The Effect of Amine Types on Breakthrough Separation of Methane on Biogas

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ABSTRACT. Methane (CH₄) and carbon dioxide (CO₂) are the main components of a renewable energy source of biogas. Separation of CO₂ from biogas is significantly important to improve biogas performance, due to heating value in biogas depends on the concentration of methane. One of the gas separation technologies that has been widely used in chemical industries is carbon molecular sieve (CMS). This research explores the potential of CMS for biogas purification. CMS was prepared by modification of palm kernel shell-derived porous carbon using amine groups such as monoethanolamine (MEA), 2-amino-2-methyl-1-propanol (AMP), and diethanolamine (DEA). The effect of amine types on the separation performance was studied by using a breakthrough experiment to obtain the most potential CMS materials. The methods of this research include the process of carbon oxidation using hydrogen peroxide, impregnation with an amine group, characterization of the CMS material obtained, CO₂ and CH₄ gas separation testing with a breakthrough system. The CMS was characterized by using N₂ sorption analysis, fourier transform infrared spectroscopy, and scanning electron microscopy. The breakthrough experiment showed that CMS-MEA had the highest performance for separating CO₂ and CH₄ gases. In addition, the results also showed that loading of amine groups on carbon caused an increase in the uptake capacity of CO₂, and the highest capacity was achieved by CMS-MEA of 13.2 mg/g.

Keywords: amine groups, biogas, breakthrough time, carbon molecular sieve, CO₂ gas separation

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1. Introduction

Renewable energy sources are used as an alternative to minimize the use and dependence of fossil fuels. Research and development of renewable energy have been widely studied. Biogas is a renewable energy source that can be an alternative solution to supply energy demand with advantages such as low environmental impact and high energy efficiency (Appels et al., 2008; Ariyanto et al., 2017; Poletti et al., 2011). Biogas has the potential as a substitute for fossil fuels in several applications, such as for heating, electricity generation, and vehicle fuel when upgraded to bio-methane (Rasi et al., 2011). Therefore, biogas can contribute to reducing greenhouse gas emissions.

The composition of biogas consists of methane (CH₄) and carbon dioxide (CO₂) and other impurities in very small amounts such as water (H₂O), hydrogen (H₂), nitrogen (N₂), hydrogen sulfide (H₂S), oxygen (O₂), and ammonia (NH₃). Biogas components are divided into two categories, namely combustible and non-combustible components. The detailed composition of biogas is presented in Table 1.

| Component         | Concentration | Category   |
|-------------------|---------------|------------|
| Methane (CH₄)     | 50-75%        | Combustible|
| Carbon dioxide (CO₂) | 25-50%     | Non-combustible|
| Water (H₂O)       | 1-5%          | Non-combustible|
| Hydrogen (H₂)     | <1%           | Non-combustible|
| Nitrogen (N₂)     | 0-5%          | Combustible|
| Hydrogen sulfide (H₂S) | 0-5000 ppm   | Non-combustible|
| Oxygen (O₂)       | <1%           | Non-combustible|
| Ammonia (NH₃)     | 0-500 ppm     | Non-combustible|

produced. The energy in biogas depends on the concentration of methane so that the biogas produced needs to be purified against other impurities.

The carbon dioxide content can limit the use of biogas as renewable energy. The high percentage of CO₂ in biogas causes the calorific value of the biogas to decrease, corrodes equipment, and increases emissions (Akkarawathkhoosith et al., 2019; Ricardez et al., 2012). Removal of CO₂ content can convert the concentration of methane to a higher level, which is commonly referred to as biomethane (>95% methane) so that the calorific value of biogas can be increased as contained in natural gas.

