

PUJI WAHYUNINGSIH¹
MUSLIMAH MUSLIMAH¹
YUSNAWATI YUSNAWATI²

¹Departemen of Chemistry,
Faculty of Engineering,
Universitas Samudra, Aceh,
Indonesian

²Faculty of Engineering,
Universitas Samudra, Aceh,
Indonesian

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LOCAL SALT PURIFICATION IN ACEH TIMUR REGENCY USING ALKALINE (KOH AND NaOH) ACTIVATED BENTONITE

Article Highlights

- The purification methods used to obtain a good quality salt
- Alkaline activation increase the characters of bentonite
- The activation process in bentonite increase in NaCl levels in local salts
- The alkaline activated-bentonite has binded Ca and Mg metals in local salts
- KOH-activated bentonite has a higher NaCl level than NaOH-activated bentonite

Abstract

Various purification methods have been developed to improve local salt quality, with the NaCl content above 94%. Bentonite is one of the materials used as binder impurities in purification. The purpose of the study was to synthesize a modified bentonite using an alkaline solution, which includes sodium hydroxide (NaOH) and potassium hydroxide (KOH) with various concentrations (1 M, 1.5 M, 2 M, and 2.5 M) and determined levels of Na⁺, Ca²⁺ and Mg²⁺ metals using AAS. The bentonite and activating agent 1 M (1:10) were stirred for three hours. Then, the bentonite was dried for two hours in an oven at 110 °C. The activated bentonite was dried for four hours at 170 °C. The activated bentonite was characterized using XRD, FTIR, and SEM. The basal spacing d_{001} on the diffractogram bentonite was significantly unaffected by alkaline activation. FTIR analysis represents the stretching vibration of -OH shifts towards a lower wavenumber. Alkaline activation reduced impurities from bentonite and increased the pore surface's porosity. NaCl levels in people's salt increased after being activated using alkaline. The increase in Na⁺ levels was followed by a decrease in Ca²⁺ and Mg²⁺ contents. KOH-activated bentonite had a higher NaCl level than NaOH-activated bentonite.

Keywords: bentonite, activation, alkaline, salt purification.

Salt, one of Indonesia's primary commodities, strategically staples consumption and industrial needs [1]. East Aceh Regency is one of the salt-producing areas in Aceh, with a total production of 433.85 tons/year. However, the amount only meets 26% of the people's salt needs (approximately

1,350 tons/year) [2]. Therefore, efforts are necessary to increase production and improve salt quality according to the Indonesian National Standard (SNI). The salt produced also contains impurities such as MgSO₄, CaSO₄, MgCl₂, KCl, and soil impurities [1].

Salt in East Aceh Regency is still processed conventionally using the evaporation method. This process results in low-quality salt that does not meet the quality standard of SNI 3556: 2010, requiring NaCl content above 94% and the part not soluble in water less than 0.5% w/w. This local salt could not be consumed directly both for consumption and industrial needs since its NaCl content is still under Indonesian National Standard (SNI) Number. The results showed that the NaCl content was 10.98%, the water content was 7%, the insoluble part in water was 4% w/w, and

Correspondence: P. Wahyuningsih, Departemen of Chemistry, Faculty of Engineering, Universitas Samudra, Aceh, Indonesian, 24416.

E-mail: puji_wahyuningsih@unsam.ac.id

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the iodine content was 42.49 mg/kg. Impurities, such as metals Ca and Mg, cause the NaCl low level. The impurities should be precipitated to obtain a good quality salt using purification methods.

Several purification methods have been developed to increase the level of NaCl in salt to fulfill requirements, including the hydro extraction method [3,4], recrystallization with the addition of $\text{Ca}(\text{OH})_2$, NaOH, and CaCO_3 [5,6], precipitation and evaporation methods [7,8], purification using sodium hydroxide (NaOH) [9], ion exchange method [10], and iodization method using a spray mixer [1]. A binder can minimize these impurities, such as NaOH, CaO, $\text{Ba}(\text{OH})_2$, and $(\text{NH}_4)_2\text{CO}_3$ [7]. Another usable binder to remove impurities in salt is bentonite. There has not been a thorough investigation on bentonite as a salt binder yet, even though bentonite is widely used for the removal of metal ions and other impurities from water [11], as well as the removal of both anionic [12] and cationic [13] pollutants.

Bentonite is a low-cost material because it is abundantly available in nature [14,15]. Bentonite is a type of clay mineral with layered structures of the 2:1 type, including mostly montmorillonite. Two silica (Si^{4+}) tetrahedral sheets and one alumina (Al^{3+}) octahedral sheet make up the fundamental structure of bentonite [16]. A net negative electric is produced by replacing some trivalent ions, like Al^{3+} , with some divalent ions, such as Fe^{2+} or Mg^{2+} in the octahedral layer, or by replacing Si^{4+} with Al^{3+} in the tetrahedral layer [17,18]. Various exchangeable hydrated alkali and alkaline earth metal cations occupy the bentonite's interlayer space to compensate for the charge deficiency [19]. Bentonite has unique physicochemical characteristics that transform it into an efficient adsorbent for different types of pollutants, such as low permeability, low cost, strong absorptive affinity with inorganic and organic substances, large specific surface, high porosity, high cation exchange capacity [18,20–23], good adsorption capacity, expansible interlayer space, flexible, and tunable acidity [18], micro and mesoporous particles [21].

