Bubble column application on purification of biogas and production of nano-calcium carbonate in continuous process

Yukh Ihsana, Putu Adhi Rama, Ratri Puspita, Sugeng Winardi*, Tantular Nurtono

Department of Chemical Engineering, Faculty of Industrial Engineering, Institut Teknologi Sepuluh Nopember (ITS), Kampus ITS Sukolilo, Surabaya 60111, Indonesia

* Corresponding author: swinardi@chem-eng.its.ac.id

Abstract

Purification of biogas by removing carbon dioxide content has been developed to increase its calorific value. The CO₂ contained in biogas was absorbed by contacting Ca(OH)₂ solution and CO₂ in the bubble column to produce high purity of biogas and generated precipitated calcium carbonate (PCC) simultaneously. Two sources of CO₂ were used in this work, which were CO₂ contained in biogas and pure CO₂. Pure CO₂ was used as a benchmark in this study to show the effect of the presence of methane contained in biogas on the absorption process. The investigation was done in a continuous process. The results showed that the highest absorption of CO₂ was obtained in biogas at 79.34 %. PCC with calcite phases was successfully formed in 225–270 nm. Rhombohedral calcite phase was formed by the use of pure CO₂ and biogas as a source of CO₂. The use of CO₂ contained in biogas and pure CO₂ in the absorption process did not have an effect on the crystalline phase and morphology of calcite formed. Several factors that significantly affected the absorption of CO₂ were gas flow rate, absorbent flow rate, CO₂ concentration in the gas inlet, and the initial liquid level in the bubble column.

Keywords: Biogas, absorption, precipitated calcium carbonate

INTRODUCTION

Biogas is one of the promising alternative energies to reduce dependency on fossil fuels. Typically, biogas consists of methane (CH₄, 50–70 %), carbon dioxide (CO₂, 30–40 %), hydrogen (H₂, 5–10 %), nitrogen (N₂, 1–2 %), water (H₂O, 0.3 %), and hydrogen sulfide (H₂S) (Mamun et al., 2015). The presence of a high concentration of CO₂ in biogas causes corrosion of the reactor due to the formation of carbonic acid. In addition, it can reduce the heating value of the biogas. Therefore, upgrading biogas into biomethane by CO₂ absorption is important to increase biogas quality and heating value. Water scrubbing is a proven, simple, and universal technology for absorption because it does not require additional CO₂ liquefaction costs.

Process of CO₂ absorption with Ca(OH)₂ solution followed by the formation of precipitated calcium carbonate (PCC) is an advantages alternative process. PCC is known as a product that has many uses in industry. Previous work has investigated the possibility of PCC production with high purity and narrow particle distribution size by reacting CO₂ and calcium extracted from blast furnace (slag). However, this process needs more pretreatment to extract calcium from slag and results in co-precipitation of magnesium carbonate more than PCC (De Crom et al., 2015). From this method, it is possible to produce PCC and purify biogas simultaneously by reacting carbon dioxide contained in biogas and Ca(OH)₂ solution. Even though this process is a classic method, it may be adopted and modified for application in a small scale of biogas production. There is rare literature available in small-scale of biogas upgrading systems. Some advantages of using this process include CaO as an inexpensive raw material, a simple reaction process, and precipitated calcium carbonate which is the result of the process can add a commercial value of the biogas purification process. In addition, regeneration and disposing of a residual solution are friendly to the environment if it is carried out in the long term. Although this process has limitations of raw materials derived from nature, a lot of research on CaO regeneration of CaCO₃ has developed by thermal dissociation process commonly referred to as Ca-Looping (Han et al., 2011).

In the previous work (Ihsana et al., 2017), the biogas absorption process in semi-batch using a bubble column has been successfully
used to purify biogas and synthesize nanoparticle precipitated calcium carbonate simultaneously. CaCO₃ has been successfully formed in calcite phase. Previously, the use of absorbent concentration is based on its solubility. Ca(OH)₂ solution is fed in the bubble column until it fully reacts. From this semi-batch process, it has been found that the use of a perforated plate with a hole diameter of 0.5 mm gives greater absorption compared to that with 1 mm diameter hole. This work was carried out in continuous absorption to determine the maximum performance of CO₂ absorption on different operating conditions. The effect of absorbent concentration, gas flow rate, solution level, and absorbent flow rate on CO₂ absorption were investigated. In addition, the optimum conditions for CO₂ capture were reported.