Table 1 Composition of biogas(Mursec et al., 2009; Surendra et al., 2014)

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The method that can be used to separate CO₂ and CH₄ in biogas is a molecular sieve. One of the effective potential molecular sieves for CO₂ gas separation is the carbon molecular sieve (CMS). CMS is a porous carbon material with most of the pores scattered in micropore size, which are able to distinguish molecules based on differences in diffusion rate, size, and shape (Mohamed et al., 2010; Prasetyo et al., 2020; Silvestre-Albero et al., 2009; Wahby et al., 2012). The separation of gas mixtures at CMS is determined by several parameters such as pore structure, affinity, and interaction of gas molecules with the carbon surface (Prasetyo et al., 2017). Due to the CMS has large micropores and excellent molecular sieving properties so that CMS plays an important role in gas separation (Tae-Hwan et al., 2003). One of the materials that have the potential to be used as CMS is biomass from palm kernel shells because it has a high carbon (C) content of around 50% (Rashidi & Yusup, 2017).

Several studies related to the separation of CO₂ gas using carbon molecular sieve have been studied. Palm shell was used to prepare CMS and the results showed high selectivity for the separation of O₂/N₂ and CO₂/CH₄ (Ahmad et al., 2008). The working capacity and the selectivity of the separation of CO₂ and CH₄ using commercial carbon molecular sieve have been studied, the results show that the adsorption capacity of CO₂ is greater than that of CH₄ (Song et al., 2017). Separation of CO₂/CH₄ using CMS prepared from coal tar pitch which presents molecular sieving properties to separate CO₂ from CH₄ results comparable to the yield of commercial molecular sieves (Alcañiz-Monge et al., 2011).

The performance of porous carbon such as CMS for binary gas separation can be improved by several treatments such as oxidation, heat treatment, and chemical impregnation with acid or alkaline solutions on the carbon surface (Lee et al., 2013). In previous research, hydrogen peroxide has been applied for the oxidation of carbon molecular sieve to enhance the separation of CH₄/CO₂ mixed gas (Cho et al., 2015). Other research has been reported to identify the effect of impregnation CMS using ammonium hydroxide (Bai et al., 2013). Both kinds of research show the improvement of the breakthrough separation process of CO₂ and CH₄ gases. Based on this literature, the presence of chemical species on the carbon surface greatly affects the performance of carbon materials.

Research related to CMS modification on the performance of breakthrough separation is still being developed. Porous carbon impregnated with alkaline groups such as amines have the potential to capture CO₂ in biogas due to the primary and secondary amines can react with CO₂ to form stable compounds and can be regenerated to release adsorbed CO₂ (Das & Meikap, 2020). However, CMS is less hydrophilic than other similar porous materials (Tae-Hwan et al., 2003). In this study, a combination of several treatments was used to improve the performance of CMS. CMS was prepared by modification of palm kernel shell-derived porous carbon using two treatments including oxidation and impregnation of amine groups. The oxidation treatment using hydrogen peroxide was previous carried out during the impregnation process. Oxidation is a pre-treatment that can change the nature of CMS from hydrophobic to hydrophilic so that the impregnation process of amines becomes easier. Then the types of amines used for the impregnation process include monoethanolamine (MEA), 2-amino-2-methyl-1-propanol (AMP), and diethanolamine (DEA). The three types of amines were studied to determine their effect on breakthrough time so that the material with the best performance for CO₂ separation was obtained.

2. Materials and Methods

2.1 Materials

The materials used in this study were palm kernel shell-based carbon molecular sieve (CMS) provided by PT Home System, Indonesia, hydrogen peroxide (H₂O₂) 50% and monoethanolamine (MEA) 99.5% supplied by Merck, 2-amino-2-methyl-1-propanol (AMP) 90%, and diethanolamine (DEA) 98% obtained from Sigma Aldrich. Then, the gases used for separation testing such as nitrogen (N₂) gas 100% and a gas mixture of methane (CH₄) 55% and carbon dioxide (CO₂) 45% which represents the composition of the biogas were imported from PT Samator Gas.

