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Original Research Article

Biogas Purification using Modified Red Mud Adsorbent with a Study of the Length of the Adsorbent Column

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Abstract

This study used red mud as an adsorbent for biogas purification. However, red mud had to undergo dealumination and calcination processes before being used as an adsorbent. Several acids, including 1 M HCl and 1M H2SO4, were employed in the red mud dealumination process, which was then analysed by XRF and GSA instruments. The results show that the best type of acid for the dealumination process was HCl with a Si/Al ratio, total pore volume, surface area and pore radius of 1.697; 67,081 m2/g; 0.1624 cm3/g; 48.4242 Å. The biogas purification process was carried out using red mud adsorbent dealuminated with HCl by varying the length of the adsorbent column, such as 10, 20 and 30 cm, and the contact time of 5, 10, 15 and 20 min. The optimum column length variation was 30 cm. The results obtained for this variation were a decrease in CO_2 content of 292742.30 ppm with an efficiency of 19.08%. The increase in CH_4 was 378232.69 ppm with a percentage increase of 240.28%.

Keywords: Adsorbent; biogas; dealumination; purification; red mud

1. Introduction

Indonesia possesses a significant amount of bauxite resources The country's total bauxite reserves will be 2.38 billion tonnes in 2020, with 1.9 billion tonnes located in West Kalimantan, according to the Ministry of Energy and Mineral Resources (ESDM). Bauxite is a rich source of alumina hydroxide, This makes it a commonly used raw material to produce alumina (Al₂O₃) using a Bayer process. However, processing bauxite into alumina generates red mud as a solid waste (Liu et al., 2014).

Bayer process can produce approximately 1 to 1.5 tons of red mud per ton of alumina. The production of red mud has significantly increased due to the global demand for alumina. (Liu & Naidu, 2014) reported that the alumina industry produces approximately 4 billion tons of red mud waste globally. The significant impacts on the environment that may arise from the huge amount of red mud generated and its notable high pH must be considered. Therefore, it is crucial to implement effective methods to reuse the red mud.

Typically, red mud contains six main oxides, namely AlO₃, Fe₂O₃, Na₂O, SiO₂, CaO, and TiO₂. The properties of red mud relies on the bauxite ore source used on the production. According to a study by (Sen, 2018), red mud contains a metal oxides with the potential to act as adsorbents for pollutant gases. Earlier studies have also stated that red mud effectively removes CO₂, CO and H₂S (Hien et al., 2019; Sushil & Batra, 2012). In order to use red mud as the adsorbent for pollutant gases, the surface morphology of the red mud needs to be improved by modification through various

methods, namely dealumination and calcination processes. Dealumination involves the release of aluminium from the framework to the outer surface by acid treatment. Aluminum released on the adsorbent surface could increase the Si/Al ratio (Maleiva, 2015). Dealumination can be achieved by reacting the adsorbent with an acid solution. This study investigate an adsorbent without dealumizing it using H_2SO_4 and HCl. The acid treatment has the potential to increase the adsorbent surface area (Minh, 2017). Specifically, the acid solutions will give a distinctive changes in the material and facilitates the substance adsorption (Ismettulloh et al., 2019). Furthermore, calcination removes water compounds that may block adsorbent pores (Mrosso et al., 2020).

Red mud, which has undergone dealumination and calcination, is used as an adsorbent to enhance the methane (CH₄) content in biogas, resulting an increase in its energy content (calorific value). This process is required to reduce the amount of the impurities, in particular water vapour (H₂O), carbon dioxide (CO₂), hydrogen sulphide (H₂S), and nitrogen (N₂) gases, which is known as the purification process (Pertiwiningrum, 2016). The SiO₂ and Fe₂O₃ compounds present in red mud have the potential to significantly reduce impurities in biogas, particularly CO₂, by up to 29% with 6.4 mmol/g absorption capacity (Aulia, 2022). Additionally, Fe₂O₃ compounds can adsorb biogas impurities on the adsorbent surface. The Fe₂O₃ surface can effectively bind with CO₂ compounds, with a bond strength of 14.98 kcal/mol. Furthermore, Fe₂O₃ also has a very reactive property towards H₂S with a binding strength of 27.07 kcal/mol. Although Fe₂O₃ has a weaker bond energy with CH₄ than with biogas impurities, resulting in weaker interactions (Thanakunpaisit et al., 2017), these interactions cause CH₄ gas to be released earlier from the adsorbent pores and lead to the absorption of impurity gases on the adsorbent surface. So the CH₄ gas level increases and the impurity gas level decreases.

Based on theory, the length of the column impacts the amount of adsorbent. As the column's length increases, the amount of adsorbate that can be adsorbed also increases (Gantina and Farhani, 2020). Increasing the mass of adsorbents will enhance the purity of biogas (Widhiyanuriyawan et al., 2014). Thus, this study aims to further investigate how column length affects the rise in CH_4 content and the reduction of impurities within biogas through the utilization of red mud adsorbents which have been modified via dealumination. This study also aims to evaluate the acid influence in the dealumination process to the properties of the red mud. X-Ray Fluorescence (XRF) and Gas Sorption Analyzer (GSA) will be used for characterization, and Gas Chromatography (GC) will be used to investigate the levels of CH_4 and impurities in the biogas before and after purification.

