International Journal of Technology 12(2) 263-274 (2021)
Received June 2020 / Revised June 2020 / Accepted January 2021

International Journal of Technology

http://ijtech.eng.ui.ac.id

Characterization of the Chemical and Physical Properties of Bar Soap Made with Different Concentrations of Bentonite as a Filler

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Abstract. The use of bentonite in cosmetics is considered beneficial for absorbing dirt and moisturizing and detoxifying the skin. In this study, different amounts of bentonite (10, 12.5, 15, 17.5, and 20 g) were used as a filler in 5 separate formulations of natural bar soap having coconut oil as the primary ingredient. Each type of bentonite soap was analyzed in accordance with the Indonesian National Standards (SNI). The free alkali content for each of the five preparations of bentonite soap was zero, the moisture content ranged from 0.88 to 1.15 %, the pH ranged from 8.7 to 8.8, the total free fatty acid content ranged from 0.28 to 0.72, and the foam or bubble stability was above 70%, while the soap's hardness ranged from 1.49 N/m^2 to 1.54 N/m^2. All five soap types fulfilled SNI standards, but the soaps containing 15 g and 17.5 g of bentonite had a far better appearance. The chemical compositions of the bar soap composed of 15 g bentonite (BS 15) and the bar soap composed of 17.5 g bentonite (BS 17.5) were calculated using material balance and compared by Energy Dispersive X-ray (EDX) analysis. The EDX analysis revealed that both BS 15 and BS 17.5 contained the Si and Al metals originating from the bentonite. No heavy metals considered harmful to skin health (e.g., Hg, Pb, As, or Cd) were detected. Scanning electron microscopy (SEM) of the topography and texture of BS 15 and BS 17.5 revealed a more even distribution of Si and Al throughout BS 17.5 than in BS 15. BS 17.5 was therefore deemed the best formula as it meets SNI standards and has a better appearance and a more even mineral distribution.

Keywords: Bar soap; Bentonite; Characterization; SEM-EDX; Stoichiometry

1. Introduction

Current trends in consumer preference indicate an increased demand for natural ingredients in skin care and cosmetic products. This has led to an increase in the economy of small-scale homemade (artisanal) industries that offer a wide range of products with a variety of natural ingredients. One skincare product with natural ingredients that is now in vogue is natural bar soap. The global market for bar soap has been studied in key regions, including North and South America, Europe, China, Japan, India, the Middle East, and Africa. The market for bar soap in the Asia-Pacific Region is likely to show remarkable growth.

Bar soap made from natural ingredients, such as vegetable oil, does not dry out the skin because this soap type contains glycerin. Natural herbal soap ingredients are generally vegetable or saponification oil (even animal fat), glycerin, natural fragrance, various organic additives, and sodium hydroxide. One soap-making process involves oil saponification, a

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doi: 10.14716/ijtech.v12i2.4130

chemical reaction that occurs between fats like coconut oil ($C_{33}H_{62}O_6$) and alkali or sodium hydroxide (NaOH) to producing glycerol ($C_3H_8O_3$) and soap (Vidal et al., 2018). Chemically, soap is symbolized by the general formula RCOONa, in which R is a long-chain alkyl group consisting of 12 to 18 carbon atoms.

Natural herbal soaps usually contain a mixture of coconut oil, palm oil, olive oil, rice bran oil, and sunflower seed oil (Araseretnam and Venujah, 2019). The distribution of saturated and unsaturated fatty acids determines the hardness, aroma, cleansing, power, foaming, and moisturizing ability of the soap. The properties of the resulting soap are also further influenced by the additives or fillers that are used. One common filler used in soap-making is bentonite clay, a natural clay that is used as a traditional skin regimen and medicine in many cultures (Moosavi, 2017).