In other words, the quality of bentonite needs improvement through activation or treatment prior to being used effectively in the industry. Bentonite may be activated in four main methods to enhance many features. The procedures are: (i) chemical, (ii) physical, (iii) pillaring, and (iv) thermal. Inorganic acids (such as H_2SO_4 or HCl) activate bentonite to replace the exchangeable ions of bentonite during chemical processes [17]. The activators used are strong acids and bases, such as chloric acid (HCl), nitric acid (HNO_3), sulphuric (H_2SO_4), phosphoric acid (H_3PO_4), calcium chloride (CaCl_2), magnesium chloride (MgCl_2),

zinc chloride (ZnCl_2), sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium carbonate (Na_2CO_3), and sodium chloride (NaCl). The activation of bentonite by acids and bases makes the exchangeable cations in its structure more effective [24]. Bentonites and other clays' mineralogical and physicochemical properties substantially change with acid and alkali treatments [25]. The activation can increase the pore size and surface area to increase the adsorption performance of metal impurities (Ca and Mg) in the recrystallization process and improve the quality of salt.

A promising way of activating bentonite-like clays is alkaline activation. This technique is more versatile than acidic activation in obtaining more active sorbents and having lower requirements for the initial feedstock. Alkaline metal hydroxides and mixtures of calcium hydroxide with salts of mineral acids (e.g., NaCl, Na_2SO_4 , Na_3PO_4 , Na_2CO_3 , etc.) are commonly used as alkaline agents. Alkaline treatment with sodium and calcium hydroxides using the salts mentioned above in the activating solutions will transform them into oxides of aluminum, iron, magnesium, and other metals, along with silica [24]. It may form an aluminosilicate component, calcium, aluminium hydrosilicates, and compounds with an intrinsically developed porous structure and acidic surfaces upon subsequent precipitation of the hydroxides and the activated clay.

The study aimed to synthesize and characterize bentonite from Aceh Tamiang and modified bentonite preheated with acid and alkaline solution. The acid and alkaline activation technique was applied for bentonite modification. Acid and alkaline were selected because of their activated bentonite with a high specific area, porosity, and adsorption capacity. The modified bentonite was studied to remove calcium and magnesium in salt water to increase the content of NaCl. Additionally, bentonite structure characteristics (raw and modified form) were investigated.

MATERIAL AND METHODS

Materials

The materials used were distilled water, aquabidest, a pH meter, Whatman 42 filter paper, sodium hydroxide (NaOH), and potassium hydroxide (KOH). They were obtained from E. Merck (Germany) in pro-analytical grade and used without further purification. Bentonite samples were collected from Aceh Tamiang Regency, Aceh Province, and salt samples from Aceh Timur Regency, Aceh Province.

Sample preparation

Sample preparation refers to the procedure

described elsewhere [26]. The bentonite was crushed and sieved using a 250-mesh sieve. Powder of bentonite (50 g) was put into the ultrasonic bath for 15 minutes with a power of 750 watts. Bentonite has been distributed at room temperature for equilibrium (about three days) to swell all the clay minerals. A filtration tank was filled with the bentonite solution and then agitated at a speed of 60 rpm. The solution was filtered, and the resulting sludge was called fraction 1. The filtrate resulting from stage I was stirred three times, left for three days, and filtered to produce a precipitate called fraction 2. Furthermore, the filtrate produced in stage II was stirred three times, let stand for seven days, and filtered until the sediment or fraction three was obtained. The filtrate in the final stage evaporated to produce fraction 4 [27,28].

Synthesis of activated-bentonite

Bentonite was crushed and repeatedly rinsed for three hours with distilled water. The solution was filtered using a centrifuge at 5000 rpm for 30 minutes. Then, the precipitate was dried for six hours at 110 °C, smoothed out, and sieved through a 270 mesh sieve. Additionally, sodium and potassium hydroxide at various concentrations (1 M, 1.5 M, 2 M, and 2.5 M) activated the bentonite. The bentonite and activating agent 1 M (1:10) mixture was agitated for three hours, and the filtrate was separated from the original mixture. The bentonite was neutralized by distilled water washing before being dried in a 110 °C oven for 2 hours. The activated bentonite was dried at 170 °C for 4 hours.

Samples characterization

Fourier transform infrared (FTIR) spectra of the materials (raw bentonite and modified bentonite) were recorded on a Shimadzu Prestige-21 FT-IR spectrophotometer. The X-ray diffractogram of the materials was recorded on an X-Ray Diffraction (XRD) Shimadzu S-6000. The X-Ray Diffraction data were obtained by XRD Multiflex Rigaku with Ni-filtered Copper K α radiation of wavelength 1.5406 Å operated at 40 kV and 30 mA. The surface morphology of the materials was measured by a scanning electron microscope (SEM) on JEOL JSM-6510.

