

# Synthesis and characterization of acid-activated bentonite from Aceh Tamiang

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**Abstract.** Bentonite is new class of microporous materials with high surface areas which have been extensively studied as interesting material in catalyst application and adsorbents. The characterization of bentonite were performed using Infrared spectrophotometer, X-ray Diffraction, and Transmission Electron Microscopy (TEM) instruments. The results showed that the characteristic of natural bentonite and acid-activated bentonite does not give significant different results. XRD pattern of natural bentonite and acid-activated bentonite indicates those bentonites have the main content such as montmorillonite, quartz and alumina. The TEM profile showed the basal spacing in layered structure of acid-activated bentonite was increasing compared to the natural bentonite. The natural bentonite showed the characteristic absorption peaks such as Si-O, Al-O, Si-O-Si which is observed at wave numbers  $1149,57\text{ cm}^{-1}$ ,  $945,12\text{ cm}^{-1}$ ,  $640\text{ cm}^{-1}$ , and  $459\text{ cm}^{-1}$  but the acid-activated bentonite only showed the characteristic absorption peaks such as Si-O and Al-O which is observed at wave numbers  $1018,41\text{ cm}^{-1}$  and  $493,78\text{ cm}^{-1}$ . The absorption intensity of stretching -OH peak on acid-activated bentonite higher than natural bentonite. It indicates that the bentonite after activation is more pure compared to bentonite before activated.

## 1. Introduction

Clay is a new class mineral of phyllosilicate family with a layered structure and crystalline shaped. The type of clay has attracted considerable attention is bentonite. Bentonite is a type of rock which main content materials is montmorillonite (MMT). The characteristic of bentonite is swelling in water, intercalation and ion exchange that make this material interesting to use as catalyst, pillared clay, nanoclay, adsorbent material, membrane and nanocomposite polymer. The main content of bentonite is the montmorillonite (85%) with the chemical formula  $\text{M}_x(\text{Al}_{4-x}\text{Mg}_x)\text{Si}_8\text{O}_{20}(\text{OH})_4 \cdot n\text{H}_2\text{O}$  [1].

Bentonite belongs to the 2:1 clay family, the basic structural unit of which is composed of two tetrahedrally coordinated sheets of silicon ions surrounding a sandwiched octahedrally coordinated sheet of aluminum ions. The isomorphous substitution of  $\text{Al}^{3+}$  for  $\text{Si}^{4+}$  in the tetrahedral layer and  $\text{Mg}^{2+}$  or  $\text{Zn}^{2+}$  for  $\text{Al}^{3+}$  in the octahedral layer results in a net negative surface charge on the clay [2] (Eren dan Afsin, 2008).

Recently montmorillonite is mineral has attracted considerable attention and widely used in various industrial applications due to advantage such as high aspect ratio, easier to obtained, low costs and economic in the processing [3] (Dobrzanski, et al., 2012). It's supported by the fast development of nanoscale science and technology, mainly due to the availability of new strategies for synthesizing nanomaterials [4] (Pavlidou, et al., 2008).

The characteristics of bentonite as an adsorbent are high surface area and pore size, but the use of bentonite as an adsorbent has disadvantages such as swelling properties, low thermal and hydrothermal stability (below 600–700°C) [5] (Kloprogge et al., 2005). To improve the ability of adsorption on bentonite can be done by chemical activation using acid. Activation with acids is intended to dissolve impurities found on the bentonite lattice and surface, besides it was increase acid sites in the bentonite surface. Activation using  $\text{H}_2\text{SO}_4$  is also used to exchange cations ( $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Ca}^+$ ) found in the interlayer of bentonite with  $\text{H}^+$  from sulfuric acid. The bentonite surface will become more acidic and it is expected can increase the pore size and the adsorption ability [6] (Nafsiyah et al, 2017).

In this study, the synthesis and characterization of acid-activated bentonite is focused. The characterization of bentonite were performed using Infrared spectrophotometer, X-ray Diffraction, and Transmission Electron Microscopy (TEM) instruments.

## **2. Material and Method**

### **2.1 Material**

The bentonite was collected from Aceh Tamiang, Aceh (Indonesia) in August 2018.

### **2.2 Instrument**

Infrared spectrophotometer (Prestics-21 Shimadzu FTIR), X-ray Diffraction (XRD-6000 Shimadzu), and Transmission Electron Microscopy (TEM) (JEOL JEM-1400).

### **2.3 Preparation Samples**

The bentonite was ground and filtered by a 250 mesh sieve. 50 g of bentonite powder was put into an ultrasonic batch which was filled with 2 L of distilled water for 15 minutes. The solution was filtered and the sediment yielded is called as fraction 1. The filtrate produced from stage I is stirred 3 times and left to stand for 3 days and filtered to give second sediment which is called as fraction 2. Furthermore, the filtrate produced in stage II is stirred 3 times and left to stand for 7 days and filtered to obtained third sediment or fraction 3. The last filtrate produced was evaporated to give fraction 4 [7] (Wahyudi, 2010).