![Fig. 1. Scheme of separation testing with a breakthrough system. The scheme taken from (Prasetyo et al., 2020).](image-url)
2.2 Oxidation Process

The CMS was crushed and sieved with a size of around 20-25 mesh and then oxidized using a 10% H₂O₂ solution. The oxidation process begins by diluting the 50% H₂O₂ solution to 10%. Oxidation was carried out in a three-neck flask by mixing 25 grams of CMS with 250 ml of 10% H₂O₂ solution at room temperature for 2 h. After that, the oxidized carbon was separated with H₂O₂ solution and dried in an oven at 70 °C for 24 h.

2.3 Impregnation Process

In this method, amines were mixed with the CMS with the help of a solvent. The solvent was then removed by evaporation. The impregnation process was prepared by mixing 25 grams of oxidized CMS, 1.3 grams of amines such as MEA, AMP, and DEA (5% w/w) in the beaker, and adding distilled water until form slurry. The slurry was stirred using a magnetic stirrer at 300 rpm for 1 h at room temperature. The mixture of CMS and amines was allowed to stand for 24 h at room temperature until the distilled water evaporated and then dried in an oven at 70°C for 24 h. The materials impregnated were labeled as CMS-MEA, CMS-AMP, and CMS-DEA.

2.4 Characterization

Total surface area, pore-volume, and pore size of the CMS materials were characterized by the N₂ sorption method using the NOVA 2000 analyzer (Quantachrome) at 77 K. Identification of the functional groups in the wavenumber range 400-4000 cm⁻¹ was carried out by Fourier transform infrared spectroscopy (FTIR) using 8201 PC FTIR, Shimadzu. Then, the morphology of the materials was characterized using scanning electron microscopy (SEM) with the JEOL JSM-6510LA instrument at a voltage of 15 kV.

2.5 Separation testing

The separation process of CO₂ and CH₄ was carried out using a breakthrough system whose equipment scheme is shown in Fig.1. Inside the bed was filled with 14 grams of CMS material. CMS was first flushed by nitrogen gas with a flow rate of 200 ml/min until the gas detector showed no detected CH₄, CO₂, and O₂ gas content. After that, the gas mixture of CH₄ and CO₂ flowed into the system with a flow rate of 100 ml/min at ambient temperature and pressure of 1.2 bar. The composition of methane and carbon dioxide was detected by a portable gas analyzer (Biogas Analyzer Gas board 3200plus, Hubei Cubic-Ruiyi Instrument Co., Ltd). The separation process was stopped when the concentrations of CO₂ and CH₄ gases detected by the portable gas analyzer were 35.5% and 54.5% respectively. After the separation process, CMS was flushed by passing nitrogen gas to the separator column at a flow rate of 200 ml/min.

Table 2

| Materials   | Total surface area (m²/g) | Total pore volume (cm³/g) | Average pore size (nm) |
|-------------|--------------------------|---------------------------|------------------------|
| CMS         | 708                      | 0.33                      | 1.87                   |
| CMS-MEA     | 164                      | 0.10                      | 2.40                   |
| CMS-AMP     | 228                      | 0.14                      | 2.39                   |
| CMS-DEA     | 211                      | 0.13                      | 2.40                   |