Bentonite clay acts as a detoxifying agent due to its polycationic property, which promotes the absorption of negatively charged toxins (Williams et al., 2009). Bentonite, better known as grey clay powder, has been used for centuries for medicinal purposes. Because of its active properties, this mineral is able to fight lipodystrophy, a disease that causes humans to lose fat tissue in body parts, giving the appearance of aging, acne, and cellulite (Carretero and Isabel, 2002). Clay powder can also be formulated into topical applications to serve as a dermatological protector. The active properties of clay powder are what give it the ability to provide opacity, cover up blemishes, and remove shine from the skin. Its activity comes from the two prevalent metal oxides, SiO_2 (silica), which ranges in content from 48% to 50%, and Al_2O_3 (alumina), which ranges in content from 14.5% to 15%. Other less prevalent metal oxides include Fe_2O_3 (iron), CaO (calcium), K_2O (potassium), Na_2O (sodium), Ti_2O (titanium), and P_2O_5 (phosphorous); these are all found in relatively small amounts (Shah et al., 2013; Nweke et al., 2015).

The density of bentonite is higher than that of water, so it is more stable and can maintain its form for a longer time than it can in the bentonite soap that is produced (Asad et al., 2013). Much research has already been done on the production of detergent formulas that contain soil additives such as clay, bentonite, or even kaolin. Patents (Dahlan, 2010) have been granted for clay-containing detergents for external use and for the cleaning of equipment contaminated with impurities; these detergents range in pH from 5.5 to 6.5. Research has also been conducted aimed at the optimization of bentonite use as a filler for liquid detergents used as agents to clean extreme "*najis mughalladhah*" (Wati et al., 2019). *Najis Mughalladhah* in the Islamic religion is a form of ritual uncleanliness originating from dogs and pigs, and this uncleanliness requires special treatment for purification or cleansing.

Generally speaking, the main raw material for solid bath soap is currently sodium lauryl sulfate (SLS), which acts as a detergent. However, SLS is produced from petroleum, so this is not a sustainable industrial process. Therefore, industry must develop an adequate and appropriate strategy to maintain long-term competitiveness for soap making. Tatiana and Mikhail (2020) discuss the relationship between the innovative strategy recommended for industry life cycle stages and the specification of regional and territorial factors. The industry for skincare products with natural ingredients can be developed in Indonesia because the industry lifecycle sustainability can be maintained, especially when considering that Indonesian territories are the largest global producers of vegetable oils, including coconut and palm oils.

Studies of important implications of the development and success of products have identified six standard factors that influence the success of a variety of products; these are price, product performance, brand, esthetic design, service, and marketing (Setyaningrum et al., 2020). A viable esthetic design for bath soap is influenced by the soap's physical properties, such as its hardness, and the compatibility of the bentonite filler with the solid soap.

The production of solid bath soap using coconut oil and bentonite filler as the only main raw materials has not yet been reported. Therefore, the objectives of the present research were to develop a soap formula using coconut oil and sodium hydroxide as the main raw materials and bentonite as a filler, resulting in a solid soap that contains glycerin and bentonite. The characteristics of the chemical and physical properties of the resulting solid bath soap, including the metal content, were analyzed by EDX. The compatibility of bentonite in solid soap production was examined by SEM.

2. Materials and Methods

2.1. Materials

The main ingredients used to make soap in this study are coconut oil, sodium hydroxide (NaOH), and bentonite. The coconut oil, purchased from Simeuleu, Aceh, Indonesia, consisted of 48% lauric acid ($C_{12}H_{24}O_2$), 21% myristic acid ($C_{14}H_{28}O_2$), and 9.5% palmitic acid ($C_{16}H_{32}O_2$). Solid form 98% sodium hydroxide (NaOH) was obtained from Asahi Mas, Banten, Indonesia. Solid-state bentonite, in the form of fine brown particles that were slippery on contact and contained in excess of 85% montmorillonite mineral content, had a chemical formula of $Al_2O_3.4SiO_2$ xH_2O. Bentonite used in pharmaceutical and cosmetic products must meet a number of requirements related to its chemical, physical, toxicological, and organoleptic properties. Consequently, this research used cosmetic grade bentonite from T & T Chemicals, Bandung, West Java, Indonesia, which has 61–68% SiO_2 and 21–24% Al_2O_3 as the primary mineral content and 10–11% H_2O content. Previous research found that bentonite from Sarimanggu, West Java, Indonesia, had 66.9% silica SiO_2 and 16.07% Al_2O_3, as well as other ingredients (Wulandari et al., 2016). Bentonite requires no special treatment for use as filler prior to mixing into soap solutions.

