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Reactivity improvement of $\text{Ca}(\text{OH})_2$ sorbent using diatomaceous earth (DE) from Aceh Province

M Mariana^{1,2}, M Mahidin^{1,2}, F Mulana^{1,4}, T Agam³ and F Hafdiansyah³

¹ Department of Chemical Engineering, Syiah Kuala University, Banda Aceh – Darussalam 23111, Indonesia

² Environmental and Natural Resource Research Center, Syiah Kuala University, Banda Aceh – Darussalam 23111, Indonesia

³ Undergraduate Program of Chemical Engineering, Syiah Kuala University, Banda Aceh – Darussalam 23111, Indonesia

⁴ Faculty of Chemical Engineering, University Teknologi MARA (UiTM), Shah Alam 40450, Malaysia

E-mail: mariana_hasyim@yahoo.com

Abstract. In this study, the diatomaceous earth (DE) from Aceh Province was used to increase the reactivity of $\text{Ca}(\text{OH})_2$ sorbent. The high silica (SiO_2) content of about 97% in the diatomaceous earth allows the increasing reactivity of $\text{Ca}(\text{OH})_2$ sorbent by forming calcium silicate hydrate (CSH). The CSH improved the porosity characteristic of the sorbent. The improvement process was performed by mixing $\text{Ca}(\text{OH})_2$ sorbent, diatomaceous earth and water in a beaker glass at the $\text{Ca}(\text{OH})_2$ /DE weight ratio of 1:10 for 2 h and then dried at 120 °C for 24 h. The dried sorbent was calcined at 500 °C and 800 °C for 2 h. The activated sorbent was characterized using Scanning Electron Microscopy (SEM) for the morphological properties; X-Ray Diffraction (XRD) for the materials characteristics. The adsorption capacity of the sorbent was tested by methylene blue adsorption. The results showed that the $\text{Ca}(\text{OH})_2$ /DE sorbent had a higher porosity than the $\text{Ca}(\text{OH})_2$ adsorbent. The results also showed that $\text{Ca}(\text{OH})_2$ /DE which was calcined at higher temperature of 800 °C had a higher adsorption capacity compared to $\text{Ca}(\text{OH})_2$ /DE which was calcined at lower temperature of 500 °C.

1. Introduction

There are various SO_2 removal process technologies in the gas purification technology and the most commonly used process is dry process and wet process (Iswandi, 1996). The use of dry process has several advantages compared to wet process, among others such as cheap, easy handling and non producing liquid waste. One of the dry method that has an effective, simple, handy and inexpensive SO_2 separation processes is to use bag filter reactor by using $\text{Ca}(\text{OH})_2$ sorbent. However, this process also has several weaknesses among others such as the low conversion of sorbent and the low efficiency of gas separation [1].

To improve the efficiency of gas separation and increase the reactivity of sorbents (conversion of calcium), various additives can be added to the sorbent. Sorbents obtained by mixing calcium hydroxide with a silica-containing substance can increase the conversion of calcium compared to the calcium conversion obtained without additive mixture [2]. Diatomaceous earth (DE) is a pozzolonic material containing SiO_2 , Al_2O_3 and CaO [3].



2. Method

Experiment was conducted as follows: (1) Equipment of Material; and (2) Sorbent Preparation.

2.1. Equipment and material

Equipment used in this study were mixer, water bath, hot plate, magnetic stirrer, oven, erlenmeyer, balance, ball mill, sieve shaker, thermometer, SEM and XRD. Further, materials used in this study were Ca(OH)_2 , diatomaceous earth (DE), methylene blue and aquadest.

2.2. Sorbent preparation

Sorbent was prepared by mixing Ca(OH)_2 and DE together with 100 ml of water in a beaker glass with ratio of $\text{Ca(OH)}_2/\text{DE}$ was 1:10. The pH and density values of the sorbent were previously also analysed. The beaker was then closed and inserted into the water bath, then stirred at various temperatures for 2 hours. After stirring, the pH value and the density of slurry was measured again and then dried at temperature of 120 °C for 24 h. The dried slurry was analysed the SiO_2 content that bound in Ca(OH)_2 sorbent. The solids were then calcined at 500 °C and 800°C for 2 hours. The characterization of the samples was performed using SEM and XRD.

