

# NS-9

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**Submission date:** 08-May-2021 10:26AM (UTC+0700)

**Submission ID:** 1581095574

**File name:** FULLTEXT\_1.pdf (641.84K)

**Word count:** 2039

**Character count:** 10176



## Synthesis of Nanobentonite as Heavy Metal Adsorbent with Various Solvents

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<http://dx.doi.org/10.13005/ojc/3404020>

(Received: July 27, 2018; Accepted: August 02, 2018)

### ABSTRACT

The nanobentonite has been synthesized from natural bentonite taken from Tapanuli Utara, Indonesia using coprecipitation method with various solvents (HCl, H<sub>2</sub>SO<sub>4</sub>, and HNO<sub>3</sub>). Its properties as a metal adsorbent were investigated by Atomic Adsorption Spectrophotometry. X-Ray Diffraction analysis revealed that the bentonite produced is in nanometer scale. The characterization results obtained from the SiO<sub>2</sub> single phase with highest d<sub>hkl</sub> was at miller index (101) with 2θ of 21.9°, 22.0°, 22.07° respectively. The results of Microscope-Scanning Electron Energy analysis of nanobentonite dispersion indicated a reduction in agglomeration and finer nanobentonite surface. The Surface Area Analyzer results showed the S<sub>BET</sub> nanobentonite for solvent variation of HCl, H<sub>2</sub>SO<sub>4</sub>, and HNO<sub>3</sub> respectively were 731.76 m<sup>2</sup>/g, 868.11 m<sup>2</sup>/g, 493.97 m<sup>2</sup>/g. Lastly, Atomic Adsorption Spectrophotometric test showed that the optimal absorption of the metal content possessed by variety of HCl and nanobentonites with adsorption power of 91.16% for Pb, 76.39% for Cu, and 82.74% Co.

**Keywords:** Nanobentonite, Coprecipitation, Various Solvent, Metal Adsorbent.

### INTRODUCTION

Pollution of the aquatic environment by heavy metals becomes a serious problem in public health and a very important environmental issue.<sup>1</sup> Lead is toxic to public health. Lead can cause brain damage, inhibition of growth of children, increased blood pressure and digestion, kidney damage, nerve damage and joint disorder.<sup>2</sup> Similarly to copper

metal, it can accumulate in body tissues that can be toxic to humans. The poison generated can result vomiting, burning sensation in the esophagus and stomach, colic, diarrhea, which is then followed by hypotension, liver necrosis and coma.<sup>3</sup> Another heavy metal which widely found in aquatic environment and can cause a harmful effect is cobalt. It is a toxic metal that can cause vomiting and nausea, vision problems, heart problems, thyroid damage.<sup>4</sup>



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Recently, various researches that utilize minerals have been addressed to tackle this problem by optimizing the adsorption properties to bind the heavy metals and other contaminants and prevent them from polluting the environment.<sup>5-8</sup> Accordingly, one of the mineral that has been used to be an effective heavy metal adsorbent is bentonite. A study reported that nanobentonite can be used as a heavy metal ion adsorption where 90% of the cobalt is absorbed by the nanobentonites with concentration of 25 mg/L and 0.5g/50 mL.<sup>9</sup> Zuzana (2012) made the adsorbent of heavy metal ions Pb with nanobentonite where in the activated bentonite, the adsorption capacity for metal ions Pb<sup>2+</sup> and Cu<sup>2+</sup> ions are 32.68 mg/g and 11.34 mg/g respectively.<sup>10</sup> Converting natural bentonite into a nano-scale bentonite can be conducted by using coprecipitation method.<sup>11</sup> This method is proven to be effective to result a bigger area of nanobentonite produced. Pawar (2015) reported, with a surface area method of coprecipitation, produced an area of 294 m<sup>2</sup>/g. From these study, it was found that coprecipitation method is suitable to produce a large surface area that can improve the adsorption capacity of nanobentonite.<sup>12</sup>

In this particular study, the use of nanobentonite as a heavy metal adsorbent will be varied with various solvents, namely HCl, H<sub>2</sub>SO<sub>4</sub>, and HNO<sub>3</sub>. This is considered to enhance the optimum adsorption capacity, a large surface area, and the size of the smallest nanobentonite. In terms of the adsorption of heavy metal ions, the metal that will be examined is Pb, Cu and Co. Lastly, preparation of nanobentonite from natural bentonite was conducted to improve the quality of nanobentonite as adsorbent that can trap heavy metal ions.

