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3

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Preparation and Characterization of Natural Bentonite in to Nanoparticles by Co-precipitation Method

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Abstract. The nanoparticle based on natural bentonite from Pahae village had been prepared using co-precipitation method. Bentonite was dried in the oven at 100°C during a week. Bentonite is crushed using a mortar and milled by planetary ball mill to obtain the powder form. Further, the bentonite powder is activated with chemical reaction by dissolves the 50 g bentonite to 100 ml of HCl at 10 M. The magnetic stirrer was employed to mix the solution at 300 rpm and temperature 70°C. After that, the bentonite solution is washed using distilled water until the pH is neutral. The bentonite powder is calcined at temperature of 600°C for 1 hour with fix increment 150°C. Finally, the powder is given High Energy Milling (HEM) treatment for 30 minutes to obtain the particle size. The X-ray Diffractometer (XRD) and Scanning Electron Microscope (SEM) were used to characterize. From the characterization results it is reported that the average of bentonite nanoparticle size is 35.26 nm and the chemical constituents of natural bentonite Pahae are Al, Si, Ca, Fe and Ti.

INTRODUCTION

Indonesia is one of country in the world which recorded a huge potential in mining clay. Reviews of clay, clay minerals and their application in industry are widely reported in national and international publications. Clay is a clay mineral in accordance with the nomenclature given by the *Association Internationale Pour l'etude des Argiles* (AIPEA) in 2006. Clay minerals include some minerals such as bentonite and kaolinite have different molecular structures and are used in different purposes. These materials can be found in the nature from different sources and can also be synthesized in the laboratory [1]. Mineral clay is one part of a class of silicate minerals; phyllosilicates. The main component of clay is a clay mineral cilia layer of alumina with a sheet of tetrahedral and octahedral [2]. One of example of clay is bentonit. The content of the bentonite is montmorillonite minerals so that can absorb and expand in water, intercalation and cation exchanger [3-5]. They are a few types of bentonite and their names depend on the dominant elements, such as K, Na, Ca, and Al. With characteristic of this bentonite, bentonite interesting to use a catalyst [6] and adsorbents in the form of nanoclay [7,8].

Water treatment technology developed lately by technology adsorbs used to reduce levels of metal ions, oil, solvents and in water. This technology can make the water more clean and free of heavy metal ions. According to [9], that bentonite can be used to reduce the ion parameter Fe^{3+} and Cu^{2+} coagulation method, and combined with Upflow Anaerobic Filter methods (UAF) and Sand-Slow Filter (SSF) can reduce color concentration PtCo by using a pump aeration and filter from sand. According [10] saying that the bentonite has a very good ability in the process adsorbs, so bentonite can be used to absorb organic material and Fe contained in peat water.

Nanoparticles have a size of about (1-100) nm [11]. The material properties can be changed by controlling the size of the material, setting the chemical composition, surface modification and control the interaction between the particles. In the manufacture of nanoparticle made of various methods, one of which is the method of coprecipitation [12], solvothermal [13]. Coprecipitation method using acid-base reactions or chemical activation. Acid-base reaction

aims to reduce the particle size, eliminates impurity that accompany natural bentonite and bentonite content increases performance. Acid-base reactions carried out with stirring at room temperature or reflux. The activation process can also be carried out physically by way of heating [14]. In this study, conducted synthesis of bentonite from Janji Natogu village, Pahae district North Tapanuli, North Sumatra Province with coprecipitation method.

EXPERIMENTAL METHOD

Bentonite originating from Janji Natogu village, Pahae district North Tapanuli, North Sumatra Province is used as the main raw material in the manufacture of nanoparticles of bentonite. First step, natural bentonite dried in the oven with a temperature of 100°C for 7 days with the aim to eliminate moisture, bentonite ground with mortar to break chunks of bentonite. Bentonite which has been crushed then milling by planetary ball mill (PBM) at ratio of 1 : 5 for 24 hours (15 rpm) [15]. Bentonite powder sieved with a 200 mesh and diluted with HCl (Merck 32%, 10M) at a temperature of 70°C (90 minutes) and stirred with a speed of 300 rpm. Furthermore, the solution was washed using distilled repeatedly - again to obtain a normal pH, dried at 70°C for 5 hours. The process is then performed calcination process at a temperature of 600°C which aims to eliminate Cl bond on bentonite, and the results in the form of nanoparticles of bentonite. Nanoparticles bentonite milling by the High Energy Milling (HEM), which performed for 30 minutes. Characterization was conducted on the X-ray Diffraction (XRD). XRD measurements were performed using a Rigaku Smartlab. The particle sizes were estimated using the scherrer formula, morphological properties was determined with SEM type Hitachi SU3500 and the chemical composition of the raw material was determined with EDX type Horiba X-max silicon drift X-ray detector.