3. Result and discussion

3.1. Chemical content

To see the component of $\text{CaO.SiO}_2.2\text{H}_2\text{O}$ was formed or not as a result of the reaction, it can be seen from Analysis of X-ray Diffraction Patterns. XRD analysis was conducted at Indonesian Institute of Sciences, Center for Physical Research of PUSPIPTK Serpong Complex. Figure 1 to Figure 3 show the results of XRD analysis for $\text{Ca(OH)}_2/\text{DE} = 10: 1$ sorbents with particle sizes of 200 mesh in various reaction temperature. XRD analysis showed that the reaction between Ca(OH)_2 and SiO_2 produced CSH ($\text{CaO.SiO}_2.2\text{H}_2\text{O}$). This is in accordance with the results of research that has been done by Mariana, et al [4,5].

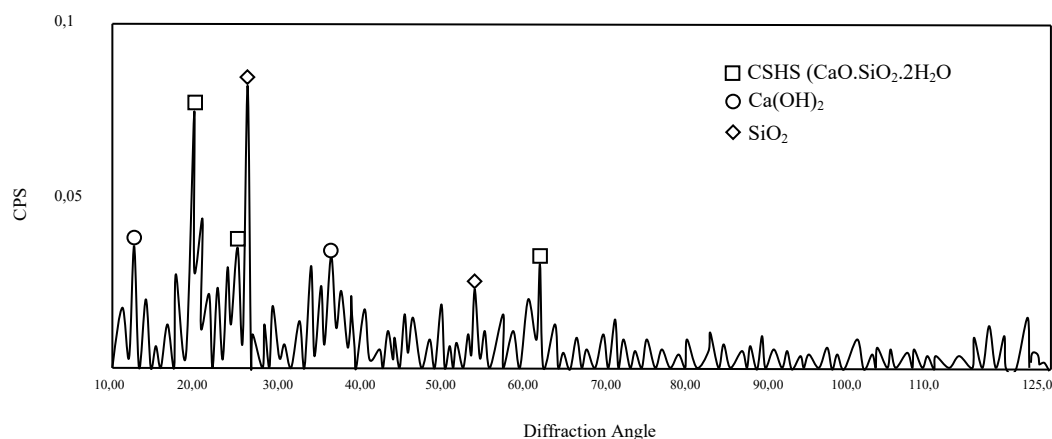


Figure 1. XRD profile of $\text{Ca(OH)}_2/\text{DE} = 10: 1$ sorbent with a particle size of 200 mesh at a reaction temperature of 65 °C

