The preparation and characterization of bentonite nanoparticle from Bener Meriah, Indonesia

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Abstract. The isolation and the content determinations of bentonites obtained from Bener Meriah, Aceh, Indonesia has been carried out. The process involved dissolving bentonites in water, ultrasonication and gravitational sedimentation process. Bentonites were milled with a planetary high energy ball-mill to obtain nanoparticles of montmorillonite with the milling times varied at 20 and 30 hours. Ultrasonication and sedimentation process increased the montmorillonite content from 1.53 % to 5.22 %. The diverse milling times of 20 and 30 hours respectively produced montmorillonite with average size of 388 nm and 186nm.

1. Introduction

Bentonite is a type of rock which contains mineral such as montmorillonite. Its distinctive characteristic is known to be able to swell in water, and do intercalation (ion-exchange) makes this material attractive as catalyst/ catalyst support, organo clay, pillared clay, nanoclay, absorbent material, membrane and nanocomposite polymer [1].

The use of bentonite in the country is large enough for various industrial purposes. This can be seen from the needs of Ca-bentonite for the cooking oil industry, basic chemicals, and non-metallic minerals which in 1993 consumed about 90.4% of total bentonite consumption of 20,498 tons. The oil purification industry is the largest consumer of bentonite as a CPO pallor material, estimated at about 200,000 tonnes of bentonite is required by the oil purification industry [2].

Montmorillonite is the main mineral contained by bentonite. The quality and characteristics of bentonite largely depend on the quality and quantity of the montmorillonite it contains [3]. Pure montmorillonite can be utilized in a variety of areas of use, such as carbon photocopier papers, selective adsorbents, treatment, membranes, organoclay, polymer clay, pillared clay, nanoclay and catalyst production. Previous research on the isolation of montmorillonite with ultrasonication and sedimentation method has been successfully conducted with montmorillonite content of 24% [4].

The development of nanotechnology can not be separated from research on nanomaterials. The development of nanoparticle synthesis method is one of the fields that attract many researchers. Nanoparticles can occur naturally or through a process of synthesis by humans. The synthesis of nanoparticles means the manufacture of nanoparticles of less than 100 nm and at the same time altering their properties or functions [5].

2. Materials and methods

2.1. Materials

Bentonite was obtained from Kecamatan Pintu Rime Gayo, Kabupaten Bener Meriah, Indonesia.
2.2. Bentonite preparation
Bentonite was pounded until smooth and filtered with a 250 mesh. After that, it was weighed as much as 45 g and incorporated into an ultrasonic bath that has been filled with 2 liters of distilled water and given with ultrasonic wave for 15 minutes. The filtrate was stirred 3 times for 3 days and the precipitate was taken and is called fraction 2. The filtrate was stirred 3 times and was allowed to stand for 6 days. Then, the precipitate was taken and categorized as fraction 3. Finally, the filtrate was fully.

2.3. Bentonite nanoparticle preparation
Montmorillonite was pounded with Planetary Ball Mill (PBM) and then analyzed with Particle Size Analyzer (PSA) to determine the size of the particles of the morillonite.

2.4. Bentonite characterization
Characterization of the use of the bentonite function using the FT-IR spectrophotometer, the determinations of the compounds contained in the samples of bentonite using an XRD spectrophotometer and the determination of the theoretical dimensions of the manganese films using a Particle Size Analyzer (PSA).

3. Result and Discussion
3.1. Bentonite analysis by sedimentation and ultrasonication methods
Based on the ultra-sonication and sedimentation process, the biggest fraction was fraction 1 at 50% where fraction 1 has the largest particle size, so it easily settled after being left for 15 minutes. Fraction 1 is a coarse fraction of bentonite and showing a fraction with a large density of the species.

Fraction 2 was obtained at 25%, where fraction 2 had smaller particle size compared to fraction 1, where fraction 2 was found to had finer particle and had less density than fraction 1.

Fractions 3 and 4 are the smallest fractions with 13.5% and 11.25% respectively and possess the lesser density than fractions 1 and 2. These fractions are minerals of small gravity and indicating the presence of montmorillonite content in which montmorillonite minerals have the smallest density compared to other minerals [4].

With ultrasonic waves giving rise to disaggregating process, the montmorillonite particles were separated from the impurities (other compounds were formed). The overdue ultrasonic waves may cause damage to the montmorillonite structure, so that the ultrasonication process was conducted for only 15 minutes [6].

3.2. X-Ray Diffraction (XRD) analysis of raw bentonite
The X-ray diffraction characterization of the natural bentonite extracted in the True-Fluid area can be seen in Figure 1. The peak identification results show that this bentonite contains Montmorillonite minerals. Besides montmorillonite, this bentonite also contains anorthite, quartz and clinoptilolite minerals.

![Figure 1. XRD of bentonite](image-url)
Based on the result above, it can be seen that the biggest fraction obtained from bentonite is Anorthite with 76.76%. The lattice parameter is recorded to be $a = 8.180 \, \text{Å}$, $b = 12.873 \, \text{Å}$, $c = 14.223 \, \text{Å}$ and $\alpha = 93.44^\circ$, $\beta = 116.305^\circ$, and $\gamma = 90.21^\circ$ with triclinic crystal form.