2.2. Methods

The soap-making method was the semi-cold saponification process that uses alkali (sodium hydroxide) as the basic material in the saponification process conducted in a cold state.

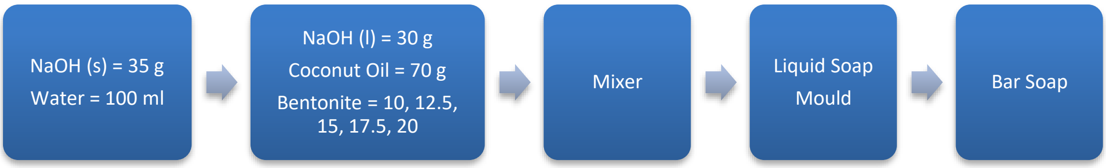

Figure 1 Flow diagram of the process of making bentonite bar soap

The flow diagram in Figure 1 shows that, for each preparation, 35 g NaOH was mixed with 100 g of water and left to cool for 60 minutes (Moulay et al., 2011). A 30 g aliquot of the sodium hydroxide alkaline solution and 70 g of coconut oil were combined and stirred, and then bentonite was added at either 10, 12.5, 15, 17.5, or 20 g. The stirring was continued until the soap mixture appeared trace. The mixture was then put into the mold and the soap was left for 24 h before the next analytic process.

2.2.1. Analysis of soap

Moisture content was analyzed using ISO 672, free alkali content was analyzed by the IUPAC titration method, free fatty acid content was analyzed by ASTM D5555-95, and pH was measured with an ATC pH meter. The foam stability of the soap was determined by calculating the percent decrease in the height of the soap foam from its initial height, and soap hardness was measured using a hardness testing meter (needle penetrometer).

2.2.2. SEM-EDX analysis

The chemical compositions of the soaps formed by the addition of 15 g of bentonite (BS 15) and 17.5 g of bentonite (BS 17.5) were calculated by the stoichiometry method and compared with Energy Dispersive X-ray (EDX) analysis results. The topography and surface characteristics and the texture of the soap were examined by scanning electron microscopy (SEM).

3. Results and Discussion

3.1. Effect of Adding Bentonite on the Characteristics of the Soap Produced

Bentonite soap destined for marketing in Indonesia must meet several quality benchmarks from the Indonesian National Standard (SNI). Table 1 shows several characteristics of the soaps produced, namely their free alkali content, moisture content, free fatty acid content, pH, foam stability, and hardness.

Table 1 Some characteristics of bentonite soaps

No	Characteristics of Soap	Content of Bentonite (%)					SNI Standard	Unit
		10	12.5	15	17.5	20		
1	Free alkali Content	0.00	0.00	0.00	0.00	0.00	<0.10	%
2	Moisture Content	1.15	1.07	1.00	0.91	0.88	<15.00	%
3	Free Fatty Acid Content	0.28	0.44	0.68	0.70	0.72	<2.50	%
4	pH	8.70	8.70	8.80	8.80	8.80	9–11	-
5	Foam Stability	71.00	72.00	77.00	81.00	80.00	-	%
6	Hardness	1.49	1.50	1.51	1.52	1.54	-	N/m^2

SNI: Indonesian National Standard

Table 1 shows that the free alkali content, moisture content, and free fatty acid content of the five types of soap clearly meet the SNI standards. The pH of the soap ranges from 8.7 to 8.8, in accordance with SNI standards, showing that the soap is more neutral. The highest foam stability was 81%, produced by the soap with 17.5 g bentonite. The soap with 10 g bentonite content had the lowest hardness, at 1.49 N/m^2 (softer), while the soap with 20 g bentonite filler had the highest hardness, at 1.54 N/m^2.

