Clove leaf ethanol extract (*Syzygium aromaticum* L. Merr. and Perr) is formulated as antiseptic liquid soap

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Abstract. Secondary metabolite compound is compound that have medicinal properties, cosmetics, dyes, fragrances. Secondary metabolite compound can be modified into one form of pharmaceutical dosage, namely cosmetic products. One of the cosmetic products is liquid soap preparation. Soap that can kill bacteria is known as antiseptic soap. Clove leaves (*Syzygium aromaticum* L. Merr. And Perr) contain eugenol compounds which have antiseptic properties. This study aims to determine the effectiveness of the ethanol extract of clove leaves as an antiseptic liquid soap preparation. The formulation of clove leaf extract with concentrations of 5%, 10% and 15% with HPMC concentrations of 1 gr, 2 gr and 3 gr. The liquid soap formulation of clove leaf ethanol extract was tested for organoleptic, pH, specific gravity, high foam and viscosity. The results of testing the quality of all liquid soap formulations meet the requirements according to the standards set by SNI. Based on the evaluation of this liquid soap preparation, the ethanol extraction preparation can be formulated into liquid soap preparations. After being tested for its antibacterial activity, Sedian Liquid Soap from Ethanol Extract of Clove Leaves has effectiveness as an antiseptic.

1. Introduction

Secondary metabolite compounds are compounds that have medicinal properties, cosmetics, dyes, fragrances. Secondary metabolite compounds can be modified into a pharmaceutical dosage form that can be used as an antibacterial known as antiseptic soap. One of the natural ingredients that can be used as an antiseptic is the leaves of cloves (*Syzygium aromaticum* L. Merr. And Perr).

Cloves are a traditional medicine that is often used to cure various diseases, as well as a food flavoring. The distinctive aroma of cloves comes from the eugenol compound, where the eugenol compound is the main compound contained in the clove plant (72-90%). Eugenol also has antiseptic and anesthetic properties [1].

Clove leaf extract contains various compounds such as flavonoids, triterpenoids, phenolics, and tannins which are antibacterial compounds that have been shown to have bacterial activity [2]. Soap is a product that results from the reaction between fatty acids and strong bases, which function to wash and clean fat (dirt). Besides being able to clean the skin from dirt, soap can also be used to free the skin from bacteria [3]. Antiseptic soap contains a special composition that acts as an antibacterial.

This material functions to reduce the number of harmful bacteria on the skin. A good antiseptic soap must have a special standard. First, the soap must be able to get rid of dirt and bacteria. Second, soap does not damage skin health, because healthy skin is part of the immune system [4]. The skin covers the surface of the body and has the main function of protecting it from various kinds of disorders and...
external stimuli. The skin is the main defense against bacteria and if the skin is no longer intact, it becomes very susceptible to infection [5].

2. Methods

2.1. Equipments
The tools used in this research are analytical scales, measuring cups, test tubes, erlenmeyer, beaker glass, dropper pipettes, watch glasses, stirring rods, aluminum foil, bunsen, tripod, evaporating cup, pH meter, homogenizer, brookfield viscometer, moisture balance and pycnometer.

2.2. Materials
The simplicia used in this research is clove leaves from Cipancar Village, Serang Panjang District, Subang Regency. The chemicals used are alcohol, olive oil, potassium hydroxide (KOH), Hydroxypropyl Methylcellulose (HPMC), Sodium Lauryl Sulfate (SLS), stearic acid, Butyl Hydroxy Toluene (BHT), distilled water, 0.1 N HCl, dragendorff reagent, mayer reagent, gelatin and iron (III) chloride.

2.3. Plant determination
Plant determination was carried out at Herbarium Bandungense, School of Biological Technology (SITH), Bandung Institute of Technology.

2.4. Water content
The water content in simplicia was determined by using Moisture Balance. A total of 2 g of the sample was put into the Moisture Balance that had been prepared at 100 °C for 10 minutes. The levels listed on the Moisture Balance were then recorded [6].

