Preparation and Characterization of Coconut Oil Based Soap with Kaolin as Filler

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Abstract Soap is the product of saponification of fats with a base, which is used as a body cleanser, in the form of solid, foamy, with or addition of other ingredients and do not cause skin irritation. Clay (kaolin) is used as an active ingredient in cosmetics because of the high level of adsorption of substances such as oil, toxins, and others. The use of clay also been considered as a potential tanning agent in halal industrial applications. This study aimed to search for the effect of temperature reaction and addition of kaolin to the solid soap produced. The saponification reactions was carried out for 10 minutes in a reaction flask at a temperature that varied from 50ºC - 80ºC. The beginning stage was to heat the coconut oil to the reaction temperature and then mixed it with kaolin (10%, 12.5%, 15%, 17.5%, 20% wt.), followed by adding 35% NaOH. The reaction mixture was stirred at 250 rpm. The results of this study showed that all soap products met the Indonesia standard SNI.

1. Introduction
Bar soap is a kind of regular soap which is solid and not transparent, while liquid soap is soap formed in liquid. Soaps which have good quality, are affected by the raw material used. The main raw material for making soap is fat or oils obtained from plant and animal. Oil used in this research was coconut oil. Coconut oil is easily saponified. The most predominant fatty acid in coconut oil is lauric acid. Lauric acid is indispensable for making soap. This is caused by saturated fatty acids contained in lauric acid which is able to provide high solubility and excellent foam properties for soap products [1]. Soap is the result of saponification. Saponification is a process that reacts fats or glycerides with bases. According to the Indonesian National Standard (SNI) 1994, soap is defined as a sodium compound with fatty acids which are used as a body cleanser, in a solid, foamy form, with or without the addition of other fillers which do not cause irritation to the skin. [2] The saponification reaction is regarded as auto-catalytic because the resulting soap is able to dissolve alkali and also able to disperse neutral fat oil into colloidal suspension [3].
Coconut oil
The components of coconut oil consist of 50% lauric acid, followed by other acids such as myristic acid, caprylic acid, palmitic acid, capric acid, oleic acid, stearic acid, and linoleic acid. There are many benefits of coconut oil including antiviral, antibacterial, antifungal, antiparasitic, insect repellent, antioxidant and anticancer [5].

Kaolin Clay
Clay minerals are used as active ingredients in cosmetics because of the high adsorption rate on substances such as oils, toxins, and others. Clay minerals that are often used include kaolin, smectite, talc, and palygorskite [6]. Kaolin is a rock mass consisting of white or slightly whitish clay material as shown in Figure 2. Kaolin does not absorb water so it cannot expand when mixed with water [7]. The use of clay has also been considered as a potential tanning agent in halal industrial applications [8]. The composition of kaolin consists of 46.54% SiO₂, Al₂O₃ 39.50%, and 13.96% H₂O. Kaolin particles are usually hexagonal sheets with a diameter of about 0.05-10 μm (0.5μm average). This study aimed to examine the effect of adding kaolin to the properties of the resulting solid soap.

\[ \text{Figure 1. Saponification reaction [4]} \]

\begin{align*}
\text{R} & \quad \text{R'} \\
\text{O} & \quad \text{O} \\
\text{O} & \quad \text{MOH} \\
\text{M} = \text{Na, K} \\
\text{O} & \quad \text{O} \\
\text{R'} & \quad \text{R'} \\
\text{O} & \quad \text{HO} \\
\text{H} & \quad \text{HO} \\
\end{align*}

\(\text{Figure 2. Kaolin Clay}\)

2. Method
Coconut oil, sodium hydroxide, distilled water, and kaolin were used as the main ingredients for making soap. Other materials such as ethanol, phenolphthalein and hydrochloric acid as analyzers for the soap samples produced.