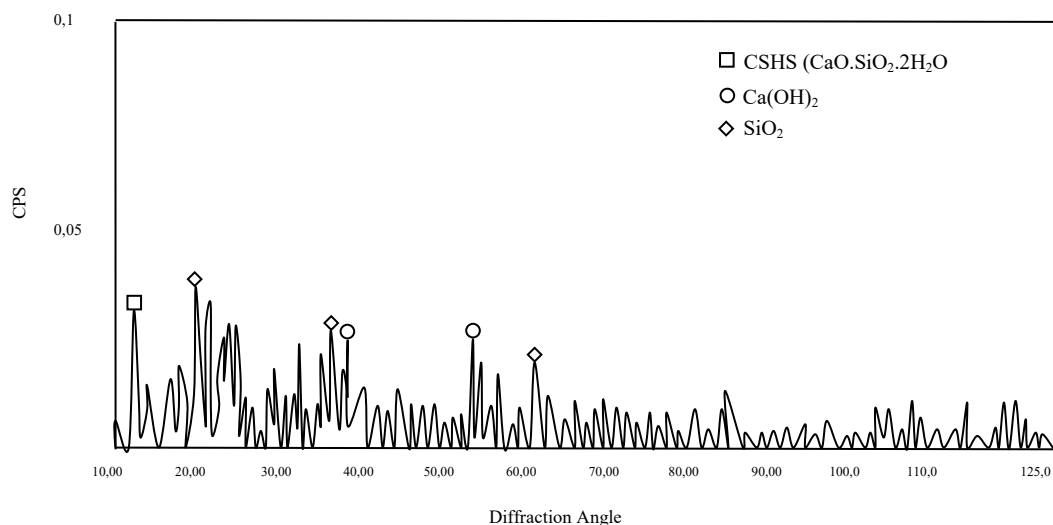


Figure 2. XRD profile of $\text{Ca(OH)}_2/\text{DE} = 10: 1$ sorbent with a particle size of 200 mesh at a reaction temperature of 70 °C

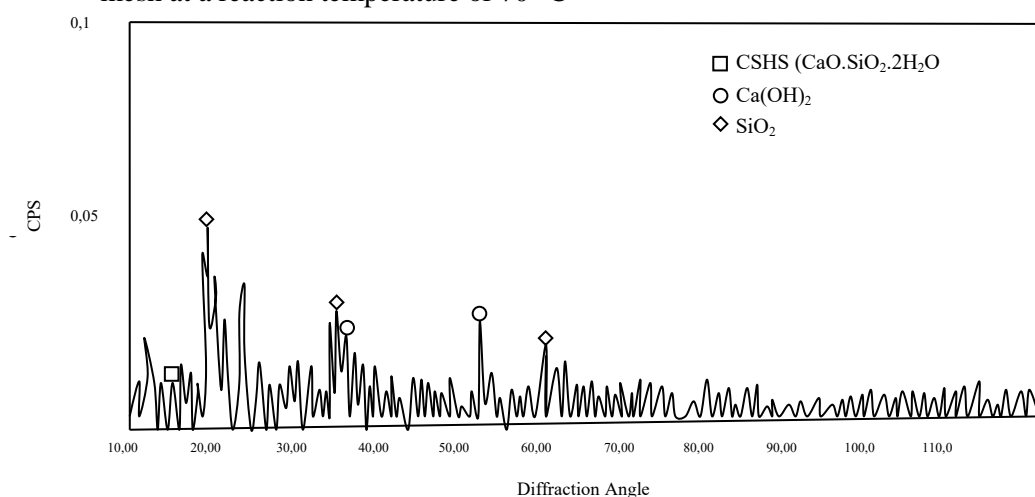


Figure 3. XRD profile of $\text{Ca(OH)}_2/\text{DE} = 10: 1$ sorbent with a particle size of 200 mesh at a reaction temperature of 75 °C

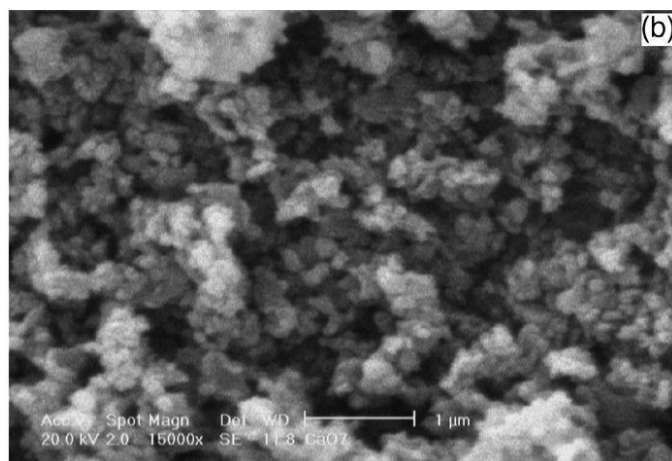


Figure 4. SEM profile of Ca(OH)_2 .