The process of salt purification

Before crystallization, the salt product was purified using alkaline-activated bentonite as a binder for impurities. The salt sample was first dissolved to create a saturated salt solution. The alkaline-activated

bentonite was added to the salt solution in a 1:10 ratio. The mixture was then stirred for three hours. Filtration was used to separate the solid phase from the filtrate in the mixture. The obtained filtrate was then evaporated to crystallize it. After obtaining the NaCl crystal product, it was dried for two hours at 110 °C. Atomic Absorption Spectroscopy (AAS) measured the concentration of Na⁺ Ca²⁺, and Mg²⁺ metals released from the bentonite into a solution mixture. The predicted values for Na, Ca, and Mg yields were obtained using the quadratic model [29].

RESULTS AND DISCUSSION

The characteristics of NaOH activated-bentonite

X-ray diffraction analysis (XRD) is measured to confirm the structural changes of bentonite after activation using NaOH. According to the standard pattern COD Card Number 96-901-0958, the diffraction peaks of raw bentonite are observed at $2\theta = 19.7^\circ$, 26.5° , 34.91° , 54.34° , and 61.80° , which characterizes the bentonite structure with the montmorillonite type [30]. The diffraction peaks d_{001} of the raw bentonite at a low angle of $2\theta = 2^\circ - 10^\circ$ is a characteristic peak of smectites, which is the main clay mineral of bentonite. Unfortunately, this study did not detect the diffraction peaks d_{001} of the raw bentonite at $2\theta < 10^\circ$ position.

The diffraction peaks d_{001} for NaOH activated-bentonite shifted to the left with increasing concentration of NaOH used in the activation process (Figure 1). It was confirmed that the smectite phase was present when a typical characteristic peak of the bentonite d_{001} , activated using 1 M NaOH, was at a 2θ value of 5.56° (d spacing 15.87 Å). With increasing NaOH activation concentration, the d_{001} peak shifted to the right and decreased the basal spacing value. After activation with NaOH 1.5 M, 2 M, and 2.5 M, the diffraction peaks of bentonite are 5.73° (d spacing 15.38 Å), 5.34° (d spacing 16.50 Å), and 5.48° (d spacing 16.09 Å), respectively. The cation exchange capacity was preferable in the presence of sodium cations between layers because sodium cations (Na⁺) are more easily exchanged (except Li⁺), and sodium has the greatest hydration strength and lower valence, reducing the bond strength with the bentonite mineral surface. The reaction was an exchange reaction between Ca²⁺ ions in the interlayer of the mineral bentonite with Na⁺ ions in an alkaline solution.

The FTIR spectra of NaOH-activated bentonite and bentonite are represented in Figure 2. The absorption peaks of bentonite without activation appear at wave numbers, namely 466.77 cm⁻¹; 632.65 cm⁻¹; 979.84 cm⁻¹; 1103.28 cm⁻¹; 1654.92 cm⁻¹; 2040.69 cm⁻¹; 2441.88 cm⁻¹; 2781.35 cm⁻¹; 3402.43 cm⁻¹; 3606.89 cm⁻¹; and 3645.46 cm⁻¹.

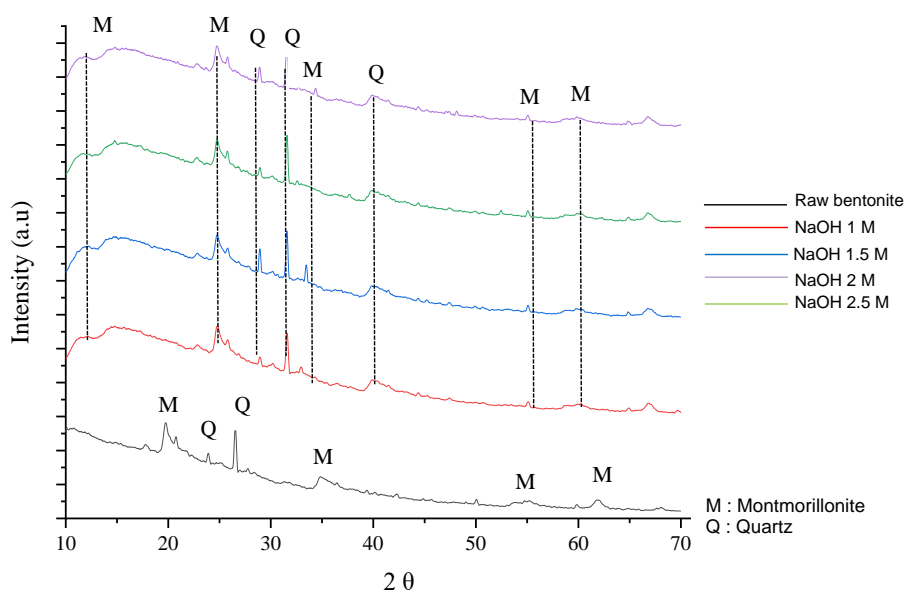


Figure 1. Diffractogram XRD NaOH-activated bentonite.