The saponification reaction were carried out in a beaker glass at varying temperatures of 50°C, 60°C, 70°C and 80°C and the kaolin consistency of 10%, 12.5%, 15%, 17.5%, 20%. The reaction
lasted for 10 minutes with a ratio of the amount of coconut oil and 35% NaOH was 7: 3 (w/v), 100 mesh kaolin particle size and a constant stirring speed of 250 rpm.

The analysis performed on each sample in this study is based on the Indonesian National Standard (SNI) in the form of:
1. Analysis of saponification
2. Analysis of water content
3. Analysis of free alkali content
4. Analysis of foaming stability
5. Analysis of degree of acidity (pH)
6. Analysis of hardness.

| No. | Commentary                          | Type I (Soap Powder)                  |
|-----|-------------------------------------|---------------------------------------|
| 1   | Water content (%)                   | Max 15                                |
| 2   | Fatty acid content (%)              | > 70                                  |
| 3   | Free alkali                         |                                       |
|     | - calculated as % NaOH              | Max 0.1                               |
|     | - calculated as % KOH               | Max 0.14                              |
| 4   | Free fatty acids or neutral fats (%)| <2.5                                  |
| 5   | Mineral oil                         | Negative                              |

2.1. Experimental apparatus
In order to perform the study, experimental apparatus has been arranged as shown in Figure 3. It consists of beaker glass, thermometer, hotplate and magnetic stirrer.

![Experimental apparatus](image)

Figure 3. Experimental apparatus

3. Results and Discussions

3.1 Analysis of free alkali content
From this research, it was found that in each soap product obtained with variations in the consistency of kaolin, no free alkali was found after 2 weeks of storage, i.e. 0% free alkaline content where there
was no change in the color of the soap solution after the addition of phenolphthalein indicator. This was due to the added NaOH solution had reacted completely at the end of the reaction. The amount of free alkali in soaps that meets SNI 1994 standards must be < 0.1%. Excess free alkali can cause skin irritation. According to the results of research conducted by Widyasanti, et al., [8], the addition of substances containing alkali will increase the levels of free alkali in soap.

### 3.2 Effect of kaolin addition and increase in reaction temperature on moisture content

According to the 1994 SNI for soap, the maximum amount of water content in soap is 15%. Moisture content is a parameter used in assessing the shelf life of a product. High water content in soap will cause excess water reaction with unsaponified fat to give free fatty acids and glycerol in a process called hydrolysis during storage [9]. The more water contained in soap, the easier it will shrink when used [10].

Figure 4 shows the increased consistency of kaolin causing an increase in water content in soap preparations. The lowest water content obtained from this study was 1.75% and the highest water content of 6.12% was found in solid soap products with a kaolin consistency of 20% and a reaction temperature of 80ºC.

![Figure 4. Effect of reaction temperature on moisture content at various kaolin consistency](image)

Kaolin is a type of clay. Increased water level is proportional to increased consistency because kaolin contains about 50% SiO₂ [11].

Based on the analysis, the water content increases with increasing kaolin consistency and the reaction temperature applied to soap making. In the constant consistency of kaolin the rising temperature will accelerate the saponification reaction. The faster the reaction occurs, the more water is trapped in the soap. However, at the highest reaction temperature and consistency of 20% kaolin, the moisture content of the solid soap obtained from this experiment still meets SNI standards (<15%).

### 3.3. Effect of kaolin addition and increase in reaction temperature on free fatty acid content

Free fatty acids are derived from fatty acids that are not bound to the sodium or triglycerides. The fatty acid has limited ability to dissolve in water [10].

The results of the analysis of free fatty acids in Figure 5 show that the levels of free fatty acids in soap are at 0.15 to 0.6%. Free fatty acid levels decrease with the addition of kaolin and an increase in reaction temperature. The highest free fatty acid levels were found at 10% kaolin consistency at 50ºC reaction temperature and the lowest free fatty acid content for soap at 20% kaolin consistency and 80ºC reaction temperature. A decrease in soap free fatty acid levels occurs stably at 15% kaolin consistency at each reaction temperature.
Increased reaction temperatures cause molecules to become more active in motion and can increase the frequency of collisions between molecules so that free fatty acid levels can be minimized [12]. According to SNI (2016), the maximum free fatty acid requirements are 2.5% so that the soap with the addition of kaolin 10%, 12.5%, 15%, 17.5% and 20% meets the requirements.