**EXPERIMENTAL**

**Materials**

Materials used in this work consisted of QUALI-TECH CHEM technical grade Ca(OH)₂ 99 % Pure, CO₂ 99.9 % (v/v), and aquadest. Biogas used in this research was from the installation of the reactor pilot plant in the rural area. Cow manure was used as a substrate to produce biogas. This biogas contained 67.8 % of methane (CH₄), 32.2 % of carbon dioxide (CO₂), and other components such as hydrogen sulfide (H₂S), and water (H₂O) after desulfurization using zeolite and dehydration using water trap. As shown in Fig. 1, the bubble column used in biogas purification was made from acrylic. It has an inside diameter of 7 cm with a thickness of 0.5 cm and a height of 100 cm. The biogas from the gas cylinder was supplied through a distributor consisting of a perforated plate and glass beads. This perforated plate has a hole diameter of 0.5 mm with an active area of 0.332 cm². This perforated plate was used to produce bubbles which were contacted with Ca(OH)₂ solution. At the bottom of the bubble column, there were glass beads that had an average diameter of 0.3 cm. Ca(OH)₂ solution was fed countercurrent into the bubble column. Before entering the bubble column, Ca(OH)₂ solution was stirred in a feed tank using a stirrer. Biogas flow was controlled by the flowmeter. The exit gas would be collected using the Tedlar bag for analyzing biogas content using the gas chromatograph.

**Synthesis of Precipitated Calcium Carbonate (PCC)**

The absorbent concentration used in this process was varied of 1.5, 2, and 2.5 times of Ca(OH)₂ solubility at 30 °C. This concentration was varied at 2.38, 3.068, and 3.82 gram/liter, respectively. Gas flow rates used were 0.5, 0.6, 0.7, 0.8, and 0.91 liter/min. The total volume of Ca(OH)₂ solution used in this study was 10 liters. The Ca(OH)₂ flow rate used was varied at 200, 250, 300, 350, and 400 mL/min. The initial liquid level was varied at 50, 65, and 80 cm to determine the performance of CO₂ absorption in a solution of Ca(OH)₂ at a certain height. Two conductometer probes were installed at a certain height to find out the reaction runs at a different liquid level. In this absorption process, two sources of CO₂ were used. They were pure CO₂ and biogas. Pure CO₂ was used to know the influence of other gases contained in biogas on the absorption process.

The solution of Ca(OH)₂ was prepared by dissolving 23.8 grams of Ca(OH)₂ in 1 liter of water. This solution was stirred using a magnetic stirrer at a constant speed of 450 rpm with the solution temperature of 60 °C. Ca(OH)₂ solution was fed into the feed tank with an addition of 9 liters of water and then was flowed by diaphragm pump into the bubble column to a certain height. The flow rates of inlet and outlet solutions were kept constant. At the same time, biogas was fed at the bottom bubble column. The determination of CO₂ absorbed was done by different treatment between pure CO₂ and biogas. In pure CO₂, the absorption was determined by changes in the flow rate of the outgoing gas while the biogas absorption was determined from the exit gas sampling which was analyzed by gas chromatography. Gas sampling was done when gas hold up was constant. The gas flow rate was stopped when the solution in the feed tank had run out completely. Then, the sample from the reactor was collected, filtered, and cooled for 12 hours. Calcium carbonate was dried at 60 °C for 12 hours before calculating its weight and performing its characterization. The content of CH₄ and CO₂ in biogas was analyzed using gas chromatography (Shimadzu GC-2010 Plus Kyoto, Japan) with Thermal conductivity detector (TCD). The type of CaCO₃ crystals, morphology phase, and purity of Ca were analyzed using X-ray powder diffraction (XRD), SEM (Scanning Electron Microscopy), and XRF (X-ray fluorescence), respectively. The average diameter of 300 CaCO₃ particles was calculated using image analysis software.