The absorption peaks at 3606.89 cm^{-1} and 3402.43 cm^{-1} were caused by stretching vibrations of the $-\text{OH}$ group from the bentonite structure and water molecules. The absorption band at 1654.92 cm^{-1} was the deformation of the OH group on the water molecule, while the absorption band at 1103.28 cm^{-1} was the stretching vibration of Si-O-Si . The peak in the lower wavenumber region (1200 cm^{-1} – 400 cm^{-1}) indicated that bentonite is dominated by bentonite groups. The absorption bands at 979.84 cm^{-1} , 632.65 cm^{-1} , and 632.65 cm^{-1} were Al-Al-OH stretching, Si-O-Si stretching, and Si-O-Si bending, respectively [31,32]. Activation of NaOH did not produce a significant change in the absorption of bentonite. Instead, the absorption shifted to a lower wavenumber region than bentonite without activation. In NaOH -activated bentonite, absorption appears at 931 cm^{-1} due to the OH bending vibration in octahedral Al-Al-OH [31].

The wavenumber of 3448 cm^{-1} in the unactivated bentonite (Figure 1) shows the stretching vibration of the $-\text{OH}$ group being hydrated by the adsorbed water molecules. There was an absorption band at 1635.26 cm^{-1} , representing the H-O-H bending vibration of the water in the bentonite interlayer [27]. The appearance of a strong absorption band with high intensity at 1036.98 cm^{-1} showed the asymmetric stretching vibration of Si-O . The $-\text{OH}$ stretching vibration shifts towards a lower wavenumber with increasing NaOH concentration. The interaction of octahedral $-\text{OH}$ with Na^+ cations in NaOH activator solution causes the interaction of the O and H bonds in the $-\text{OH}$ group to weaken, resulting in a decrease in wavenumber and intensity of adsorption peak $-\text{OH}$ groups.

Raw bentonite has a smooth aggregate surface and more densely packed flakes (Figure 3a). However, when activated with NaOH , the aggregate looks foggy and ragged (Figure 3 (b–e)). It is consistent with the specific surface areas of the NaOH -activated bentonite. The clay surface has become more porous due to the leaching of cations and impurities. The leaching increased the pore surface area and caused the creation of new pores [33]. The surface of the raw bentonite sample (Figure 3a) had larger pores between the particles than the NaOH -activated bentonite [34].

The characteristics of KOH activated-bentonite

The structural changes in the bentonite to the alkaline treatment were studied using the X-ray diffraction technique. Figure 4 depicts the XRD profile of the raw bentonite and KOH -activated bentonite at various concentrations. A typical characteristic peak of the d_{001} of bentonite activated using 1 M KOH was at a 2θ value of 5.48° (d spacing 16.09 \AA), confirming the smectite phase's presence. The d_{001} peak shifted to the left and caused an increase in the basal spacing value with increasing KOH activation concentration. The diffraction peaks d_{001} of bentonite after activation using KOH 1.5 M , 2 M , and 2.5 M are 5.46° (d spacing 16.15 \AA); 5.40° (d spacing 16.32 \AA), and 5.36° (d spacing 16.44 \AA), respectively. The displacement of the diffraction peaks at a low angle was followed by a decrease in the intensity of the peaks, indicating a decrease in crystallinity in the presence of activated bentonite using KOH solution.

The wavenumber 3448 cm^{-1} in unactivated bentonite, as shown in Figure 5 (a–e), shows the stretching vibration of the OH group of the silanol group

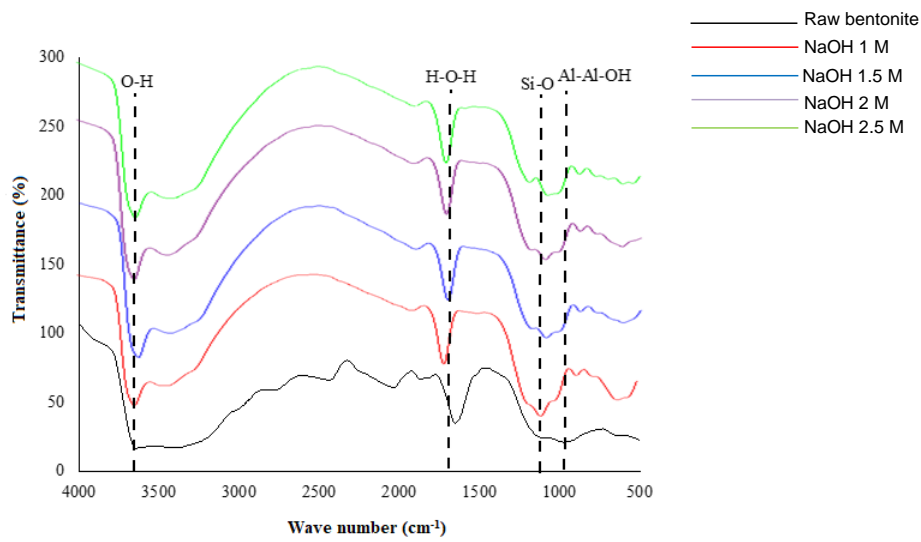


Figure 2. FTIR Spectrum NaOH-activated bentonite.