### **2.4 Activation Acid of Bentonit.**

The bentonite is prepared by chemical activation with various concentration of sulfuric acid of 1 M, 1.5 M, and 2 M (ratio 1: 4). The mixture was kept under stirring at room temperature for 6 h, the sediment was separated from slurry by filtration. The filtrate was then neutralized by adding excess water to remove  $\text{SO}_4^{2-}$  ion (tested by  $\text{BaCl}_2$  solution). Finally, the acid-activated bentonite obtained was dried in oven at 105°C for 4 hour.

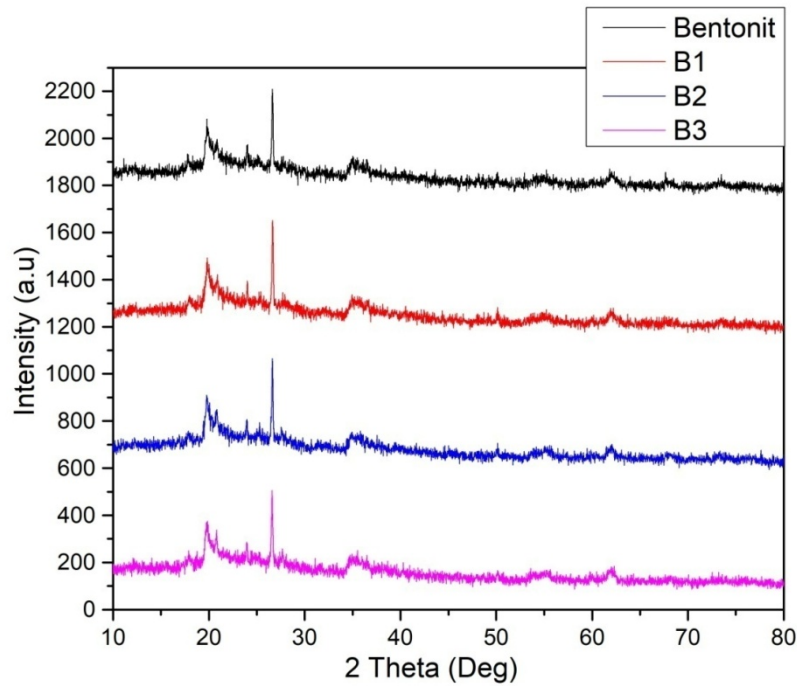
### **2.5 Acid Activated of Bentonit Characterization**

The diffractogram of natural bentonite and the acid-activated bentonite were performed using XRD Multiflex Rigaku with Ni-filtered Copper  $\text{K}\alpha$  radiation of wavelength 1.5406 Å, operated at 40 kV and 30 mA. Transmission electron microscopy (TEM) (JEOL JEM-1400) was used to observe the surface morphology of the natural bentonite and acid-activated bentonite. The existence functional groups of materials was analyzed by FTIR measurement.

## **3. Result and Discussion**

### **3.1 X-ray Analysis**

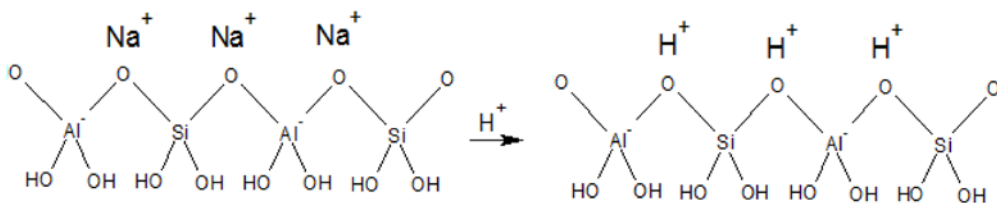
The XRD pattern of the natural bentonite and acid-activated bentonite with various concentration (1 M; 1.5 M; 2 M) in the range of  $2\theta = 2 - 65^\circ$  is represented in Figure 1.



**Figure 1.** XRD pattern of (a) natural bentonite (b) acid-activated bentonite

Based on Figure 1, bentonite shows peaks at  $2\theta = 20, 54, 26, 58, 50, 18, \text{ and } 16, 52$  which indicates that bentonite has the main content such as montmorillonite, quartz and alumina. this result is similar to the study conducted by [8] Er-Ramli and Abdebali (2014) and [9] Jesenak and Hlavaty (2000) which reported that bentonite has the main content of montmorillonite, feldspar, quartz and magnetite.