#### MATERIALS AND METHODS

Natural bentonite used in the study derived from Pahae, North Tapanuli, North Sumatra, Indonesia. Nanobentonite from natural bentonite was synthesized with the coprecipitation method using 200 mL of 12 M HCl as a solvent, stirred for 120 min. at 300 rpm and at a temperature of 70°C using the magnetic stirrer. As many as 150 mL NH<sub>4</sub>OH

was stirred with a magnetic stirrer for 120 min. at 300 rpm, 70°C. Then, HCl solution was slowly poured into a solution of NH<sub>4</sub>OH and stirred at 350 rpm, 70°C for 120 minutes. Distilled water was added to neutralize the solution pH. The solution was dried in the oven for 5 h at a temperature of 100°C. The dried precipitate was crushed by using mortar to produce nanobentonite. The similar steps were repeated with the H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub>. Nanobentonite was characterized by Scanning Electron Energy Microscope-Dispersion, X-Ray Diffraction and Surface Area Analyzer. Analysis of nanobentonite adsorption capacity on heavy metal ions was done by preparing waste water with a metal content of Pb 92.50 ppm. Nanobentonite was poured into the waste water (250 ml), then stirred with a magnetic stirrer for 1 hour. The analyzed sample was a solution on four bottles namely A (indicator), B (Addition of HCL solvent nanobentonite), C (addition of H<sub>2</sub>SO<sub>4</sub> nanobentonite solvent), and D (addition of HNO<sub>3</sub> solvent nanobentonite).

#### RESULTS

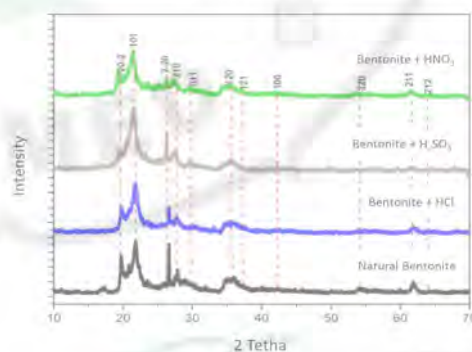


Fig. 1. The results of X-ray diffraction pattern: (a) bentonite milling, (b) bentonite with HCl, (c) bentonite with H<sub>2</sub>SO<sub>4</sub>, and (d) bentonite with HNO<sub>3</sub>.

Table 1: Estimated measurements crystalline diameter XRD test

Sample	Crystalline Size (nm)
Bentonite powder milling	80.00
Nanobentonit with HCl	16.46
Nanobentonit with H <sub>2</sub> SO <sub>4</sub>	13.60
Nanobentonit with HNO <sub>3</sub>	23.72

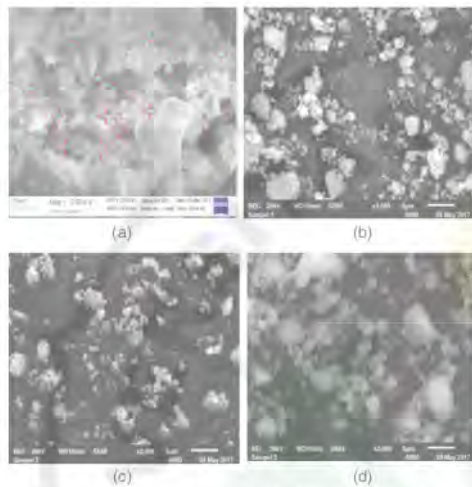


Fig. 2. The results of SEM with 3000 times magnification of: (a) bentonite milling, (b) nanobentonite with HCl, (c) nanobentonite with  $H_2SO_4$ , (d) nanobentonite with  $HNO_3$

Table 2: The particle size of bentonite with various solvents

Sample	Particle Size (nm)
Bentonite powder milling	121.55
Nanobentonite with HCl	42.95
Nanobentonit with $H_2SO_4$	33.00
Nanobentonit with $HNO_3$	41.60

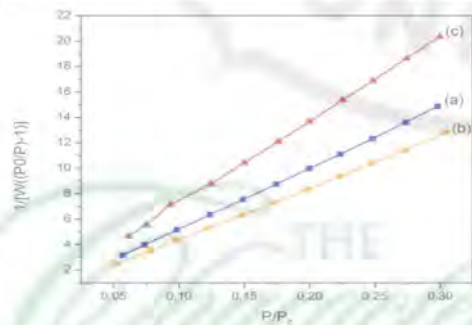


Fig. 3. BET plot of nanobentonite varied with: (a) HCl, (b)  $H_2SO_4$ , and (c)  $HNO_3$