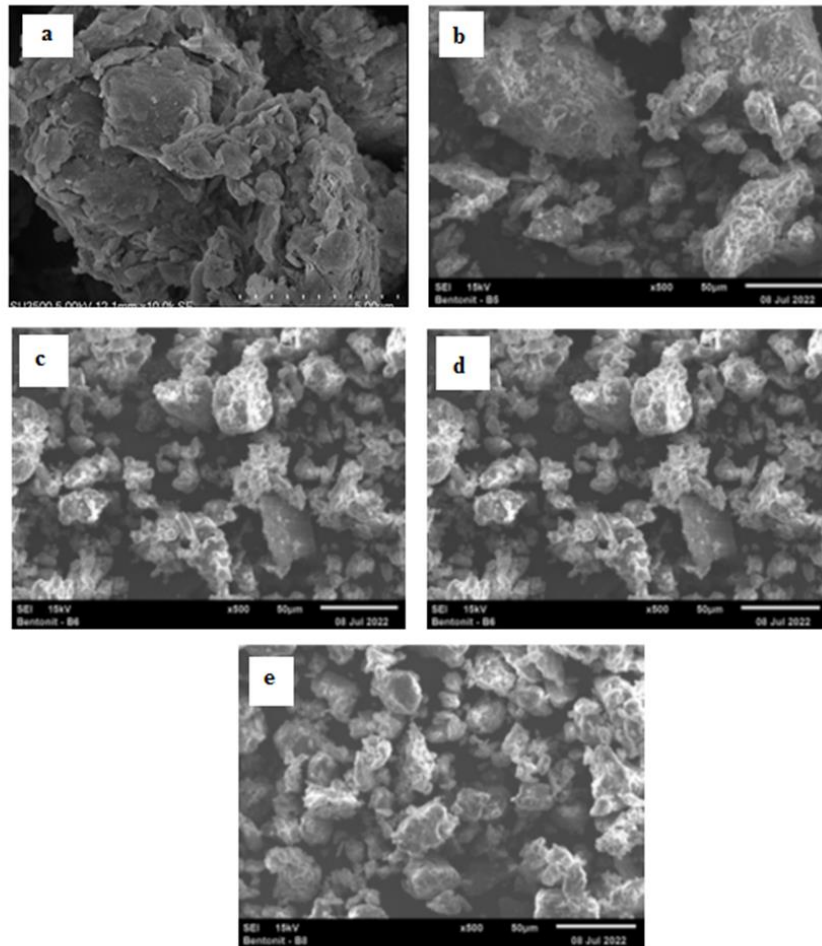


Figure 3. Micrograph SEM NaOH-activated bentonite.

(Si-OH) coordinated to the octahedral Al³⁺ cation [31]. The -OH stretching vibration shifts towards a lower wavenumber with increasing KOH concentration. The

interaction of the -OH octahedral with the K⁺ cation in the KOH activator solution causes the interaction of the O and H bonds in the -OH group to weaken, decreasing

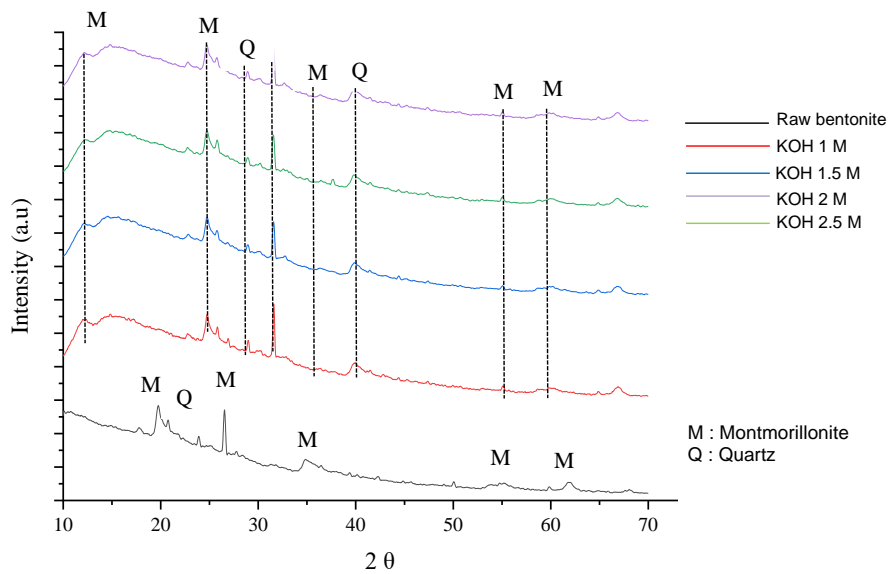


Figure 4. Diffractogram XRD KOH-activated bentonite.