**RESULTS AND DISCUSSION**

**Effect of Gas Flow Rate on Absorption**

Effect of gas flow rate on absorption has been observed at concentration, Ca(OH)₂ flow rate, and the initial liquid level of 3.06 gram/liter, 200 mL/minute, and 50 cm, respectively. In general, Fig. 2 shows the reduction in absorption of CO₂ in biogas and pure CO₂ when the gas flow rate is increased. The greater gas flow rate, the faster the pH required to go to neutral. The pH changes in the carbonation solution have been studied in previous work and report that the greater gas flow rate used made the pH change to neutral faster. The neutral pH will decrease the performance of the absorbent to absorb CO₂. When pH value is more than 10, more PCC is converted. This is because carbonate ions are dominant at this high pH. Conversely, when the pH is low, precipitation will be decreased with the presence of H₂CO₃ in the absence of bicarbonate and carbonate ions. At neutral pH, bicarbonate ions are formed and it is difficult to form carbonates with divalent ions. Hence, it is difficult to form precipitation of CaCO₃ (Bang et al., 2011).

Compared to the absorption of biogas, absorption of pure CO₂ is not as significant as in biogas. Based on film theory, increasing the inlet concentration of CO₂ allows more CO₂ molecules to travel the bulk gas to the gas-liquid interface, resulting in higher absorption efficiency. However, gas with the hight content of CO₂ in pure CO₂ at the same flow rate, the absorbent mole ratio to carbon dioxide decreased, leading to a reduction in efficiency of absorption (Yincheng et al., 2011). This is also seen in Table 1. The use of pure CO₂ is not worthy if the ratio of Ca(OH)₂/CO₂ is smaller than 1. This could have a significant effect if the concentration of Ca(OH)₂ is large.

**Fig. 1 Schematic purification experiment apparatus in a continuous process**

1. Cylindrical biogas tank
2. Flowmeter
3. Distributor
4. Bubble Column
5. Feed Tank
6. Diaphragm Pump
7. PCC tank
8. Stirrer Unit
9. Gas flowmeter
10. Flowmeter of Ca(OH)₂
11. Conductivity meter
Based on Table 1, the highest CO₂ absorption is obtained from the lowest gas flow rate in both biogas and pure CO₂. However, the mole ratio of Ca(OH)₂/CO₂ in biogas is higher than pure CO₂. It causes higher CO₂ absorption in biogas. Based on stoichiometry, every 1 mole of Ca(OH)₂ requires 1 mole of CO₂ to react completely. At least, to obtain maximum absorption, Ca(OH)₂ must be greater than CO₂ entering with a mole ratio of Ca(OH)₂/CO₂ greater than 1 but it depends on the efficiency of the column design and operating condition. There are several studies that have found that to get CO₂ absorption of around 90%, the mole ratio of absorbent and CO₂ used should be equal to 4.43. It was based on research conducted using a spray column using NaOH as an absorbent and it was contacted with 15% CO₂ inlet content (Yincheng et al., 2011). Table 1 presents the mole ratio of Ca(OH)₂/CO₂ on CO₂ absorption.

### Table 1 Mole ratio of Ca(OH)₂/CO₂ on CO₂ absorption.

| Gas flow rate (liter/minute) | *Mole ratio* | *CO₂ absorbed* | Mole ratio Ca(OH)₂/CO₂ | CO₂ absorbed (%) |
|-----------------------------|-------------|----------------|------------------------|-----------------|
| 0.5                         | 0.37        | 36.37          | 1.15                   | 69.35           |
| 0.6                         | 0.31        | 36.66          | 0.96                   | 68.74           |
| 0.7                         | 0.26        | 35.72          | 0.82                   | 68.36           |
| 0.8                         | 0.23        | 35              | 0.72                   | 68.21           |
| 0.9                         | 0.21        | 32.54          | 0.64                   | 67.43           |