3.1.1. Effect of bentonite addition on the free alkali content

Free alkali content refers to the amount of basic substances that are not bound by fatty acids or bound in the form of salts and is calculated as the percentage of Na_2O.

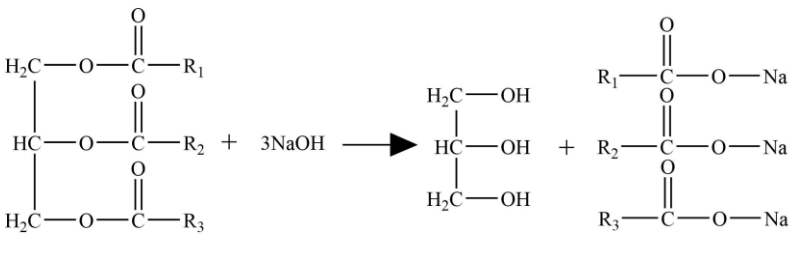

Figure 2 Triglyceride and sodium hydroxide saponification process

According to the SNI standards (Table 1), excess free alkali cannot exceed 0.10% for NaOH soap and 0.14% for KOH soap, because alkali are strongly basic and can cause skin irritation. An excess of free alkali in soap can arise due to the use of high concentrations of alkali or the use of too much alkali during the saponification process. Stoichiometry calculations are carried out and extra oil is applied during the saponification process to ensure the minimum standard for the amount of free alkali. Figure 2 shows the stoichiometric reaction of a triglyceride and sodium hydroxide in the saponification process to produce a bar of soap and glycerin. One molecule of triglyceride reacting with three molecules of sodium hydroxide will form one molecule of glycerin and three molecules of soap.

This reaction shows that the solid soap produced contains a byproduct of glycerin. Glycerin naturally has a strong influence on the rheological performance in determining the interaction of bentonite and organic tissue. According to Pusch (2015), glycerin is usually used to prepare mixtures that use bentonite. In this case, the glycerin byproduct is also beneficial in the soap-making process.

Table 2 shows that, based on calculations, the free alkali content of the entire sample was 0, which clearly meets the SNI standard that the free alkali content should not exceed 0.10%. The reaction was carried out stoichiometrically with the aim of producing a free alkali content of less than 0.10%.

Table 2 Material balances of the saponification reaction of sodium hydroxide with an excess of coconut oil

No		Coconut Oil	NaOH	Excess Coconut Oil	Glycerin	Soap
1	Mol	0.11	0.26	0.02	0.09	0.09
2	MW	658.00	40.00	658.00	92.00	684.00
3	Weight (g)	69.86	10.50	12.06	8.10	60.20
Total Weight (g)		80.36		80.36		

Table 2 shows that the sodium hydroxide reacted completely with the coconut oil, because a 20% excess of coconut oil was supplied. In addition to reducing the free alkali content, another reason for using extra coconut oil is to ensure that the formed soap still contains coconut oil, which is beneficial to skin health. The bentonite added to the soap contains various minerals that are also expected to react with coconut oil.

Several researchers, such as Betsy et al. (2013), have carried out studies on free alkali content in several commercial soaps available on the market and have found that the free alkali content of the soaps analyzed were in accordance with their countries' standards. Widyasanti et al. (2018) carried out research on the addition of glycerin to solid soap formulas using coconut oil as a raw material and reported that the free alkali content of the soap decreased as the amount of added glycerin was increased. They considered that the addition of glycerin to the soap formulation allowed it to bind to hydroxide ions, thereby reducing the free alkali content of the resulting soap. As shown in Table 2, the produced soap produced had a glycerin content as a result of the saponification reaction. This might be what led to the zero free alkali found in the produced soap. Similarly, the effect of adding excess oil or unsaponified oil on the cold saponification process has been studied by Vidal et al. (2018), who showed that excess fatty acids function as important contributors to the total sensory perception and quality of natural herbal soaps.