2.5. Maceration extraction
The powder that has been obtained from the manufacture of simplicia is 500 grams. Then the powder was extracted using the maceration method for 3 days with 96% ethanol solvent. Maceration begins by adding 96% ethanol into the maceration container until all simplicia is immersed. Then soaked for 24 hours previously, stirring. After 24 hours, filter the material with filter paper until there is no liquid dripping from the material, then the dregs are soaked again in the above method for 24 hours, do it for 3 days. The results of maceration in the evaporator use a rotary evaporator to obtain a thick extract [7]. The yield was determined by using equation:

\[
\text{Yield} = \frac{W_{\text{concentrated}}}{W_{\text{simplicia}}} \times 100\% \tag{1}
\]

2.6. Phytochemical screening
Each medicinal plant contains a variety of organic compounds that are formed and contained in these plants. The content of active compounds contained in plants can be determined by separation, purification, and phytochemical screening. Phytochemical screening includes:

2.6.1. Alkaloid test. A total of 500 mg of clove leaf simplicia was alkalized with dilute ammonia, then added 2 ml of chloroform. Then the sample was added with 2 ml of 0.1 N HCl and heated over a water bath, then filtered. The first and second filtrate were dripped with Dragendorff's reagent and mayer solution, while the third filtrate was used as a blank. Positive results are indicated by the formation of red or orange deposits for Dragendorff and white deposits for Mayer's reagent [8].

2.6.2. Flavonoid test. A total of 500 mg of clove leaf simplicia is boiled for 5 minutes, then filtered. After chilling, take 5 ml of the filtrate then add 100 mg of Mg powder and 1 ml of 2N HCl. Then add
amyl alcohol, then shake vigorously. A positive result is indicated by forming a red or yellow or orange color. 1 ml of distilled water was added with clove leaf extract, then treated the same as simplicia [9].  

2.6.3. Tannin test. A total of 500 mg of simplicia was extracted with 10 ml of distilled water, filtered then the filtrate was diluted with distilled water. Take 2 ml of the solution and then add 3 drops of 1% iron (III) chloride. Positive results are indicated by forming a blue or green-black color. The extract sample was added with 1 ml of distilled water, then treated the same.  

2.6.4. Fenol test. A total of 500 mg of simplicia is extracted with 10 ml of distilled water, filtered then the filtrate is taken 2 ml of solution then 1% iron (III) chloride is added, the formation of a greenish brown or blue-black color indicates the presence of tannins, 2 ml of sample is added with gelatin solution and 5 ml of 10% NaCl, the presence of tannins is indicated by the occurrence of yellowish deposits. The extract sample was added with 1 ml of distilled water, then treated the same [9].  

2.6.5. Saponin test. A total of 500 mg of simplicia was extracted with 10 ml of distilled water, filtered. After chilling, 5 ml of filtrate was taken, then shaken vigorously for 10 seconds, a stable foam was formed for 10 minutes, added with 1 ml of 2 N HCl. Positive results indicated that the foam did not disappear. The extract sample was added with 5 ml of distilled water, then treated the same [9].  

2.7. Antiseptic liquid soap formulation of clove leaf ethanol extract  
The formulation of antiseptic soap from clove leaf extract is made in three formulations, with active substances from different formulas. Active substances are distinguished because they are based on antibacterial activity tests, where the extracts that have antibacterial activity start from the extract concentration of 5%, 10% and 15%. Results The formulation of antiseptic soap from ethanol extract of clove leaves can be seen in Table 1.  

| Table 1. Liquid soap dosage formulations made with various concentrations of 5%, 10% and 15%. | Material                  | F 1   | F 2   | F 3   |
|-----------------------------------------------|---------------------------|-------|-------|-------|
| Ekstrak daun cengkeh (g)                      | 5%                        | 10%   | 15%   |
| Minyak zaitun (ml)                            | 10                        | 10    | 10    |
| KOH (ml)                                      | 8                         | 8     | 8     |
| HPMC (g)                                      | 1                         | 2     | 3     |
| SLS (g)                                       | 1                         | 1     | 1     |
| Asam Stearat (g)                              | 1                         | 1     | 1     |
| BHT (g)                                       | 0.5                       | 0.5   | 0.5   |
| Akuades (ml)                                  | Ad 50                     | Ad 50 | Ad 50 |