Table 3:  $S_{BET}$  value for each nanobentonite

Sample	$S_{BET}$ ( $m^2/g$ )
Nanobentonite with HCl	731.76
Nanobentonit with $H_2SO_4$	868.11
Nanobentonit with $HNO_3$	493.97

Table 4: The metal content of each nanobentonite Sample

	Pb (ppm)	Cu (ppm)	Co (ppm)
Indicator	92.50	54.2	64.3
Nanobentonite with HCl	8.18	12.80	11.10
Nanobentonit with $H_2SO_4$	60.5	16.4	18.3
Nanobentonit with $HNO_3$	10.8	13.7	11.00

## DISCUSSION

Based on Fig. 1, the main peak of the index (101) with a second angle  $2\theta = 22.04^\circ$  with  $SiO_2$  phase. It is also strengthened by the emergence of several specific dhkl peaks with miller indices of (20-2), (2-20), (210), (011) and (120), (100), (200), (212). Best spectrum of the XRD analysis results is showed by nanobentonite with  $H_2SO_4$  solvent which has a higher peak in the phase of  $SiO_2$  and trigonal crystalline structure and diameter of 13.60 nm. This result is better than Sirait's (2017) study performing nanobentonite synthesis having a crystalline size of 35.26 nm.<sup>11</sup>

Figure 2 shows the morphology of nanobentonite that tends to have a rough spherical shape. The particle size was analyzed by the Scherer equation. From Table 2, bentonite particles known to use  $H_2SO_4$  has the smallest particle size. The size of the smaller particle diameter caused by the reaction of acid (HCl,  $H_2SO_4$ , and  $HNO_3$ ), because in addition to being cleaner for impurities on bentonite particles, it is also destructive to particles so as to make the particle size smaller.

Based on the data obtained by BET analysis, the largest surface area is with  $H_2SO_4$  as solvent at  $84.84 m^2/g$  and a surface area of a specific surface area ( $S_{BET}$ ) is  $868.10 m^2/g$ . Pawar (2015) stated that the manufactured of nano bentonite with  $H_2SO_4$  as solvent obtained the specific surface area of  $294 m^2/g$ . It can be concluded that the research results are better than previous Pawar study (2015) because of the larger specific surface area.

The residue of waste water were analyzed by AAS to get the value of adsorption, which is shown in Table 4. Moreover, it can be seen in Table 3 that the absorption of Pb is more optimal using the HCl and nanobentonite variation (91.16%) compared

with the use of nanobentonite obtained using  $H_2SO_4$  (34.60%) and  $HNO_3$  (88.33%). Cu optimal absorption was obtained using the HCl nanobentonite (76.39%) compared with the use of  $H_2SO_4$  (69.74%) and  $HNO_3$  (74.72%) nanobentonite. Cobalt absorption was shown by the HCl nanobentonite (82.74%) compared to metal absorption using  $H_2SO_4$  (71.54%) but it is comparable with the  $HNO_3$  nanobentonite (82.90%).

### CONCLUSION

Nanobentonite particles have been synthesized from natural bentonite from Pahae, South Tapanuli District, North Sumatra, Indonesia, by coprecipitation method. Variations of solvent in coprecipitation method was conducted to obtain three different samples, ie variation of HCl,  $H_2SO_4$  and  $HNO_3$ . The XRD results showed that

all three samples had a single phase, the  $SiO_2$ . Nanobentonite with solvent  $H_2SO_4$  has a smaller particle size with smaller surface area compare to nanobentonite with HCl solvent. This is caused by the silica content which have absorption properties at the nanobentonite solvent  $H_2SO_4$  smaller but the most optimal absorption occurs in nano bentonite with HCl solvents that absorb Pb 91.16%, which has a surface area of 713.76  $m^2/g$ . SEM analysis is suggested for further and more accurate particle size determination. It is also suggested to perform the analysis of adsorbent capacity of this nanobentonite with other heavy metals.

### ACKNOWLEDGEMENT

Authors would like to thank to Ministry of Research and Higher Education of Indonesia for research funding support via DRPM 2018 funding scheme.

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