3.1.2. Effect of adding bentonite on the moisture content

Table 1 shows the moisture content of the solid soap made with 10, 12.5, 15, 17.5, and 20 g of bentonite. The moisture content of the soap preparations with bentonite filler varying from 10 to 20 g ranged from 0.88%, as the lowest value, to 1.15 %, as the highest. The moisture content of bentonite soap decreases with increasing bentonite content. The quality requirements for bath soap set by the SNI are that solid soap must have a maximum moisture content of 15%. All the soaps in this study made with all concentrations of bentonite had moisture contents meeting the SNI requirements.

Widyasanti et al. (2018) reported that addition of a high concentration of glycerin to the soap formula reduced the water content of the resulting soap. This is because glycerin has the ability to bind to water. The results of this research supports the idea that bentonite soap has a glycerin content that can bind water.

3.1.3. Effect of adding bentonite on the free fatty acid content

Free fatty acids are another important additive used in the formulation and production of natural solid soaps (Bernecke and Maruska, 2013). Free fatty acids are derived from fatty acids (triglycerides) that are not saponified in the final product after the saponification and curing processes. Although free fatty acid additives can reduce the odor and color stability of the final product, they are also key determinants of the quality of the soap (Burke, 2005; Bernecke and Maruska 2013). The majority (86–99%) of polyunsaturated fatty acids (5.0–7.0 μg/mg) remain unsaponified in the natural soaps, regardless of the raw materials used. These previous studies show that unsaponified free fatty are an important contributor to the quality of soaps produced using the cold saponification process. Table 1 shows that the maximum free fatty acid content of soaps with different bentonite contents was 0.72%, while the SNI stipulates a maximum free fatty acid content of 2.5%. The low free fatty acid content in all the soaps with bentonite content is due to the use of coconut oil, which does not generally contain polyunsaturated fatty acids.

3.1.4. Effect of adding bentonite on the pH

The surface of human skin is generally acidic, whereas soap is generally basic, as the majority of soaps circulating in the market having a pH that ranges from 9–10. Table 1 shows that the soap pH increases slightly with an increase in the amount of bentonite filler. For example, a soap with 10% bentonite filler had a pH of 8.7, while a soap with 20% bentonite filler had a pH of 8.8. The addition of bentonite filler did not significantly increase the pH of the soap, and these soaps with bentonite fillers in fact have a pH below the average pH of soaps circulating in the market (Parker, 2019). Tarun et al. (2014) studied the pH of 64 types of bar soap and found that 53 samples had a pH in the range of 9.01 to 10.

The ratio of sodium hydroxide to triglyceride also affects the pH of soaps, so that an increase in this ratio increases the pH (Awang et al., 2001). Naswir et al. (2013) found that the addition of bentonite to peat water does not significantly affect the pH, but it does reduce the peat water color imparted by Fe and organic matter. The addition of bentonite does not affect the pH at all, but the other minerals found in the bentonite can react with excess coconut oil to form organic salts.

3.1.5. Effect of the addition of bentonite on foam stability

Foam is defined as a dispersion system consisting of gas bubbles covered with a layer of liquid. Foam is one of the parameters that determines the quality of bath soaps. One of the ingredients that functions to produce foam in the production of bath soap is the selected triglyceride. Triglycerides with a saponification value require a greater amount of base to complete the saponification process. Coconut oil has a higher saponification value than palm oil and other fats (Susanti et al., 2018).

The very different densities found in bubbles and the liquid medium make the system quickly separate into two layers, with the bubbles rising to the top. Table 1 shows the foam stability of the soap with varying concentrations of bentonite. A greater amount of bentonite filler resulted in a greater foam stability, with the bubble stability above 70% in all preparations. Ossai (2014) concluded that a greater foam stability would increase the detergency capacity of the soap. The highest foam stability in this research was 81%, obtained for the soap produced using 17.5 g of bentonite filler.