The Quarts fraction has a mass percentage of 12.96% with the lattice parameter of $a = 4.699 \, \text{Å}$, $b = 4.699 \, \text{Å}$ and $c = 5.137 \, \text{Å}$, $\alpha = \beta = 90^\circ$ and $\gamma = 120^\circ$ with a hexagonal crystal form. The Clinoptilolite fraction has a mass percentage of 8.75% with lattice parameter of $a = 17.00 \, \text{Å}$, $b = 16.56 \, \text{Å}$, $c = 7.28$ dan $\alpha = \gamma = 90^\circ$ dan $\beta = 118.1^\circ$ with monoclinic crystal form. The smallest fraction is montmorillonite with a mass percentage of 1.53% and lattice parameter of $a = 5.64 \, \text{Å}$, $b = 5.64 \, \text{Å}$, $c = 9.77$ and $\alpha = \gamma = 90^\circ$ dan $\beta = 88.9^\circ$ with monoclinic crystal form. Therefore, the montmorillonite mineral content is very low in the bentonite with 1.53% mass.

3.3. X-Ray Diffraction (XRD) of treated bentonite

The result of X-ray diffraction characterization from natural bentonite which has been treated with ultrasonication and sedimentation process (Fraction 4) can be seen from Figure 2. The peak identification results show that this bentonite contains montmorillonite minerals. Besides montmorillonite, this bentonite also contains anorthite and clinoptilolite minerals.

Based on the XRD analysis result, the biggest fraction discovered from the bentonite is anorthite with mass percentage of 91.18%, and the lattice parameter of $a = 8.186 \, \text{Å}$, $b = 12.863 \, \text{Å}$ and $c = 14.209 \, \text{Å}$, $\alpha = 93.40^\circ$, $\beta = 116.25^\circ$, $\gamma = 90.20^\circ$ with triclinic crystal form.

Clinoptilolite fraction possesses a mass percentage of 3.60% with lattice parameter of $a = 16.91 \, \text{Å}$, $b = 16.54 \, \text{Å}$ dan $c = 7.34 \, \text{Å}$, $\alpha = \gamma = 90^\circ$ dan $\beta = 117.3^\circ$ as a monoclinic crystal form. The last fraction is montmorillonite with mass percentage of 5.22% and lattice parameter as $a = 5.68 \, \text{Å}$, $b = 8.88 \, \text{Å}$ and $c = 9.85 \, \text{Å}$, $\alpha = \gamma = 90^\circ$ and $\beta = 88.7^\circ$.

The montmorillonite minerals content in the bentonite sample which initially had a very small mass fraction of 1.58% could be increased by a mass percentage to 5.22%, the percentage of montmorillonite mass could be increased by the existence of ultrasonic waves and sedimentation. The absence of quartz compounds in XRD results is caused after ultrasonication and sedimentation of quartz compounds were spread over fractions 1, 2 and 3 because they have a large density of species that easily settles during sedimentation [4].

3.4. Particle Size Analyzer (PSA) analysis of montmorillonite nanoparticle

3.4.1. Milling process for 20 hours. Based on the characterization results by using particle size analyzer to montmorillonite nanoparticle which milled for 20 hours resulted the distribution number with average of 388 nm is shown in Figure 3.
Figure 3. The graph of distribution number of montmorillonite that milled for 20 hours

The data shown by the graph illustrates that the montmorillonite which was formed over 20 hours produced montmorillonite nanoparticles with homogeneous size. Where as many as 80% of the sample has a size of 312-414 nm with a standard deviation of ± 53.7 nm. This suggests that homogeneous nanoparticles are produced.

3.4.2. Milling process for 30 hours. From characterization results by using particle size analyzer to montmorillonite nanoparticle milled for 30 hours, the distribution number with an average of 186 nm is obtained as shown in Figure 4.

Figure 4. The graph of distribution number of monmorillonite that milled for 30 hours

The graph illustrates that montmorillonite milled for 30 hours yields montmorillonite nanoparticles with almost homogeneous size. A total of 70% of samples had a size of 142-189 nm with a standard deviation of ± 56 nm. In addition, the milling time also influences the yield of the produced nanoparticles, with a 20 hour milling produced an average of 388 nm montmorillonite nanoparticle whereas with a 30 hour milling resulted the average particle size of montmorillonite with 186 nm.

The longer grinding time will produce smaller nanoparticles, but if the grinding process is too long, it will cause the agglomeration that will produce a large nanoparticles [6].
3.5. Fourier Transform Infrared (FT-IR) analysis of bentonite

In the 3700-3600 nm band, there is a center of the OH group and in 1100-600 nm there is a Si-O-Si group confirming that the sample is bentonite containing montmorillonite and other minerals [7]. Sample of bentonite on the surface allows the presence of minerals composed of feldspar, calcite, quartz and clay minerals. Where feldspar in the rock is identified as anorthite (Na-Ca feldspar). Some rocks have been turned into clay minerals.

Based on the analysis of FTIR results shown Figure 5, it is revealed that bentonite sample contained montmorillonite, although the levels can not be determined because FTIR only shows the data qualitatively.

![FTIR spectra of bentonite](image)

The presence of Si-O-Si silicate functional groups is one of the characteristics that indicates the sample is a certain type of mineral. It can be a mineral feldspar or clay rock. The presence of OH group indicates that the sample actually belongs to a bentonite mineral with a mixture of crystoballite, feldspar, calcite, gypsum, caolinite, plagioclase and illit.

4. Conclusion

The montmorillonite isolation technique of bentonite by ultrasonication and sedimentation can increase montmorillonite levels in bentonite samples in the higher most regions. The production of nanoparticles were obtained by using planetary ball mill. The milling time varied at 20 and 30 hours each yielded montmorillonite with particle size of 388 nm and 186 nm respectively. Natural montmorillonite content of bentonite from Bener Meriah, Aceh, Indonesia was firstly obtained to be 1.52%. But it increased to be 5.22% after the ultrasonication and sedimentation process.

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