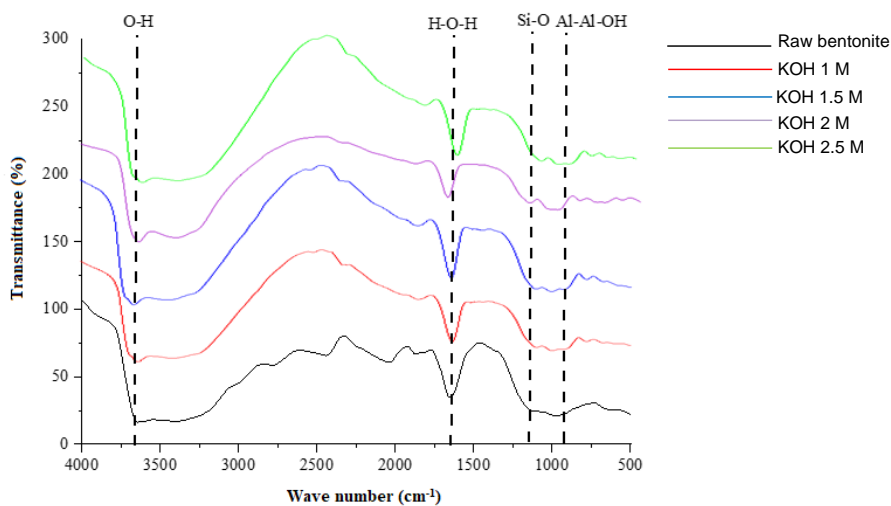


Figure 5. FTIR Spectrum KOH-activated bentonite.

the wavenumber. There was an absorption band at 1635.26 cm^{-1} , which represents the H-O-H bending vibration of water, and an absorption band at 1036.98 cm^{-1} for the asymmetric stretching vibration of Si-O-Si [31].

The SEM micrograph in Figure 6(a–e) shows that the bentonite microstructure has a smaller pore, a more uniform distribution, and a prism type [32]. The surface of natural bentonite seems to be a smooth aggregate and contains more densely packed flakes (Figure 6a). The surface of bentonite activated with KOH forms coarser aggregates than bentonite without activation (Figure 6b–e). After alkaline treatment, the particles became smaller and thinner [33]. The surface of bentonite becomes more porous due to the leaching of cations and impurities after treatment with KOH. KOH activation in bentonite results in more open pores. The

higher the concentration of KOH, the smaller the particle size and the more uniform the pore size distribution, and vice versa. Smaller pore size indicates a larger pore surface area, thereby increasing the adsorption capacity of heavy metals.

The effect of alkaline concentration of activated-bentonite on NaCl level

The activator used was a strong base, namely NaOH and KOH. The performance of alkaline-activated bentonite and bentonite as activators in the purification process of people's salt produced by salt farmers in East Aceh Regency was conducted by determining the levels of Na^+ , Ca^{2+} , and Mg^{2+} metals using Atomic Absorption Spectrophotometer (AAS). The results of AAS analysis of Na^+ , Ca^{2+} , and Mg^{2+} metals before and