3.1.6. Effect of adding bentonite on hardness

For a solid soap to maintain its form, the soap must be hard. Coconut oil is a fat commonly used to produce hard soaps. Table 1 shows that the hardness of the soaps increased with increasing bentonite content. Abd Gani et al. (2018) previously used a D-optimal mixture experimental design to study the effect of fatty acids and different oils (cocoa butter, pure coconut oil, olive oil, palm oil) used in the primary composition of soap formulations on the hardness of the final soap. Their research results indicated that soap hardness is greatly influenced by differences in the level of fatty acids and oils in the formulation. They also found an effect of ingredients on the physical properties of cosmetic products such as solid soaps and this greatly affected the aesthetic design and physical appearance of the soap. In the present research, the best soap hardness and performance, from a visual standpoint, were obtained for soaps containing 15 and 17.5 g bentonite.

3.2. Comparison of the Bentonite Soap Composition Calculation Method and EDX Analysis Results

Only the BS15 and BS17.5 soaps were used in the analysis of the atomic content and other physical properties. Both soaps showed good performance and met the SNI standards. EDX was used to analyze the atomic content and structure of BS 15 and BS 17.5. EDX is now increasingly used as a powerful technique for the analysis of elements based on surface analysis. This method has been used to study the microtopographical characteristics and to map elements calcified in the human heart (Chang et al., 2014), as areas in the heart with calcium deposits show higher amounts of C, O, P, and Ca and traces of N, Na, Mg, and Al.

Table 3 shows the results of the stoichiometry calculations used to compare the atomic content of BS 15 and BS 17.5, based on the results of the EDX analysis. The bar soap was made using coconut oil and sodium hydroxide, so the main elements in the bar soap are C, H, O, and Na. The main elements in bentonite are Si and Al, with other minerals present in very small and typically undetectable amounts. The saponification reaction shown in Figure 1 allows the calculation of the number of C, H, O, and Na atoms in BS 15 and BS 17.5 by adding the number of C, H, O, Si, and Al atoms contained in the 15 g and 17.5 g of added bentonite. The predominant minerals contained in bentonite can be assumed to be the metals Si (48–50%) and Al (14–15%), while other metals such as Ca, Na, Mg, K, Ti, and P (0–2%) are negligible because their ratios are very small.

Table 3 Differences in the percentage weight compositions of BS 15 and BS 17.5, based on calculation methods and EDX analysis

No	Atomic Name	% Atomic Weight of BS 15 Based on Calculations	% Atomic Weight of BS 15 Based on EDX	% Atomic Weight of BS 17.5 Based on Calculations	% Atomic Weight of BS 17.5 Based on EDX
1	Carbon (C)	43.92	63.69	42.19	42.25
2	Oxygen (O)	39.38	19.93	39.78	28.59
3	Sodium (Na)	2.99	12.84	2.84	16.85
4	Nitrogen (N)	-	2.41	-	-
5	Silica (Si)	7.28	0.80	8.07	8.58
6	Aluminum (Al)	6.42	0.31	7.12	3.72

The EDX analysis revealed that the main contents of BS 15 and BS 17.5, such as C, O, Na, Si, and Al atoms, arise from the raw material of the soap and the bentonite. The H atoms, while present in large amounts, were not visible on EDX because EDX is unable to detect elements with very small atomic masses, such as hydrogen, helium, and lithium. The EDX data in Table 3 indicate that Si and Al levels also increase with an increase in the bentonite content in the soap. This is because bentonite contains large amounts of Si and Al minerals; therefore, adding more bentonite will of course increase the amounts of Si and Al minerals in the final soap. The observed divergence in the amounts of C, O, Na, N, Si, and Al atoms in the BS 15 and BS 17.5 soaps, based on calculations and the EDX analysis, can be accounted for by the fact that EDX analysis uses a very small sample size; consequently, heterogeneity and uneven particle distribution can cause the analysis to disagree with the calculation results.

The EDX results clearly show that neither type of bentonite soap contained heavy metal elements, such as Hg, Pb, As, and Cd, which are dangerous to health. These types of metals are banned by the European community and are strictly limited in terms of maximum concentration by the US FDA and in Canada. The metals found in the BS 15 and BS 17.5 formulas were Si and Al, which come from bentonite, and their use has been approved by European Parliamentary Regulation 1223/2009 in dyes for cosmetics, such as lipsticks and lip glosses (Borowska and Brzóska, 2015). No regulations have been set for minimum acceptable amounts of Al metal in cosmetic products (Faveroa et al., 2019). Some concerns related to the use of products containing Al, or other heavy metals from the same category as aluminum, because of concerns that this component can have a cumulative effect on blood and tissue.

