solid soaps with the three formulations using various extract concentrations. The results in Formula 1 and Formula 2 get a light brown color from transparent soap, with a solid form and a distinctive aromatic aroma. Likewise, the third formulation produces transparent soap with a distinctive aromatic aroma and solid shape but with a darker color. This is due to the increasing concentration of the extract used.

2. Transparent Solid Soap pH Test Results

Table 4. pH Test Results

	Tuble 4. pli Test Results					
Replication	F1	F2	F3			
1	9,9	9,7	9,7			
2	9,4	9,8	9,7			
3	9,5	9,5	9,6			
Average ± SD	9.6 ± 0.26	9.7 ± 0.15	9.7 ± 0.56			

Information:

F1 : Transparent solid soap with 0,5% nutmeg leaf extract concentration

F2 : Transparent solid soap with 1% nutmeg leaf extract concentration

F3 : Transparent solid soap with 1,5% nutmeg leaf extract concentration

Table 4 shows the results of the pH test on transparent solid soap preparations of nutmeg leaf extract (*Myristica fragrans* Houtt) from each formulation with an average pH value in the range from 9.6 to 9.7.

3. Transparent Solid Soap Foam Strength Test Results

Table 5. Foam Strength Test Results

Table 5. Foam Strength Test Results							
Replication	Replication F1 F2 F3						
1	120 mm	120 mm	120 mm				
2	110 mm	130 mm	110 mm				
3	150 mm	90 mm	120 mm				
Average ± SD	127 ± 2,08 mm	113 ± 2,08 mm	$117 \pm 0.58 \text{ mm}$				

Information:

F1 : Transparent solid soap with 0,5% nutmeg leaf extract concentration

F2 : Transparent solid soap with 1% nutmeg leaf extract concentration

3 : Transparent solid soap with 1,5% nutmeg leaf extract concentration

The results on the foam strength test of the transparent solid soap preparations of nutmeg leaf extract (*Myristica fragrans* Houtt) can be seen in table 5. The results show that each formulation has foam height in the foam height value in the range of 113-127 mm.

4. Transparent Solid Soap Water Content Test Results

Table 6. Water Content Test Results

Replikasi	F1	F2	F3
1	14,7%	10%	5,7%
2	12,6%	9,4%	6,3%
3	10,5%	10%	5,7%
Rata-Rata ± SD	12,6 ± 2,12%	9,8 ± 0,34%	5,9 ± 0,36%

Information:

F1 : Transparent solid soap with 0,5% nutmeg leaf extract concentration

F2 : Transparent solid soap with 1% nutmeg leaf extract concentration

: Transparent solid soap with 1,5% nutmeg leaf extract concentration

The results of the water content test of transparent solid soap preparations of nutmeg leaf extract (Myristica fragrans Houtt) can be seen in table 6. The results of each formulation get an average value of water content in the range of 5.9 - 12.6%.

5. Transparent Solid Soap Free Alkali Content Test Results

Table 7. Free Alkali Content Test Results

Table 7.11 to Main Content Test Results						
Replication F1 F2 F3						
1	0,07%	0,11%	0,11%			
2	0,10%	0,14%	0,10%			
3	0,08%	0,12%	0,08%			
Average ± SD	$0.08 \pm 0.02\%$	$0.12 \pm 0.02\%$	$0.10 \pm 0.02\%$			

Information:

F1 : Transparent solid soap with 0,5% nutmeg leaf extract concentration

2 : Transparent solid soap with 1% nutmeg leaf extract concentration

F3 : Transparent solid soap with 1,5% nutmeg leaf extract concentration

Table 7 shows the results in the form of free alkali data which is still contained in the transparent solid soap preparation of nutmeg leaf extract (*Myristica fragrans* Houtt). The results showed that the free alkali content of each formulation obtained a concentration value with an average range of 0.08 - 0.12%.

6. Transparent Solid Soap Free Fatty Acid Content Test Results

Table 8. Free Fatty Acid Content Test Results				
Replication	F1	F2	F3	
1	0%	0%	0%	
2	0%	0%	0%	
3	0%	0%	0%	
Average ± SD	0%	0%	0%	

Information :

F1 : Transparent solid soap with 0,5% nutmeg leaf extract concentration

F2 : Transparent solid soap with 1% nutmeg leaf extract concentration

F3 : Transparent solid soap with 1,5% nutmeg leaf extract concentration

The results of the free fatty acid test on transparent solid soap preparations of nutmeg leaf extract (*Myristica fragrans* Houtt) can be seen in table 8. The results obtained in each formulation get a free fatty acid content value of 0%, which means that free fatty acids are no longer found. contained in soap preparations.