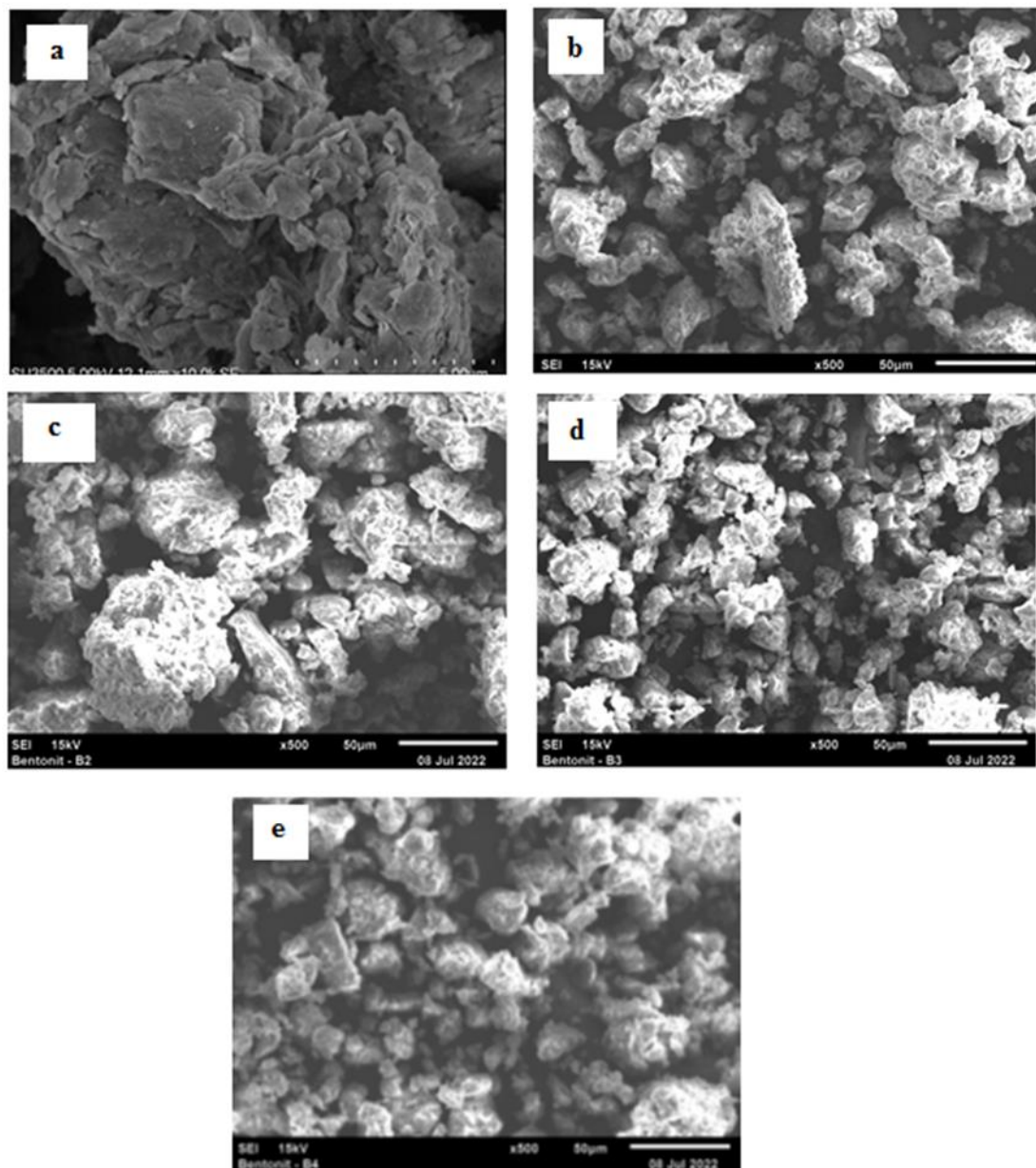


Figure 6. Micrograph SEM KOH-activated bentonite.

after recrystallization using KOH and NaOH are shown in Figure 7 and Figure 8.

Figure 7 and Figure 8 represent the levels of Na^+ , Ca^{2+} , and Mg^{2+} metals before and after recrystallization using strong bases (NaOH and KOH). Na^+ , Ca^{2+} , and Mg^{2+} values in people's salt before leaching using NaOH and KOH are 3032.50 ppm, 48.89 ppm, and 533.03 ppm, respectively. Figure 7 displays Na^+ levels in people's salt increased after being activated using 1 M KOH is 4004.50 ppm and decreased with increasing KOH concentration are 3796.05 ppm (1.5 M), 3829.05 ppm (2 M), and 3755.60 ppm (2.5 M). The increase in NaCl levels was followed by a decrease in the levels of Ca^{2+} and Mg^{2+} metals because the activation process can increase the pore size and

surface area to increase the adsorption performance of activated bentonite against metal impurities, such as Ca^{2+} and Mg^{2+} in the crystallization process and improve the quality of the recrystallization results. The activation increases the surface area and porosity. In addition, bentonite also has a lot of negative charges (O⁻) contained in the tetrahedral and octahedral layers, which can bind metals such as Ca and Mg.

The Na^+ level in KOH-activated bentonite is greater than that of NaOH-activated bentonite. The Na^+ levels in people's salt increased after being activated using NaOH 1 M is 3704.10 ppm and increased with increasing KOH concentration are 3794.90 ppm (1.5 M), 3953.55 ppm (2 M), and 3846.25 ppm (2.5 M). The high Na^+ level was due to the presence of Na^+

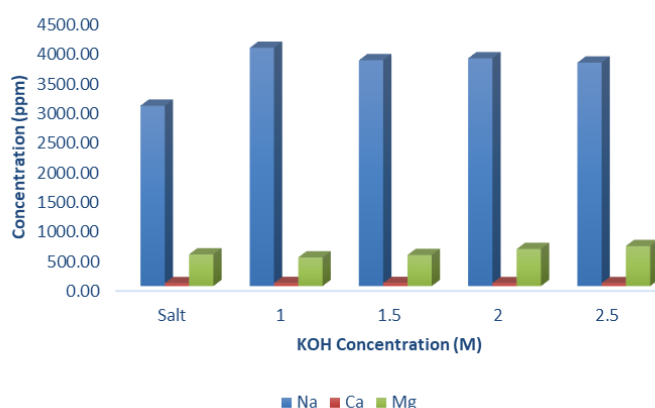


Figure 7. The results of AAS analysis of NaCl, and Ca and Mg metals before and after recrystallization using KOH-activated bentonite.