Bentonite clay has been proven to act as a detoxification agent, due to its polycationic properties that promote the absorption of negatively charged toxins (Williams et al., 2009), Bentonite is abundant, inexpensive, and natural, and a review of scientific papers reporting the effects of this clay on the human body has been conducted by Moosavi (2017). The review included more than 2500 articles in PubMed about bentonite clay, and Moosavi determined that about 100 articles were related to the effects of bentonite on the body. The results indicated that bentonite has characteristics that merit further study. Natural ingredients such as bentonite should be considered in modern medicine because nature holds medicinal value for all.

No studies on the toxicity and epidemiology of bentonite on the skin were found in our own database search. The available research on this topic only indicates that the main exposure point of concern is the inhalation of bentonite dust during production (Adamis et al., 2005; Maxim et al., 2016). Various data on the chemical composition of natural clays used in pharmaceutical and cosmetic products vary widely, with SiO_2 composition reaching 63.37% and Al_2O_3 composition reaching 39.49% (López-Galindo, 2007). BS 15 had an Al content of about 6.42% and a Si content around 7.28%, while BS 17.5 had an Al content around 7.12% and a Si content around 8.07%. The Al and Si content of both types of soap was far below that found in the natural clay used in pharmaceutical and cosmetic products.

3.3. Scanning Electron Microscopy (SEM) of BS 15 and BS 17.5

The selection of raw materials and raw material additives, combined with the production process itself, greatly affects the quality of the final bar soap. A better understanding of the micro-crystallization of soap is needed, as this has a direct impact on the nature of the final product. The microstructure and composition of the soap are determined by the length of the hydrophobic fatty chain, by the degree of saturation of the fatty acid chain or the distribution of fatty acids, and by the cation effect.

Figure 3A shows a SEM image of pure bentonite at 250× magnification. A bright white layered pile shows the content of metal minerals, such as Si and Al, along with other metals. Figure 3B shows the SEM image of solid soap without bentonite filler; it appears bright and very homogenous, indicating that sodium metal is distributed and bound to the elements C, H_2, and O_2 in the form of the soap formula (RCOONa) and glycerol ($C_3H_8O_3$). Micro pores can also be seen on the surface of the soap and a slightly uneven distribution of metals. Figures 3C and 3D, by contrast, show the differences in the soaps following the addition of 15 g and 17.5 g of bentonite filler. The distribution of Si and Al minerals from bentonite particles indicates a less even distribution in BS 15 (Figure 3C) than in BS17.5 (Figure 3D). The overall distribution of metal minerals in both soap types appears uneven and piled up on the porous areas of the soap. A more even distribution of minerals could possibly be obtained by treating or modifying the bentonite before its addition to the soap. Previous research (Hasan et al., 2020) found that modified clay showed a better dispersion than clay that was not modified and led to differences in the physical properties of the vulcanized natural rubber that they obtained. Figure 3 shows that bentonite is compatible as a filler for solid soap made with coconut oil as its raw material and that its inclusion can affect the esthetics of the soap. As stated by Setyaningrum et al. (2020), the esthetic design is one factor that influences the success of a product.