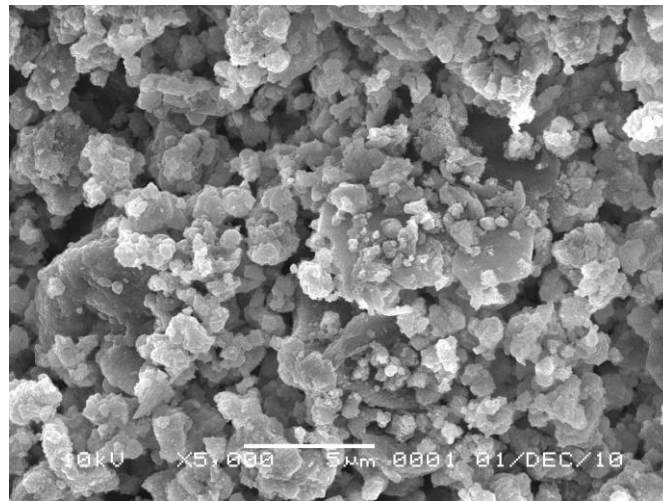


Figure 5. SEM profile of Ca(OH)₂/DE = 10: 1 sorbent before reaction on a particle size of 200 mesh.

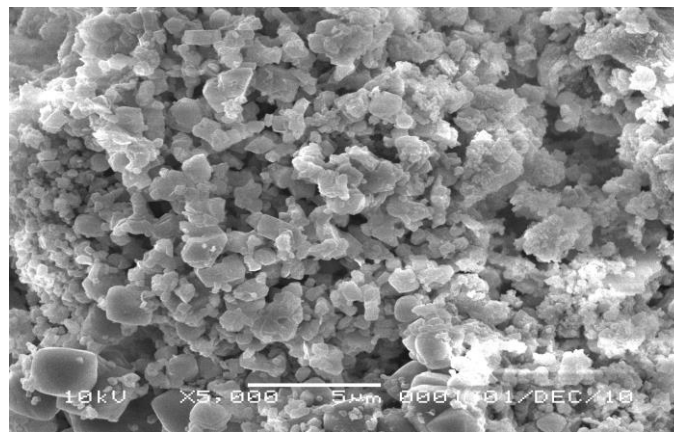


Figure 6. SEM profile of Ca(OH)₂/DE = 10: 1 sorbent after reaction on a particle size of 200 mesh

3.2 Morphology

Figure 4 to figure 6 showed some differences between sorbent of Ca(OH)₂:DE before and after the reaction where the apparent difference is that the pore of particle of sorbent after the reaction appears to be more open than the one before the reaction. As a result of this open pore would affect on adsorption capacity where adsorption capacity would be increased. The porosity of Ca(OH)₂ sorbent is smaller than the Ca(OH)₂:DE sorbent as shown in Figure 4.

3.3 Sorbent activity test

In this study, the sorbent activity was measured by conducting adsorption process of methylene blue on the sorbent. The results of adsorption test were shown in Table 1 and Table 2. Table 1 and Table 2 show that the adsorption of methylene blue on Ca(OH)₂/DE sorbent increases with increasing of adsorption time. Table 1 and table 2 also show that the calcination temperature of the sorbent affects the sorbent adsorption capacity.

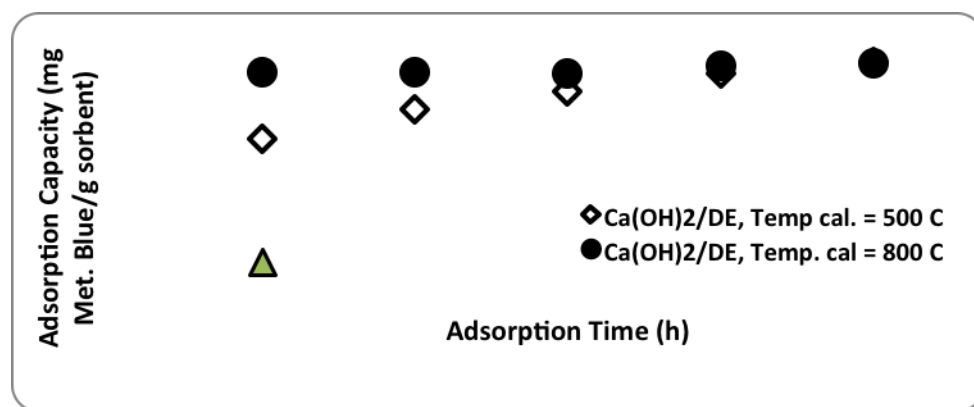
Table 1. Adsorption capacity of methylene blue solution on activated $\text{Ca}(\text{OH})_2/\text{DE}$ sorbent with particle size of 200 mesh and calcined at temperature of 500 °C