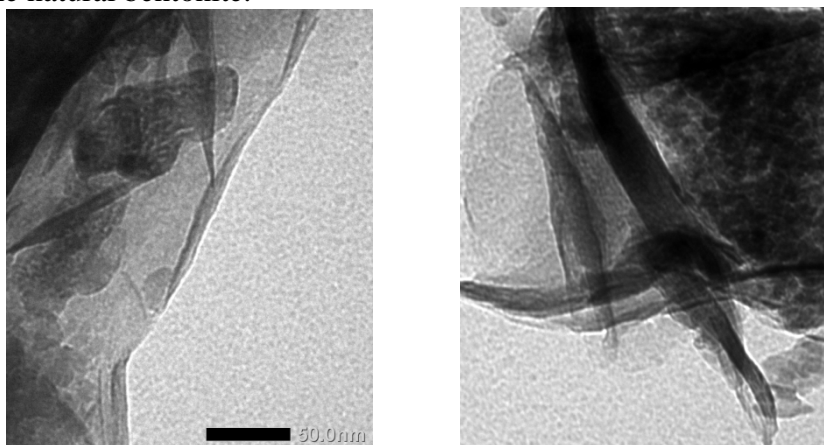
Activation with acids is intended to dissolve impurities found on the bentonite lattice and surface, besides it was increase acid sites in the bentonite surface. Activation using  $\text{H}_2\text{SO}_4$  is also used to exchange cations ( $\text{Na}^+$ ,  $\text{K}^+$ , and  $\text{Ca}^+$ ) found in the interlayer of bentonite with  $\text{H}^+$  from sulfuric acid. The bentonite surface will become more acidic and it is expected can increase the pore size and the adsorption ability [5]. The mechanism of cation exchange is shown in Figure 2.



**Figure 2.** Mechanism of cation exchange in bentonite

### 3.2 Transform Electron Microscopy Analysis

The TEM profile of both the natural bentonite and acid-activated bentonite were shown in Figure 3, basal spacing in layered structure of acid-activated bentonite was increasing compared to the natural bentonite.

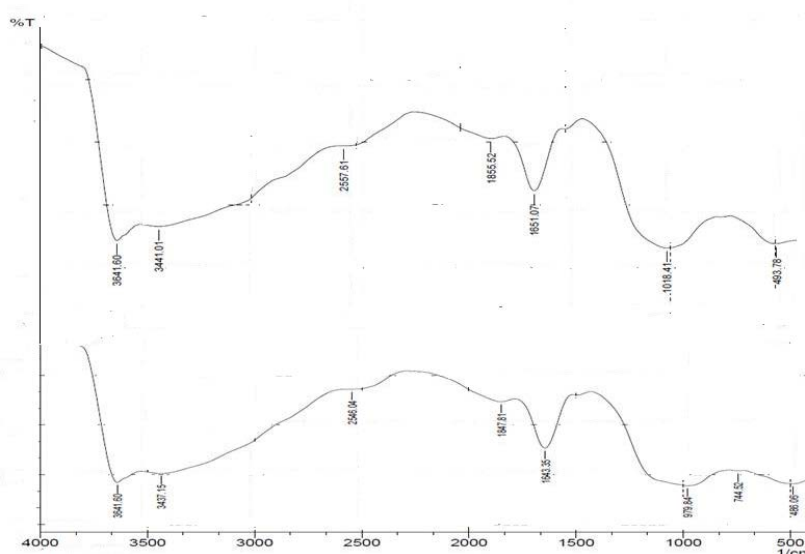


**Figure 3.** TEM profile of (a) natural bentonite (b) acid-activated bentonite

### 3.3 Fourier Transform Infrared (FTIR) Spectroscopy

The functional groups found on bentonite surfaces were identified by FTIR instrument. The FTIR spectra showed that the activation of bentonite using  $H_2SO_4$  does not change the characteristics of the functional groups in bentonite as shown in Figure 4. The natural bentonite showed the characteristic absorption peaks such as Si-O, Al-O, Si-O-Si which is observed at wave numbers  $1149,57\text{ cm}^{-1}$ ,  $945,12\text{ cm}^{-1}$ ,  $640\text{ cm}^{-1}$ , and  $459\text{ cm}^{-1}$  but the acid-activated bentonite only showed the characteristic absorption peaks such as Si-O and Al-O which is observed at wave numbers  $1018,41\text{ cm}^{-1}$  and  $493,78\text{ cm}^{-1}$ .

Figure 4 showed the absorption intensity of stretching -OH peak on acid-activated bentonite higher than natural bentonite. It indicates that the bentonite after activation is more pure compared to bentonite before activated.



**Figure 4.** FTIR spectra of (a) natural bentonite (b) acid-activated bentonite

#### 4. Conclusion

The synthesis and characterization of bentonite showed that the characteristic of natural bentonite and acid-activated bentonite does not give significant different results. XRD pattern of natural bentonite and acid-activated bentonite indicates those bentonites have the main content such as montmorillonite, quartz and alumina. The TEM profile showed the basal spacing in layered structure of acid-activated bentonite was increasing compared to the natural bentonite. The natural bentonite showed the characteristic absorption peaks such as Si-O, Al-O, Si-O-Si which is observed at wave numbers  $1149,57\text{ cm}^{-1}$ ,  $945,12\text{ cm}^{-1}$ ,  $640\text{ cm}^{-1}$ , and  $459\text{ cm}^{-1}$  but the acid-activated bentonite only showed the characteristic absorption peaks such as Si-O and Al-O which is observed at wave numbers  $1018,41\text{ cm}^{-1}$  and  $493,78\text{ cm}^{-1}$ . The absorption intensity of stretching -OH peak on acid-activated bentonite higher than natural bentonite. It indicates that the bentonite after activation is more pure compared to bentonite before activated.

#### Acknowledgments

We would like to thank Universitas Samudra for Grant of Penelitian Dasar Unggulan Scheme and technical supports.

#### 5. References

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