3. Results and Discussion

3.1 Characterization Results

3.1.1 N₂ Sorption Analysis

Table 2 shows the total surface area, pore-volume, and average pore size of the unmodified and modified CMSs. The results of the N₂ sorption analysis showed that the modified CMS experienced a significant reduction in surface area and pore volume. The decrease in pores indicates that the impregnation process has been successful. The total surface area of the CMS is 708 m²/g after impregnation, the surface area drops to 164 m²/g (CMS-MEA), 228 m²/g (CMS-AMP), and 221 m²/g (CMS-DEA). The total pore volume respectively decreases to 0.10 cm³/g (CMS-MEA), 0.14 cm³/g (CMS-AMP), and 0.13 cm³/g (CMS-DEA). This decrease is caused by amine groups that have filled the pores of the carbon and blocked most of the pore structures. Although they clog pores, amines provide an active site that can increase the interaction between CO₂ and carbon. The specific surface area and pore volume are considered very important parameters during physiosorption. However, in this study, the pores have a role to increase the number of amines that enter the carbon through the impregnation process.
3.1.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fig. 2(a) shows the FTIR spectrum of unmodified CMS and oxidized CMS using hydrogen peroxide. A strong peak at 3400 cm\(^{-1}\) indicates O-H stretching, while the strong spectra at 1085 cm\(^{-1}\) show the presence of C-O stretching of alcohol groups. In the FTIR spectra, it was shown that oxidized CMS had a sharper peak. This indicates that oxidation using hydrogen peroxide produces an OH group. The pre-oxidation helps to increase the N loading during the impregnation process, due to the presence of the O functional on the surface plays an important role in determining the amount of N loading to the carbon surface.

FTIR spectrum of the amine impregnated CMS is shown in Fig. 2(b) the success of the amine impregnation process into the carbon structure can be seen from the presence of the N-H bending vibration of amines groups at the spectra of 1655-1641 cm\(^{-1}\) (Kongnoo et al., 2016). In this study, FTIR results on CMS-MEA, CMS-AMP, and CMS-DEA materials showed that the N-H bending was indicated at the peaks of 1650 cm\(^{-1}\). In addition, it is seen that the C-O stretching peak of the carbon-bound alcohol group is not as sharp as in Fig. 2(a), due to the C-O has reacted with an amine. The wavenumber 3400 cm\(^{-1}\) associated with the N-H stretching where shows that CMS-MEA has a sharper peak than other materials. This allows that the loading amount of amines is greater.

3.1.3 Scanning Electron Microscopy (SEM)

SEM analysis results showed a significant difference in morphological structure between carbon without impregnation and carbon loaded with amines. The carbon
surface before impregnation is presented in Fig. 3(a), while the carbon surface after impregnation with amines is presented in Figs. 3(b-d). Fig. 3(a) shows that carbon without impregnation has a denser morphology. Meanwhile, the amine impregnated carbon has a more hollow morphology as shown in Figs. 3(b), 3(c), and 3(d). CMS-MEA shows a large cavity compared to CMS-AMP and CMS-DEA. SEM images only show the surface morphology of the carbon, not showing a micro or mesopored.

3.2 Breakthrough time result

The breakthrough time was studied to measure the performance of CO\textsubscript{2} and CH\textsubscript{4} gas separation. Fig. 4(a) shows the breakthrough curves of CO\textsubscript{2}, while the breakthrough curve of CH\textsubscript{4} can be seen in Fig. 4(b). In the beginning, CH\textsubscript{4} and CO\textsubscript{2} gas detected by the portable gas analyzer showed the same concentration of 0%. However, the difference in the diffusion rate of gas molecules causes the CH\textsubscript{4} molecule to pass through the pores faster than the CO\textsubscript{2} molecule which allows the two gases to separate. The diffusivity of CH\textsubscript{4} and CO\textsubscript{2} was 1 x 10\textsuperscript{-10} m\textsuperscript{2}/s and 1 x 10\textsuperscript{-11} m\textsuperscript{2}/s respectively (Prasetyo et al., 2020). In addition, carbon is more selective towards CO\textsubscript{2} than CH\textsubscript{4} because of its higher affinity for carbon. Due to the diffusion rate of the CO\textsubscript{2} molecule is slower, it allows this molecule to be trapped on the carbon. The CO\textsubscript{2} gas started to breakthrough out from the carbon when carbon is in the saturation stage. Thus, the CO\textsubscript{2} concentration displayed increases to its initial concentration and the methane gas concentration decreases again.