*Pure CO₂*

**Effect of Ca(OH)₂ Flow Rate on Absorption**

As seen in Table 2, the absorption of CO₂ and the mole ratio of Ca(OH)₂/CO₂ at 400 mL/min in pure CO₂ are 61.85% and 0.52, respectively. With the same concentration of Ca(OH)₂, the increased absorbent flow rate gives a greater mole ratio. The absorbent requirement will be greater if the gas inlet has lower CO₂ content. This is due to the CO₂ content of the inlet used that is equal to 15%. Increasing CO₂ absorption by increasing the absorbent flow rate is considered to be an efficient option to avoid using high absorbent concentrations (Tippayawong et al., 2010). This is be useful for reducing the regeneration load of the absorbents used and reducing the use of CaO as raw materials. The presence of CH₄ as an inert in biogas followed by increasing absorbent flow rate provide a large mole ratio of Ca(OH)₂/CO₂. In biogas, at a Ca(OH)₂ flow rate of 400 mL/minute, the mole ratio of Ca(OH)₂/CO₂ is 0.26 and 0.82 in pure CO₂ and biogas, respectively. Increasing CO₂ absorption by increasing the absorbent flow rate is considered to be an efficient option to avoid using high absorbent concentrations (Tippayawong et al., 2010). This is be useful for reducing the regeneration load of the absorbents used and reducing the use of CaO as raw materials. The presence of CH₄ as an inert in biogas followed by increasing absorbent flow rate provide a large mole ratio of Ca(OH)₂/CO₂. In biogas, at a Ca(OH)₂ flow rate of 400 mL/minute, the mole ratio of Ca(OH)₂/CO₂ is 0.26 and 0.82 in pure CO₂ and biogas, respectively. The absorbent flow rate gives a greater mole ratio. The absorbent requirement will be greater if the gas inlet has lower CO₂ content. This is due to the CO₂ content of the inlet used that is equal to 15%.

### Table 2 The mole ratio of Ca(OH)₂/CO₂ on pure CO₂ and biogas absorption in a continuous process.

| Absorbent flow rate (mL/minute) | *Mole ratio* | *CO₂ absorbed* | Mole ratio Ca(OH)₂/CO₂ | CO₂ absorbed (%) |
|---------------------------------|-------------|----------------|------------------------|-----------------|
| 200                             | 0.26        | 35.71          | 0.82                   | 61.85           |
| 250                             | 0.33        | 46.42          | 1.02                   | 79.34           |
| 300                             | 0.39        | 50             | 1.23                   | 82.13           |
| 350                             | 0.46        | 57.14          | 1.43                   | 74.64           |
| 400                             | 0.52        | 61.86          | 1.64                   | 68.36           |

* Pure CO₂
Effect of Ca(OH)\textsubscript{2} concentration on CO\textsubscript{2} absorption

Fig. 4 shows that CO\textsubscript{2} absorption increases when Ca(OH)\textsubscript{2} concentration increases. However, the absorption of CO\textsubscript{2} in the biogas is greater than that of the pure carbon dioxide absorption. It is apparent that the use of absorbents above the solubility shows greater absorption. However, the trend of increasing CO\textsubscript{2} absorption is not obvious with an increase in concentration. The reaction occurring between Ca(OH)\textsubscript{2} and CO\textsubscript{2} is an instant reaction type, with gas resistance as a control. Ca(OH)\textsubscript{2} concentration must be excessive to obtain high absorption. When the concentration of the absorbent solution used is low, the reaction occurs in the liquid body. Whereas, if the absorbent concentration is high, the reaction will move from the liquid body to the interface between the liquid and the gas. So, it will increase the gas absorption because the gas resistance becomes small. However, the reaction will tend to remain at the interface when the absorbent concentration is constantly raised (Hayashi et al., 1975). At certain concentration limits, the absorption does not occur noticeably due to the increase in the concentration of the solution. Increasing CO\textsubscript{2} absorption by increasing absorbent flow rate becomes a more efficient option than using high absorbent concentrations. This will be useful for reducing the regeneration load of the absorbents used.