RESULTS AND DISCUSSION

XRD analysis of nanoparticles bentonite is shown in Figure 1. From these figure nanoparticles bentonite have several phases of low quartz and anortithe. In this diffraction peak has a index miller as follows (120), (20-2), (121), (101), (2-20), (110), (200), (220), (211), (112) and (212). The results obtained from the peak in accordance with previous studies [16]. It shows that both samples have phase of low quartz with trigonal structures and anortithe with triklin structures. Beside that we also can know the average particles size of material and we can using scherrer equation.

$$d = \frac{k\lambda}{B \cos\theta} \quad (1)$$

Where d is particles size, k is a Scherrer constant, λ is wavelength of the X-ray and B is full width at half maximum (FWHM).

The particles size calculated using Scherrer equation were 35.26 nm for nanoparticles bentonite. Based on these results, it is clearly that the nanoparticles had been succesfully synthesized from natural bentonite using this technique.

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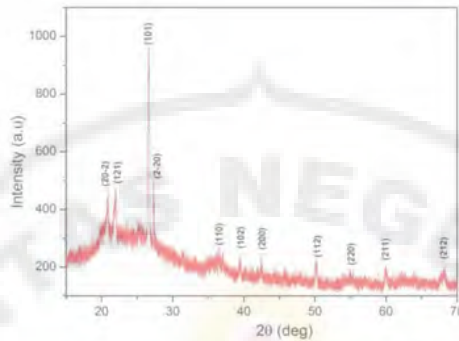


FIGURE 1. XRD pattern of Nanoparticles Bentonite

Figure 2 shows morphological properties of nanoparticles bentonite. Nanoparticle bentonite samples characterized by SEM to determine the homogeneity of the grain sample as shown in Figure 2. The nanobentonite tend to form rough spherical in shape. Figure 2 shows that bentonite is formed uneven in size and is still occur clotting (agglomeration), in addition, less focus when shooting forming uneven. This can be caused by oxygen before being characterized to bind to the particles forming the compound of bentonite so that agglomeration of the bentonite.



FIGURE 2. SEM results for Bentonite with a magnification of 5.000 times

Chemical composition of nanoparticles bentonite samples were analyzed by Energy Dispersive X-ray analyzer (EDX). In this test, carried out the shoutings at a point. EDX characterization result shown in figure 3.

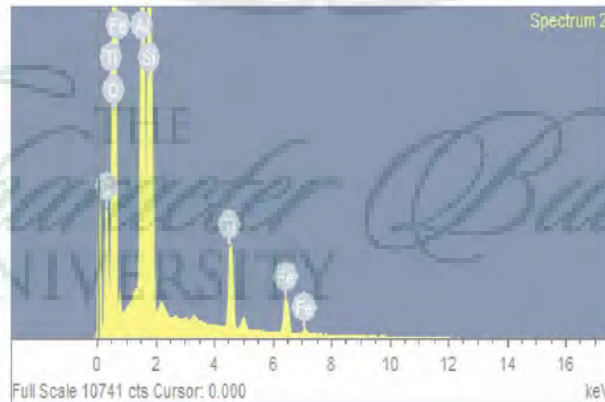


FIGURE 3. Spectrum EDX Nanobentonite

From the results of EDX, has elements of Al , Si , Ca , Fe and Ti in every spectrum. Bentonite is $(Mg,Ca)_xAl_2O_3.ySiO_2.nH_2O$, seen from the results of EDX in Figure 3 accordance with the chemical formula of bentonite which has a compound $CaCO_3$, SiO_2 , and Al_2O_3 [17], and the value composition is 13.73% ; 69.81% and 14.27 % whereas the elements Fe and Ti is impurities caused by several factors: the purification of bentonite is less clean, less clean oven circumstances and other factors. Based on EDX analysis, component content identified on the surface can be seen in Table 1.

TABLE 1. Element Results of EDX

Elements	Composition (% wt)
$CaCO_3$	13.73
SiO_2	69.81
Al_2O_3	14.27
Ti	0.87
Fe	1.322
Total	100

Based on EDX analysis, which identified component content contained in the element obtained nanobentonit is $CaCO_3$, SiO_2 , Al_2O_3 , Ti, and Fe. According to [18] bentonite is divided into several types Na - bentonite and Ca - bentonite. The second difference of this type is the domain elements Na and Ca on every kind. If the sodium content is more dominant then the bentonite is Na - bentonite, and vice versa. Based on the results of EDX obtained Ca content of 13.73 % and Na content of 0%, so that it can be concluded that the nanobentonit including Ca - Bentonite.

CONCLUSION

Preparation nanoparticles of bentonite has been created by the coprecipitation method based raw materials the natural bentonite from Janji Natogu village, district Pahae on North Tapanuli, North Sumatra Province. From the results of XRD analysis showed there were some phases that are characteristic of bentonite is quartz with trigonal crystal structure and has a mean particle size is 35.26 nm. State morphology of bentonite nanoparticles were characterized using SEM. Based on these images, agglomeration accours on bentonite, is due to the interaction between bentonite with oxygen, the particles showed a round shape. Indications chemistry of nanoparticles of bentonite samples were analyzed using Energy Dispersive X- ray analyzer (EDX). In this test was obtained $CaCO_3$, SiO_2 , Al_2O_3 , Ti, and Fe as the content of bentonite. The impurity of bentonite is Ti and Fe. and bentonite classified into Ca - bentonite.

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