3.4 Effect of kaolin addition and increase in reaction temperature on degree of acidity (pH)

Based on the analysis, the pH value of soap tends to increase with increasing kaolin consistency and reaction temperature. According to the SNI standard solid soap pH between 9 - 11, pH range between 4 to 10.5 will not cause skin irritation [13]. The pH value of the soap obtained in this study ranged from 8.7 to 9.2 as shown in Figure 6. Thus, the pH value of all the soaps produced was in accordance with SNI standards.

The increase in soap pH is due to an increase in kaolin concentration because the pH of kaolin ranges from 4 to 7.5, so the presence of kaolin influences the increase in pH. Soaps with alkaline pH tend to open up the pores of the skin so as to maximize the removal of impurities from the skin [13].

3.5 Effect of kaolin addition and increase in reaction temperature on foam stability

Foams is a gas trapped by a thin layer of liquid containing a number of soap molecules adsorbed on the thin layer. In a bubble, the surfactant hydrophobic group will point to the gas, while the hydrophilic portion will lead to the solution and then the bubble will come out of the liquid body.
Figure 7. Effect of reaction temperature on foam stability at various kaolin consistency

Figure 7 shows that the amount of kaolin added to soap does not show a significant difference in the stability value of the foam. Soap foam stability values have been above 70% in each sample variation.

The quality of foam for soap products is not determined in SNI, because the height of foam is not related to the ability of a soap product in the cleaning process, but it is related to consumer perception and aesthetics. Foam height of a soap product can be influenced by unsaturated compounds such as oil mixtures and types of additives used in water [13]. Soap made from palm oil can produce foam very well in water containing high salt or alkali. Because Iodine numbers (8-10) are very low and saponification number is high (250-260). Lauric acid and myristic acid can produce soft foam, whereas palmitic and stearic must stabilize foam. Oleic acid and linoleic acid can produce stable and soft foams [14].

3.6 Effect of kaolin addition and increase in reaction temperature on soap hardness

Hardness test is carried out after the soap has been stored for 2 weeks. Test results show an increase in the value of soap penetration in Figure 8. The value of penetration is inversely proportional to hardness. The greater the penetration value, the softer the soap.

Figure 8. Effect of reaction temperature on soap hardness at various kaolin consistency
Figure 8 shows the increase in penetration value which is proportional to the increase in the amount of kaolin and the increase in reaction temperature. An increase in reaction temperature at a certain kaolin consistency the value of penetration increases, and the higher the kaolin consistency at a certain reaction temperature the penetration value also increases. The highest penetration value was 1.8 N/cm² at 20% kaolin consistency and reaction temperature was 80ºC. This increase in penetration indicates that the soap is softer on adding kaolin consistency and increasing the reaction temperature. This happens because the more kaolin is added the more soap water content. The water contained in the soap has been trapped and bound by kaolin so that the water does not evaporate and the resulting soap becomes softer.

Medium range of penetration at 15% kaolin consistency gives good results in visual analysis, not too soft or hard to use.

To help the soap last longer in the bathroom, bar soap is made to be hard. Saturated fat that is commonly used to harden soap is coconut oil. Improved water solubility helps create more foam and improves cleaning ability [15].

4. Conclusions
The addition of kaolin affects the properties of the resulting solid soap (moisture content, free fatty acid, acidity, foam stability and hardness). However, the results of the study showed that all solid soaps produced in the range of study met SNI requirements. The best hardness soap was obtained at 15% kaolin consistency in all temperature ranges tested.

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