![Fig. 4 Effect of Ca(OH)\textsubscript{2} concentration on CO\textsubscript{2} absorption.](image)

Effect of initial Ca(OH)\textsubscript{2} solution level on CO\textsubscript{2} absorption

As shown in Fig. 5, the initial level of Ca(OH)\textsubscript{2} solution is varied, i.e. 50, 65, and 80 cm. The figure shows that the higher absorption is obtained on the higher initial liquid level. This trend is due to the higher initial liquid level in the bubble column resulting in longer residence time and increased interfacial area. The greater the interfacial area, the more gas that contacts to the liquid so that absorption occurs maximally. The highest CO\textsubscript{2} absorption is produced at 80 cm solution level in the continuous biogas system. In this study, the percentage of CO\textsubscript{2} absorbed in biogas were twice of pure CO\textsubscript{2}. This is due to the presence of other gas mixtures in biogas such as CH\textsubscript{4} and H\textsubscript{2}S. The other gas mixture in biogas serves as an inert. The methane gas non-polar so it cannot react with Ca(OH)\textsubscript{2} (Rashed et al., 2011). CO\textsubscript{2} in certain pH variations has different forms, namely CO\textsubscript{3}\textsuperscript{2-}, HCO\textsubscript{3}-, and H\textsubscript{2}CO\textsubscript{3} at pH> 10, 6–10, and <6, respectively. At the beginning of the reaction, calcium ions from a solution of Ca(OH)\textsubscript{2} react with CO\textsubscript{3}\textsuperscript{2-} forming CaCO\textsubscript{3} at a high pH because of the availability of hydroxide ions which can maintain the pH level in solution. When pH decreases, the presence of CO\textsubscript{3}\textsuperscript{2-} decreases and forms HCO\textsubscript{3}-, which reduces the precipitation formation. At this stage, it is also called the hatched region (HR), which is identified by a steep pH or conductivity change. Whereas at neutral pH, bicarbonate ions are formed and it is difficult to form carbonates with divalent ions. In this situation, it is difficult to form precipitation.

![Fig. 5 Effect of initial Ca(OH)\textsubscript{2} solution level on CO\textsubscript{2} absorption.](image)

To study the performance of a reaction that runs in a bubble column at a certain liquid level, conductivity measurements are carried out at two points as shown in Fig. 6. The first point is located at a height of 25 cm, while the second point at the liquid level of 50 cm, 65 cm, and 80 cm is located at a height of 45 cm, 60 cm, and 75 cm, respectively. As seen in Fig. 6, there are two stages of the mechanism in the process of forming PCC. The first stage runs for about 20 minutes where there is still high conductivity. While the second stage is characterized by a decrease in conductivity to a constant. In the first stage, the pH is still around the range 12–10, while in the second stage, the pH is ranged from <10 to neutral. According to Bang et al. (2011), CO\textsubscript{2} in certain pH variations has different forms, namely CO\textsubscript{3}\textsuperscript{2-}, HCO\textsubscript{3}-, and H\textsubscript{2}CO\textsubscript{3} at pH> 10, 6–10, and <6, respectively. At the beginning of the reaction, calcium ions from a solution of Ca(OH)\textsubscript{2} react with CO\textsubscript{3}\textsuperscript{2-} forming CaCO\textsubscript{3} at a high pH because of the availability of hydroxide ions which can maintain the pH level in solution. When pH decreases, the presence of CO\textsubscript{3}\textsuperscript{2-} decreases and forms HCO\textsubscript{3}-, which reduces the precipitation formation. At this stage, it is also called the hatched region (HR), which is identified by a steep pH or conductivity change. Whereas at neutral pH, bicarbonate ions are formed and it is difficult to form carbonates with divalent ions. In this situation, it is difficult to form precipitation.

In Fig. 6 (a), observations of conductivity at the liquid level of 50 cm by installing two points or probes at a height of 25 cm and 45 cm. It can be seen that at the liquid level of 50 cm, the formation of the maximum PCC occurs approximately 20 minutes which is indicated by high conductivity. At the initial stage, at a height of 25 cm, there is still high conductivity compared to the height of 45 cm. However, at a height of 25 cm and 45 cm, it has the same tendency in the second stage where there is a decrease in conductivity to a constant.

Next, Fig. 6 (b) shows observations of conductivity at the liquid level 65 cm by installing two points or probes at a height of 25 cm and 60 cm. It can be seen that at the liquid level of 60 cm, the formation of the maximum PCC occurs a little longer than the liquid level 50 cm. Then, this is followed by the second stage which is a decrease in the value of conductivity to a constant.