DISCUSSION

In this study, three transparent solid soap formulations were made with various extract concentrations of 0,5%, 1% and 1,5%. It aims to obtain a formula with the best results seen from the physical stability of the preparation. This research was carried out in several stages, namely preparation of nutmeg leaf samples, nutmeg leaf extraction, phytochemical screening of nutmeg leaf extract, formulation and evaluation of transparent solid soap preparations of nutmeg leaf extract. The evaluation carried out in the form of organoleptic test, pH test, foam strength test, water content test, free alkali test and free fatty acid test. Nutmeg leaf extract was made using the maceration method starting from nutmeg leaf simplicia powder which was macerated with 96% ethanol for 3x24 hours. The maceration method was chosen because it has the advantages of simple, easy-to-reach and relatively inexpensive equipment and working methods. Secondary metabolic compounds such as flavonoids are easily damaged when using high-temperature heating, so this maceration method was chosen to avoid damage to compound

components on heating. The choice of the 96% ethanol solvent was based on the degree of safety and ease of evaporation. Its properties are able to dissolve almost all substances, whether polar, semipolar, nonpolar, and can attract flavonoid and phenolic compounds optimally (Ramadhani *et al.*, 2020).

Phytochemical screening of the thick extract aims to determine the presence of secondary metabolites in the sample using color reagents to see what is contained in the extract preparation (Ramadhani *et al.*, 2020). Anggriani (2018) say that nutmeg leaves contain secondary metabolites in the form of alkaloids, flavonoids, triterpeneoids and tannins. The alkaloid test was carried out by dissolving the extract using HCl and adding three reagents. The use of HCl aims to extract the alkaloid content of the sample. The addition of three reagents (*Mayer*, *Wagner*, and *Dragendorff*) to identify the presence of alkaloid compounds was characterized by a precipitation reaction (Iskandar, 2020). The precipitation reaction occurs because the nitrogen atom has a lone pair of electrons on the alkaloids replacing the iodine ion in the reagent (Nurlani & Situmorang, 2021).

Alkaloid test using *Mayer*'s reagent got positive results in the presence of a white precipitate. White-yellow precipitate on the addition of *Mayer*'s reagent because nitrogen in the alkaloids will react with metal ions K+ from K2[HgI4] (potassium tetraiodomercurate (II)) to form potassium-alkaloid complexes which precipitate The alkaloid test using *Wagner*'s reagent got a positive result in the form of a brown precipitate. Wagner's reagent is a mixture of iodine (I2) with KI. The brown precipitate comes from the bond between the nitrogen atom (N) which has a lone pair of electrons on the alkaloid to the metal ion K+ to form a potassium-alkaloid complex compound. Positive alkaloid test with the addition of *Dragendorff*'s reagent will form an orange precipitate. *Dragendorff* reagent makes nitrogen in alkaloids form coordinate covalent bonds with K+ which is a metal ion so that an orange precipitate can be formed (Wahyuni & Marpaung, 2020).

The flavonoid test was carried out by adding magnesium (Mg) powder and concentrated HCl. Positive results of flavonoids with the formation of an orange color. The addition of concentrated HCl breaks the glycoside bonds of flavonoids, the glycoside bonds with flavonoids in the extract must be broken first by reducing the bond with magnesium powder, thus forming an orange color indicating the presence of flavonoids in the sample (Iskandar, 2020). The tannin test got the results that formed a blackish green color. Positive in the tannin test will be indicated by the appearance of a blue green blackish color (Pratiwi *et al.*, 2019). The extract was dissolved using aquadest, because tannins are polar phenolic compounds. The addition of FeCl₃ which reacts with the tannin compound will find a mechanism for the formation of a green-blue color as a sign of the presence of a phenol group contained in the sample, so that the identification results show that it contains positive tannins. In the triterpenoid test, the sample was dripped with concentrated HCl and concentrated H₂SO₄, producing a brownish red color. The addition of concentrated HCl was used to attract the triterpenoid content in the sample, and the addition of concentrated sulfuric acid to the triterpenoid compound resulted in the formation of a red color that occurred (Iskandar, 2020).

In soap making, a saponification reaction will occur which is the process of breaking the triglyceride chain in the form of oil or fat using alkaline compounds. The alkaline compound used is in the form of NaOH base where NaOH is suitable to produce solid soap products, and can be seen in the characteristics of NaOH which is difficult to dissolve or not easily soluble in water. This saponification process will produce a product in the form of solid soap and a byproduct in the form of glycerol (Agustini & Winarni, 2017).

Organoleptic test is one of the physical parameters to see changes in color, shape, and odor changes in transparent solid soap preparations (Susanti & Guterres, 2018). In formula 1 using 0,5% extract, it is obtained transparent soap with a light brown solid form with a distinctive aromatic odor derived from fragrance oil. Formula 2 using 1% extract produces a transparent soap in the form of a solid with a light brown color with a distinctive aromatic odor. In formula 3 using 1,5% extract, you get solid soap with a distinctive aromatic smell, and dark brown in color with less visible transparency, this is because the more extracts used, the darker the soap will be and the transparency will also look better (Pratiwi *et al.*, 2019).