*Formulation of Antiseptic Liquid Soap with Ethanol Extract of Pacar Water Flower (Impatiens balsamine) and its Effectiveness Test against S.aureus Bacteria in Vitro [10]  

2.8. Preparation of clove leaf ethanol extract liquid soap  
Weigh all ingredients to be used. Put 10 mL of olive oil in a beaker, then add stearic acid. Add 8 mL of 40% potassium hydroxide gradually while continuing to heat at 50 ° C to get a paste soap. Then enter the HPMC that has been developed in hot distilled water, stirring until homogeneous. Then add SLS (Sodium Lauryl Sulfate), stir until homogeneous. Add BHT (Butyl Hydroxy Toluene), then stir until homogeneous. Add clove leaf extract, stir until homogeneous. Add distilled water to a volume of 50 mL, put it in a clean container that has been prepared. The manufacture of liquid soap for clove leaf ethanol extract was adjusted to each concentration. After that, the quality test of clove leaf ethanol extract liquid soap was tested.
2.9. Physical evaluation of organoleptic liquid soap preparations

Organoleptic observation is a method of observation using the human senses as the main tool to assess the quality of the preparation. The nature of subjective observation. The odor, color and texture of the preparations were observed [11].

2.9.1. Determination of specific gravity. The empty pycnometer that has been dried is weighed, then liquid soap and distilled water are put into each pycnometer. Pycnometer is closed, the volume of wasted liquid is cleaned. The pycnometer is then left to stand at 25 ° C for 15 minutes then the weight of the pycnometer containing water and the weight of the pycnometer containing distilled water and liquid soap is weighed. According to SNI, the specific gravity requirement for liquid soap is 1.01-1.1 g / ml.

2.9.2. Viscosity test. Determination of viscosity is carried out using a Brookfield type LV viscometer by observing the numbers on the viscometer scale with a certain speed. The flow properties were determined by determining the viscosity at the shear speed (rpm) of 30 rpm. This examination is carried out at room temperature and a temperature of 40 ° C. The range of viscosity values included in the quality requirements for liquid bath soap is 400-4000 cP.

2.9.3. Determination of pH value. A total of 1 g of soap to be examined is diluted with distilled water to 10 mL. Enter the pH meter into the soap solution that has been made, then wait until the pH meter indicator is stable showing a constant pH value. PH examination was carried out 3 times. The pH value for liquid soap is 8-11.

2.9.4. Foam height measurement. The 2 ml liquid soap sample was put into a test tube then added with distilled water. The tube is shaken for 20 seconds and the high foam is read. The height and stability of the foam were observed after shaking and 5 minutes after shaking.

2.9.5. Cycling test. Liquid soap samples were stored at 4 ° C for 24 hours, then transferred to an oven at 40 ± 2 °C for 24 hours. The test was carried out in 6 cycles and then carried out organoleptic observations (changes in color, smell and synergy) [11].

2.9.6. Hedonic. The hedonic test was carried out by conducting an analysis according to the preference test (parameters of aroma, color, texture and foam preparation) using 20 panelists who were given liquid soap preparations. To see the level of preference of respondents to antiseptic liquid soap based on each parameter

3. Results and discussion

3.1. Determination results
Determinaton is carried out to find out the correct identity of the plants. Based on the letter of determination, the plant used is true Clove Leaf (Syzygium aromaticum L. Merr. And Perr.).

3.2. Moisture content
The water content of the simplicia obtained was 4%. This aims to prevent fungal rot. This value meets the requirements, because the moisture content is not more than 5%

3.3. Results of phytochemical screening
Phytochemical screening was carried out on simplicia and extracts to determine the content of secondary metabolites. There was no difference in yield between simplicia and extract, both were positive for flavonoids, tannins, saponins and phenols. This shows that the secondary metabolites in the simplicia are not damaged during the extraction process.
3.4. Results of making clove leaf extract
The purpose of extraction process is to attract the desired compound by using a suitable solvent, in the present study the extraction is done by maceration method, the advantage of this method is that it can prevent damage to the compound having thermolabile properties. A total of 500 g of simplicia was macerated using 1 L of 96% ethanol for 3 days and the solvent was replaced every 24 hours. Concentration of the liquid extract using a rotary vaporator with a temperature of 40 °C and then stored in a water bath until it becomes a thick extract, the randemen value of clope leaf extract obtained is 22.9%.