In Fig. 6 (c), observations of conductivity at the liquid level 80 cm by installing two points or probes at a height of 25 cm and 75 cm are presented. At a height of 25 cm, it has the same tendency as before, which is a decrease in conductivity value after 20 minutes. At this height, pH decreases after the 20th minute which is marked by a decrease in the conductivity value. It can be seen at a liquid level of 80 cm, the formation of the maximum PCC occurs longer at a height of 75 cm compared to the previous one which is more than 20 minutes. In the second stage, we can see a decrease in the value of conductivity to a constant, indicating that the higher the level of liquid, the more maximum the PCC is formed but at a certain high limit. Maintaining pH>10 is the most important key to ensure that the process of absorption and formation of precipitation runs optimally. By increasing the initial level of liquid at the liquid level limit, the time needed to form PCC in the first stage is much longer to allow the CO\textsubscript{2} absorption process to run maximally.

![Fig. 6 (a) shows observations of conductivity at the liquid level of 50 cm by installing two points or probes at a height of 25 cm and 45 cm.](image)

![Fig. 6 (b) shows observations of conductivity at the liquid level 65 cm by installing two points or probes at a height of 25 cm and 60 cm.](image)

![Fig. 6 (c) shows observations of conductivity at the liquid level 80 cm by installing two points or probes at a height of 25 cm and 75 cm are presented. At a height of 25 cm, it has the same tendency as before, which is a decrease in conductivity value after 20 minutes.](image)
Conductivity at different levels of liquid (a) 50 cm (b) 65 cm and (c) 80 cm.

**Figure 6** Conductivity at different levels of liquid (a) 50 cm (b) 65 cm and (c) 80 cm.

### Characteristic of Particle

As shown in Fig. 7, from several PCC samples, characterization of crystallinity with X-ray or XRD (X-ray Diffraction) was performed. XRD pattern in Fig. 7 shows the presence of peaks at $2\Theta = 23.1, 29.4, 34.2, 36, 39.5, 43.2, 47.6, 48.6, 57.6, 60.9$, and $64.9$. The peaks show the calcite phase in calcium carbonate formed in the carbonate process.

Data on X-ray fluorescence were taken on a precipitated calcium carbonate (PCC) sample obtained from the reaction between a solution of Ca(OH)$_2$ with pure CO$_2$. The composition of calcium carbonate produced in this process is shown in Table 3.

| Elements | Composition (%) |
|----------|-----------------|
| Ca       | 99.43           |
| Fe       | 0.36            |
| Cu       | 0.09            |
| Zn       | 0.02            |
| Sr       | 0.05            |
| Mo       | 0.02            |
| Ra       | 0.04            |
| Total    | 100             |

Based on the results of the morphological analysis using SEM as illustrated in Fig. 8, it shows that the calcite phase which is rhombohedral or cube is dominantly formed. The PCC size obtained from the image analysis software is between 225–270 nm.

The use of CO$_2$ contained in biogas and pure CO$_2$ in the absorption process has no effect on the crystalline phase and morphology of calcite formed. In another study, it was stated that the morphology of calcite was strongly influenced by the absorbent concentration used. Calcite with the seed-like prismatic and scalenohedron form is dominantly formed by the presence of high absorbent concentrations (Jimoh et al., 2017). Changing the properties of CO$_2$ sources using biogas will affect the density and viscosity of the gas. Biogas has a higher density than pure CO$_2$. The higher the density of the gas will change the properties of bubbles such as bubble size. This smaller bubble size will cause a greater gas hold up (Bang et al., 2015). The bigger the gas hold up the more gas is trapped in the solution so that it will increase the solubility of the gas. The use of biogas as a source of CO$_2$ will only affect the increase in solubility of...
CONCLUSION

The carbonation process using a bubble column was successfully performed to purify biogas and synthesize CaCO₃ nanoparticles simultaneously. The absorption of CO₂ increases as the absorbent flow rate fluctuates and the gas flow rate increases. The use of absorbent above the solubility shown in the continuous process shows higher absorption of CO₂. However, the upward trend of CO₂ absorption is not noteworthy with increasing concentration. The highest absorption of CO₂ obtained in the continuous process is 79.34%. Overall, the crystalline phase formed in this study is calcite with rhombohedral shape and has a particle size between 225–270 nm. The use of CO₂ contained in biogas and pure CO₂ in the absorption process has no effect on the crystalline phase and morphology of calcite formed.

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