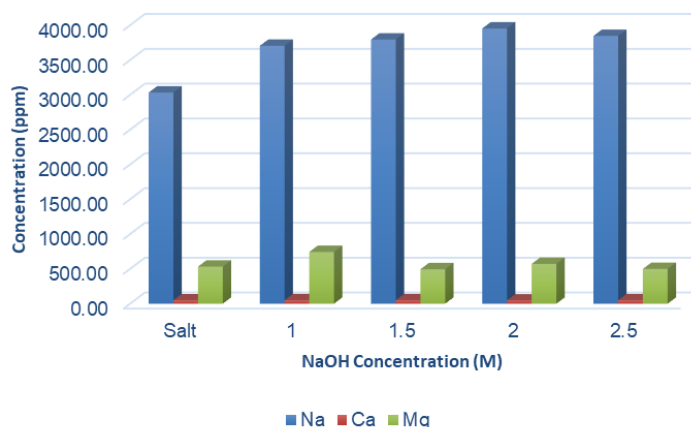


Figure 8. The results of AAS analysis of NaCl, and Ca and Mg metals before and after recrystallization using NaOH-activated bentonite.

cations unexchanged between bentonite layers, while in KOH activation, Na^+ cations will be exchanged with K^+ from the KOH activator. The concentration of NaOH producing the highest level of Na^+ is 2 M. At a concentration of 2 M, Ca^{2+} and Mg^{2+} metals will precipitate to form $\text{Ca}(\text{OH})_2$ and $\text{Mg}(\text{OH})_2$ during the filtration process before recrystallization [9]. It follows the research by [10], stating that a concentration of 2 M is very effective in increasing Na^+ levels. The overly high concentration of NaOH makes the bentonite structure unstable due to the dealumination process, so the NaCl content decreases at 2.5 M NaOH concentration.

CONCLUSION

Significantly, the activation of bentonite using alkaline activation (NaOH and KOH) have not resulted in changes to the physicochemical properties of bentonite. However, it can remove impurities contained in bentonite. Therefore, it can increase pore size and porous in surface materials. According to XRD analysis

data, alkaline activation to bentonite causes a minor shift toward a higher angle and does not alter the basal spacing d_{001} on the diffractogram. The FTIR analysis indicated that the stretching vibration of $-\text{OH}$ shifts towards a lower wavenumber as alkaline concentration increases. The wavenumber is reduced because the $-\text{OH}$ of the octahedral becomes weak due to contact cations in the layer with the activator solution. The NaCl level in KOH-activated bentonite was greater than that of NaOH-activated bentonite. The increase in Na^+ levels was followed by a decrease in the levels of Ca^{2+} and Mg^{2+} metals because the activation process can increase the pore size and surface area to increase the adsorption performance of activated bentonite against metal impurities, such as Ca^{2+} and Mg^{2+} in the crystallization process, and improve the quality of the recrystallization results.

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PUJI WAHYUNINGSIH¹
MUSLIMAH MUSLIMAH¹
YUSNAWATI YUSNAWATI²

¹Departemen of Chemistry,
Faculty of Engineering,
Universitas Samudra, Aceh,
Indonesian

²Faculty of Engineering,
Universitas Samudra, Aceh,
Indonesian

PROČIŠĆAVANJE LOKALNE SOLI U REGIONU ACEH TIMUR KORIŠĆENJEM ALKALNOG (KOH i NaOH) AKTIVISANOG BENTONITA

Razvijene su različite metode prečišćavanja za poboljšanje kvaliteta lokalne soli, sa sadržajem NaCl iznad 94%. Bentonit je jedan od materijala koji se koristi za vezivanje nečistoća u postupku prečišćavanju. Cilj istraživanja je bio da se sintetiše modifikovani bentonit korišćenjem alkalnog rastvora, koji uključuje natrijum hidroksid (NaOH) i kalijum hidroksid (KOH) različitih koncentracija (1 M, 1,5 M, 2 M i 2,5 M) i utvrđenih nivoa Na, Ca. i Mg pomoću AAS. Bentonit i sredstvo za aktiviranje 1 M (1:10) su mešani tri sata. Zatim je bentonit sušen dva sata u pećnici na 110 °C. Aktivirani bentonit je sušen četiri sata na 170 °C i karakterisan metodama KSRD, FTIR i SEM. Bazalni razmak d_{001} na difraktogramu bentonita nije značajno pod uticajem alkalne aktivacije. FTIR analiza je ukazala na pomeranje vibracije istezanja -OH ka nižem talasnom broju. Alkalna aktivacija je smanjila nečistoće u bentonitu i povećala poroznost površine pora. Nivoi NaCl u soli za ishranu se povećavaju nakon alkalne aktiviranja. Povećanje nivoa Na^+ praćeno je smanjenjem sadržaja Ca^{2+} i Mg^{2+} . Bentonit aktiviran KOH imao je viši nivo NaCl od bentonita aktiviranog NaOH.

Ključne reči: bentonit, aktivacija, alkalna, prečišćavanje soli.

NAUČNI RAD