Modification of CMS by impregnated amine is the method mostly used to enhance the interaction between the carbon surface and acid molecules, such as CO\textsubscript{2}, H\textsubscript{2}S, and SO\textsubscript{2} (Sevilla et al., 2012). The results showed that CMS-MEA, CMS-AMP, and CMS-DEA take a longer time to reach saturation point compared to non-impregnated CMS. The amine group provides the active site on the carbon surface which can increase the capture of CO\textsubscript{2} molecules due to the chemisorption effect. The active site from the N functional group will cause increasing the polarity and basicity properties of the carbon surface (Sevilla et al., 2012). The interaction between CO\textsubscript{2} and amines occurs based on the Lewis acid-base principle. Amine acts as Lewis’s base inducing electron-donor, while CO\textsubscript{2} acts as an electron-accepting Lewis acid. Its properties provide a high affinity for a stronger interaction between CO\textsubscript{2} and carbon (Chiang & Juang, 2017).

On the breakthrough curve seen that CMS-DEA is the first to reach the saturation point of the three types of amines. It may be related to DEA which is a group of secondary amines and has a larger molecular weight (105 g/mol). Secondary amines were less reactive than primary amines (Zelenak et al., 2008). MEA and AMP were included as primary amines. However, for the same amine groups, the structure of the smaller amine and have fewer branches will provide more active sites (Khalil et al., 2012). AMP has a larger molecular weight (89 g/mol) and more branched which makes it difficult for the amines molecule to occupy the pores. Therefore, this experiment shows that CMS-MEA has a longer breakthrough time than CMS-AMP and CMS-DEA.

The CO\textsubscript{2} sorption capacity can be determined when CO\textsubscript{2} first interacts with carbon until the CO\textsubscript{2} molecules started to breakthrough out from the column. The uptake capacity of CO\textsubscript{2} for impregnated and non-impregnated CMS is presented in Table 3. Based on Table 3 the loading of amines on carbon shows a better CO\textsubscript{2} sorption performance. The sorption capacity of CO\textsubscript{2} increased to 13.2 mg/g for CMS-MEA, 11.8 mg/g for CMS-AMP, and 9.9 mg/g for CMS-DEA, respectively. Thus, the active site on the carbon surface has stronger properties so that in this study the process is more likely to be chemisorption. In previous studies, CMS impregnated with ammonium hydroxide had increased CO\textsubscript{2} sorption capacity by 24% (1.61 mmol/g to 2.0 mmol/g at 298K) as an effect of the introduced nitrogen function (Bai et al., 2013). Other materials such as fly ash carbon-impregnated by MEA can increase the CO\textsubscript{2} uptake up to 64% (41.8 mg/g to 68.6 mg/g at 30°C) (Mercedes Maroto-Valer et al., 2008).

![Fig. 4 Breakthrough curve of a) CO\textsubscript{2} and b) CH\textsubscript{4}](image-url)
Table 3
Uptake capacity of CO₂

| Materials | CO₂ uptake (mg/g) | Enhancement (%) |
|-----------|------------------|-----------------|
| CMS       | 7.9              |                 |
| CMS-MEA   | 13.2             | 67              |
| CMS-AMP   | 11.8             | 49              |
| CMS-DEA   | 9.9              | 25              |

6. Conclusion

CMS was prepared by modification of palm kernel shell-derived porous carbon using amine groups that have the potential for biogas purification. The introduction of amine groups into the carbon structure causes the pore structure to become clogged so that the surface area is reduced as shown in the BET results. However, impregnation of carbon with base groups has increased the chemical surface for carbon capture. The presence of N functional groups can increase the acid-base interaction between CO₂ and the carbon surface due to the chemisorption effect. The oxidation treatment before the impregnation process gives the functional group O on the carbon surface as seen in the FTIR results, where the O functional group has an important role in increasing the N loading on the carbon. Loading of amines on CMS showed better breakthrough separation performance compared to CMS without amine impregnated. CMS impregnated with MEA showed a longer breakthrough time and higher CO₂ sorption capacity.

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