3.5. Results of physical evaluation of organoleptic liquid soap preparations
Organoleptic test was carried out by visually observing liquid soap including color, smell and texture for 28 days at 25 °C, 40 °C and 4 °C, with 3 different formulas. The thick liquid dosage form is, the higher the HPMC concentration used, the thicker the dosage is. The extract concentration in the three formulas was also different, the higher the extract concentration, the more intense the color on the preparation and the more pungent the smell was. Formula 1 at each temperature changes color, a rapid color change occurs at a temperature of 40 °C. For formulas 2 and 3, there were also changes, namely broken / separated preparations at week 28.

3.6. Result of determination of specific gravity
Density observations were carried out on day 1 to day 28, the results obtained were 1.016 - 1.035 g / mL. This test is conducted to determine the effect of the ingredients used in the liquid soap formulation on the density of the soap produced. Specific gravity test aims to determine the viscosity of liquid soap. Based on SNI, the standard density for liquid soap is 1.01-1.1 g / mL. Based on the results obtained, all formulas are in accordance with SNI.

3.7. Viscosity test results
The extract concentration and HPMC affect the viscosity of liquid soap. This is due to the fact that the extract can provide thickness to the preparation. Meanwhile, HPMC can affect the physical properties of liquid soap preparations, especially on the viscosity which causes a thick texture in high concentrations. The amount of viscosity is 800-2400 cP, during storage there is a decrease in viscosity, this is due to storage of the preparation at a temperature of 40 °C, heating causes the liquid molecules to gain energy and move so that the interaction force between molecules weakens. Thus the viscosity will decrease. The formulation that has a viscosity value that is close to the criteria is the third formulation because the HPMC used is also higher. But the three formulations still meet the criteria for a good liquid bath soap, namely 400-4000 cP.

3.8. Results of determination of pH value
pH test is one of the requirements for the quality of liquid soap. This is because liquid soap is in direct contact with the skin and can cause problems if the pH of the liquid soap is not suitable. In general, liquid soap products have a pH that tends to be alkaline. This is because the basic ingredient of liquid soap is KOH which is used to produce saponification reactions with fats or oils. The pH requirement for liquid soap according to the Indonesian National Standard [6] is around 8-11. From the graph, it can be seen that during the storage process there was no change in pH, this shows that during storage the preparation was stable. The results showed that all liquid soap formulas met the criteria for good liquid soap.

3.9. Foam height measurement results
Foam is one of the most important parameters in determining the quality of cosmetic products, especially soap. The purpose of foam testing is to see the foam power of liquid soap. Foam that is stable for a long time is desirable because it can help cleanse the body [12]. The foam measurement results indicate the surfactant's ability to form foam. The foam height produced from the three liquid soap formulations
ranges from 3-5.5 cm and meets the requirements, where the foam height requirement for liquid soap is 1.3-22 cm [5]. In addition, the stability of the foam after leaving for 5 minutes, when compared to the three preparations, then formulation 3 has a relatively higher number among other formulations. This means that in formulation 3 the foaming agent (Natrium Lauryl Sulfate) has the most optimum effectiveness.

3.10. Cycling test results
The cycling test was carried out for 6 cycles where 1 cycle 24 at 4 °C ± 2 °C and 24 hours at 40 ± 2 °C. This test is carried out to determine the stability of the preparation. All preparations are stable.

3.11. Hedonic results
The favorite test of antiseptic liquid soap for ethanol extract of clove leaves was carried out by 20 volunteers with an age range of 21-32 years. The most preferred formulas are F3 in terms of color, odor, texture and foam.

4. Conclusions
Based on the results of the research and discussion that has been done, it can be concluded that the ethanol extract of clove leaves can be formulated into an antiseptic liquid soap. The antiseptic liquid soap formulation of ethanol extract at a concentration of 5%, 10% and 15% met the requirements of the physical quality test. The most preferred formulation of odor, color, texture and foam is the third formulation.

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