Adsorption time (hr)	Absorbance	Concentration of methylene blue after adsorption (ppm)	Adsorption capacity (mg Met. Blue/g DE)
1	0.219	1.522	7.454
3	0.135	1.072	7.467
5	0.0876	0.809	7.475
7	0.0424	0.563	7.483
9	0.0107	0.3908	7.488

Table 2. Adsorption capacity of methylene blue solution on activated $\text{Ca}(\text{OH})_2/\text{DE}$ sorbent with particle size of 200 mesh and calcined at temperature of 800 °C

Adsorption time (hr)	Absorbance	Concentration of methylene blue after adsorption (ppm)	Adsorption capacity (mg Met. Blue/g DE)
1	0.0395	0.547	7.4835
3	0.0380	0.539	7.4838
5	0.0441	0.573	7.4828
7	0.0203	0.443	7.4866
9	0.0168	0.424	7.4872

The sorbent adsorption capacity would increase with increasing calcination temperature. At the calcined temperature of 800 °C, the adsorption of methylene blue into sorbent reached a maximum value within a relatively short adsorption time of 1 hour. As for sorbent at calcination temperature of 500 °C, the adsorption time need longer time and reach a maximum adsorption capacity at 9 hours. The results also showed that the adsorption capacity of methylene blue on sorbent of $\text{Ca}(\text{OH})_2/\text{DE}$ was higher when compared with adsorption capacity by only using $\text{Ca}(\text{OH})_2$ sorbent. This is due to the formation of a porous calcium silicate hydrate (CSH) in the sorbent of $\text{Ca}(\text{OH})_2/\text{DE}$ as shown in Figure 7. This is in consistent with a study by Mariana (2003) which states that sorbent of $\text{Ca}(\text{OH})_2/\text{DE}$ has a larger porous than $\text{Ca}(\text{OH})_2$ sorbent alone as shown in Figure 8. The larger porous or larger surface area of the sorbent caused the higher of adsorption capability [6].

**Figure 7.** Effect of adsorption time on adsorption capacity of $\text{Ca}(\text{OH})_2/\text{DE}$ sorbent.

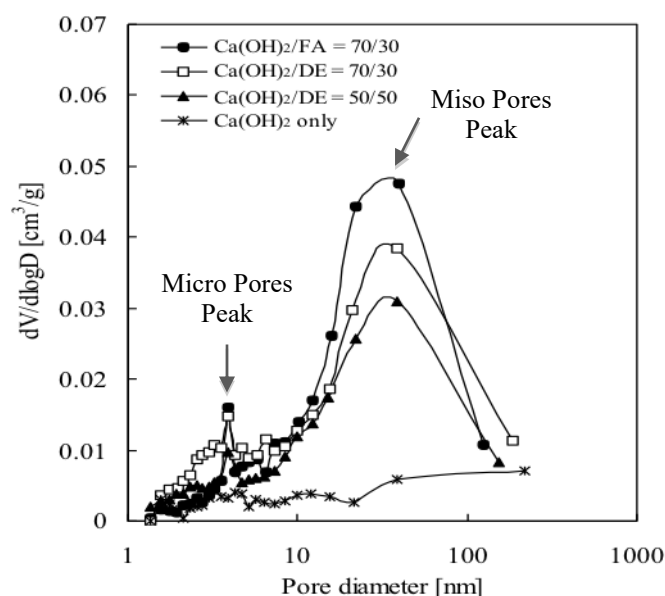


Figure 8. Effect of sorbent type on pore diameter [4].