The pH test is one of the important parameters used to determine the resulting soap preparation. The results from the average pH range of 9,6-9,7 are known to get the results in table 4 in accordance with the

requirements of SNI 06-3532-1994 soap pH ranges from 9-11 (BSN, 2016). Soaps that have a pH value that is too high or too low can increase the absorption capacity of the skin, whichh can cause skin irritation such as sores, itching and peeling (Fanani *et al.*, 2020). The high and low pH of soap can be influenced by the saponification process and the composition of the formula used when making soap. When you find a high pH value, it can be overcome by adding fat or oil. The amount of alkali present in soap can also affect the level of pH. The more alkaline content, the higher the pH of the soap (Setiawati & Ariani, 2021).

The foam strength test aims to determine the amount of foam produced by transparent solid soap preparations. This test is carried out by measuring the height of the foam produced on each soap. The results in table 5 show that the strength of the foam obtained has met the requirements of the foam height, namely 13 - 220 mm according to the requirements of SNI 06-3532-1994 (BSN, 2016). High low foam can be experienced due to a factor, namely when shaking the soap. The characteristics of the foam are influenced by the presence of materials that function as surfactants or foam stabilizers, the foam stabilizer used in this formulation is the role of Coco – DEA (Rosi et al., 2021).

The water content test is carried out to find out how much water content is in solid soap preparations. The results in table 6 find quite good results, from each replication and the average results do not exceed the required limit of water content, which is 15% in accordance with the requirements of SNI 06-3532-1994 (BSN, 2016). The water content is said to affect the level of hardness of the soap preparation. The higher the water content, the softer the soap will be. Vice versa, the lower the water content produced, the harder the soap will be. In addition, the amount of water in soap can affect the storage of soap. If it produces soap with a high moisture content or more than 15% in open storage and in contact with air, it can cause the soap to shrink from the weight of the soap. Soap preparations with a good water content value will increase their storage period or are relatively long, and will be more efficient in use because soap will not dissolve easily in water (Elmitra & Noviyanti, 2020).

Free alkali test was carried out to determine the presence of excess free alkali. The results in table 7 show that the levels of free alkali still meet the requirements of the free alkali test in accordance with the requirements of SNI 06-3532-1994, which is not more than 0,14% (BSN, 2016). Free alkali is an alkali that does not react with fatty acids during the formation of soap preparations. Free alkali has a maximum content of 0,14% in soap. The analysis on free alkali aims to find out how much or how much alkali metal is not saponified, because it is feared that it will have an impact on the quality of the soap produced (Sa'diyah *et al.*, 2018). Alkali is known to have a hard nature, so that if the free alkali level produced is too high it can irritate the skin, and can cause the skin to become dry. This is because sodium hydroxide is hygroscopic so it can cause absorption of skin moisture quickly (Fanani *et al.*, 2020).

The free fatty acids in soap are part of the fatty acids that are not bound to the alkali. The results of the evaluation of the free fatty acids in table 8 did not state that the presence of free fatty acids was detected. This is indicated by the absence of a color change that is produced. As happened in the study of Hardian *et al.*, (2016) which stated that the assessment of the free fatty acid levels in transparent solid soap was considered undetectable. It is suspected that the free fatty acid content is considered non-existent because the free fatty acid in the preparation has reacted with the base to form soap and glycerol. The free fatty acid test on soap preparations is not expected to produce a high value of not more than 2,5% in accordance with the requirements of SNI 06-3532-1994. Because it will reduce the binding power of soap to dirt, oil, fat or sweat (BSN, 2016). Temperatures that are too high and heating times that are too long can cause damage and will form free fatty acids again, so that the levels of free fatty acids will be higher (Tobing *et al.*, 2021). High free fatty acids can also affect the formation of foam, the resulting foam will be less because there are still free fatty acids that do not react with NaOH in the soap making process (Prasetiyo *et al.*, 2020). Free fatty acids are associated with the odor produced in soap, if the free fatty acids exceed the standard soap will smell rancid and can inhibit the process of cleaning the skin surface by soap (Fanani *et al.*, 2020).

CONCLUSION

Nutmeg leaf extract (*Myristica fragrans* Houtt) can be formulated into transparent solid soap preparations with various extract concentrations of 0,5%, 1% and 1,5% by getting the best dosage results in Formula 1. an average of 9.6, the highest foam height with an average of 127 mm, the water content corresponding to an average of 12,6%, the lowest free alkali content with an average of 0,08%, and the free fatty acid content that is no longer detected.

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