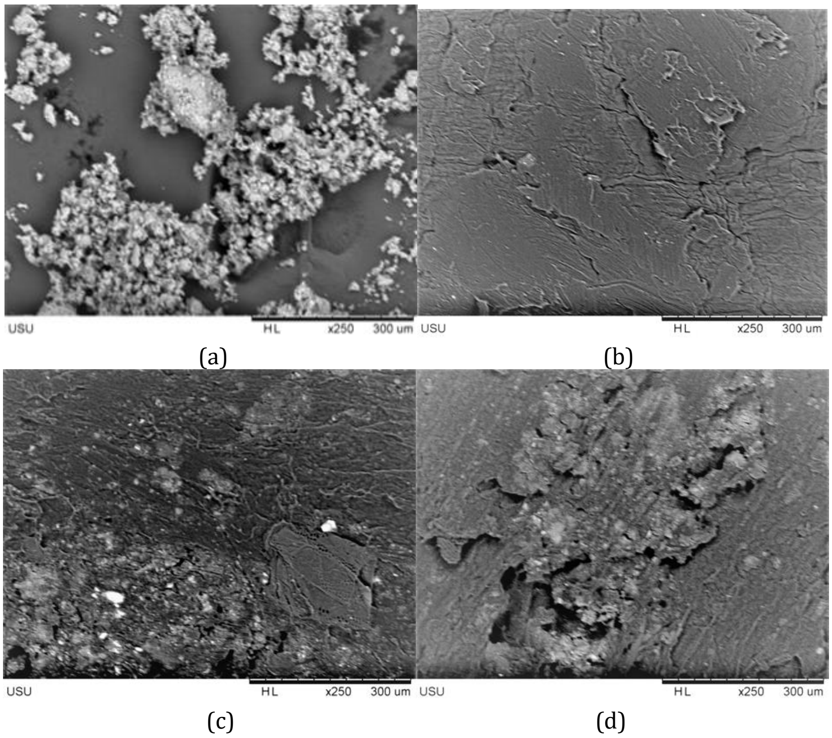

Figure 3 SEM Analysis: (a) Bentonite; (b) Soap without bentonite; (c) Bar soap with 15 g of bentonite filler (BS 15); and (d) Bar soap with 17.5 g of bentonite filler (BS 17.5)

Wati et al. (2019) carried out research using a simplex lattice experimental design for the optimization of bentonite in liquid detergents and found an optimal bentonite concentration of 6.4%. They stated that this resulting formulation can be used as an innovative agent for cleansing extreme *najis mughalladhah*. In the present study, both BS 15 and BS 17.5 had large amounts of mineral particles; therefore, both these soaps can likely be used as agents for cleansing extreme *najis mughalladhah*.

4. Conclusions

Soaps prepared by the addition 10–20 g bentonite filler showed characteristics that fulfill the SNI standards. The five tested soap formulas had zero free alkali content and a pH ranging from 8.7 to 8.8, indicating that the resulting bar soaps are extremely safe to use on the human body. The moisture content was relatively low and the free fatty acid content was also low, while the foam showed good stability. However, of these five soap formulations, the soap with 10% and 12.5 g bentonite appeared to be softer, whereas the soap containing 20 g bentonite showed a rapid onset of the trace process, to the point that the soap was difficult to mold. These separate factors affected the performance of the soap shape, so only two soaps, BS 15 and BS 17.5, with 15 and 17.5 g of bentonite filler, respectively, were analyzed further. Calculation of the soap composition using material balance theory and EDX analysis revealed that the addition of bentonite increases the mineral content of the soap preparation. A comparison of the amounts of C, O, Na, Si, and Al indicated the same trend. The EDX analysis clearly showed that the resulting soap only contains the metals Si and Al, which come from the added bentonite and are not harmful to skin health. No harmful heavy metals (such as Hg, Pb, Cu, or others) were found. Analysis of the morphology using SEM confirmed that the soap formulation is compatible with the addition of either amount of bentonite. The BS 17.5 had a more even and homogeneous distribution of mineral particles on the soap surface compared to the BS 15 soap; therefore, the BS 17.5 soap was deemed the best formula soap. It had a better appearance and it met all the parameters required by the SNI standards. The presence of Si and Al metals from the bentonite also means that this soap can be used as an agent for the cleansing of extreme *najis mughalladhah*. Therefore, further research should be conducted to obtain additional basic and accurate evidence. Aside from this, but no less important, is a need for further study of the effects of glycerin as a byproduct of the saponification process and of the cytotoxicity of bentonite soap and its capacity to hydrate the skin. Future research should aim at determining the safety and skin health benefits of bentonite clay soaps.

Acknowledgements

This work was supported by the TALENTA USU program with contract number 4167/UN5.1. R/PPM/2017; April 1, 2019.

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