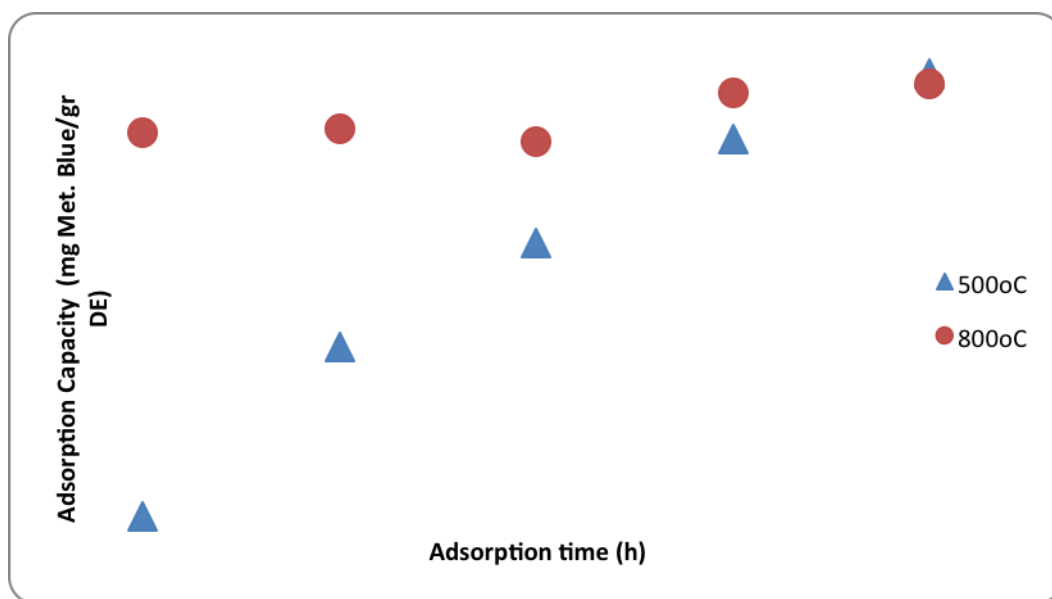


Figure 9. The effect of adsorption time toward the adsorption capacity of methylene blue solution on $\text{Ca(OH)}_2/\text{DE}$ with particle size of 200 mesh after calcination at temperature of 500 °C and 800 °C.

Figure 9 shows that the adsorption capacity of methylene blue solution on $\text{Ca(OH)}_2/\text{DE}$ sorbent both after calcination temperature of 500 °C and 800 °C has the same tendency, that is tend to increase with the increase of adsorption time. This shows that the adsorption capacity of of methylene blue solution on $\text{Ca(OH)}_2/\text{DE}$ increase at time of 1 to 9 hours, because the surface of $\text{Ca(OH)}_2/\text{DE}$ sorbent is still active to perform adsorption process. However, at the adsorption time of 5 hours of $\text{Ca(OH)}_2/\text{DE}$ sorbent that activated at calcination temperature of 800 °C, the adsorption capacity decreases, this is probably due to the possibility of the surface of $\text{Ca(OH)}_2/\text{DE}$ becomes less active, since desorption may also occur. At the adsorption time of 5 hour there is an adsorption equilibrium in

which the amount of adsorbed agent is almost proportional to the amount of the desorbed substance [7].

4. Conclusions

From the result of this research, it can be concluded that: the reaction temperature and reaction time affect the reaction of product formation and the amount of product formed. The presence of diatomaceous earth (DE) containing of much silica in Ca(OH)_2 sorbents may increase the reactivity of the sorbent. The adsorption capability of $\text{Ca(OH)}_2/\text{DE}$ sorbent toward methylene blue is greater than that of Ca(OH)_2 sorbent itself; and $\text{Ca(OH)}_2/\text{DE}$ which was activated at temperature of 800 °C has the highest adsorption capacity compared to $\text{Ca(OH)}_2/\text{DE}$ which is activated at temperature of 500 °C. This is due to the surface and pores of $\text{Ca(OH)}_2/\text{DE}$ sorbent which are activated at temperature of 800 °C was not covered by water and other impurities.

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