2. Methods

2.1. Schematic of the experimental set-up

Figure 1. displays the scheme for purifying biogas. The purification process is initiated by the output of the biogas digester, which flows into a column containing modified Red Mud adsorbent material. The columns used correspond to predetermined variations, with observations made at each specified time interval.



Figure 1. Schematic of the biogas purification set-up

2.2 Methods

2.2.1 Red Mud Pre-treatment

The samples of red mud were obtained from PT Indonesia Chemical Alumina (ICA) and dried in the sun for one day to reduce the moisture content. Afterward, the samples are oven-dried at 100°C for 2 hours to ensure that there is no residual water content (Prajapati et al., 2016)

2.2.2 Production of the Red Mud Adsorbent

Two types of acid, hydrochloric acid (HCl) and sulfuric acid (H₂SO₄), were employed in the dealumination process, each with a concentration of 1M. Red mud was combined with an acidic solution in a 20 ml/g (solid/liquid) ratio before being immersed in acid for 4 hours to ensure a perfect dealumination process. Two types of acid were used in the process to study the effect of different types of acid on the dealumination process. The mixture then was filtered and rinsed by water until it reached a neutral pH before calcined at 700°C for two hours in a kiln (Prajapati et al., 2016). The treatment of red mud with acid is known to improve the surface area (Tangde et al., 2019). Therefore, in this study red mud was calcined at 700°C, as it was found to be the optimum temperature for surface area improvement. If the material is calcinated at a temperature higher than the optimum, it will decompose and damage the red mud adsorbent, leading to a reduction in its adsorption capacity (Ahmed et al., 2020). The red mud sample was then left for 24 hours. The calcined red mud was then sieved through a 100 mesh sieve and mixed with bentonite at a ratio of 4:1 (red mud to bentonite). In this study, bentonite was used as an adhesive. Bentonite is a commonly utilized adhesive that binds material components, resulting in a more compact structure and enhanced physical strength of the material (Athallah et al., 2022). To ensure optimal blending of the red mud and bentonite, a sufficient quantity of aquades was added to the mixture before it was pelletized. The resulting pellets were subsequently oven-dried for one hour at a temperature of 100°C (Maleiva et al., 2015).

2.2.3. Red Mud Characterization

X-ray fluorescence (XRF) characterisation was used to identify the red mud composition before and after activation. Furthermore, a Gas Sorption Analyzer (GSA) instrument was utilized to identify the pore characteristics of the red mud adsorbents.

2.2.4. Biogas Adsorption

The biogas raw material was obtained from the Material Recovery Facility (MRF) Edelweis Pontianak. The biogas from the reservoir is then fed into an adsorption column at 1 liter/minute constant flow rate. The adsorbent column, with 2 inches diameter, is made of PVC (polyvinyl chloride) material with a length variation of 10, 20 and 30 cm. The variation of the column length is intended to see the effect of the column length on the reduction of biogas impurities. Biogas sampling was carried out before and after the purification process at the 5th, 10th, 15th, and 20th minute to see the effect of the length of biogas and adsorbent contact time to decreasing impurities at each time (Zulkefli et al., 2017). The biogas samples were then analyzed using gas chromatography (GC) instruments to investigate the effect of the purification process on the biogas composition.

2.3. Calculation

The CO₂ removal efficiency is calculated using Equation 1, listed below (Maleiva et al., 2015).

$$Efficiency = \frac{C_0 - C}{C_0} \times 100\% \qquad (1)$$

Where:

 C_0 is the amount of CO_2 before purification (ppm) C is the amount of CO_2 after purification (ppm)

As for the percentage of the increasing CH_4 can be calculated using Equation 2 listed below (Maleiva et al., 2015).

$$\% = \frac{C_2 - C_1}{C_2} \times 100\%$$
 (2)

Where:

 C_1 is the amount of CH_4 before purification (ppm) C_2 is the amount of CH_4 after purification (ppm)

3. Result and Discussion

3.1. Effect of Activation on the Composition of Red Mud

Data obtained from XRF analysis of the red mud before and after activation are presented at Table 1 below.

Elements	Chemical composition of red mud (%)		
	Without	Activated with	Activated with
	Activation	H_2SO_4	HCl
Mg	4.094	3.45	3.611
Al	10.209	4.539	4.941
Si	10.861	7.107	8.388
Р	1.677	1.413	1.398
K	0.16	0.254	0.237
Ca	3.93	0.723	0.771
Ti	3.93	4.214	4.957
V	0.221	0.236	0.262
Cr	0.047	0.052	0.052
Mn	0,146	0.121	0.158
Fe	64.764	77.677	74.988
Eu	0.221	0.203	0.227
Yb	0.008	0.012	0.01

Table 1. Composition of red mud before and after activation

According to the XRF analysis in **Table 1**., the Si/Al ratio in red mud adsorbents increased after activation, which was achieved by dealumination and calcination methods. The Si/Al ratio in red mud before the activation was 1.064, while in the red mud treated with dealumination using H_2SO_4 , the Si/Al ratio increased to 1.566. Meanwhile, the red mud treated with HCl experienced a greater increase in Si/Al ratio than the H_2SO_4 -treated red mud, with a ratio of 1.698. This is due to the difference in acid strength between H_2SO_4 and HCl, as the strength of HCl acid is greater than that of H_2SO_4 .

The dissociation constant (Ka) value determines the strength of an acid. HCl and H2SO₄ have Ka values of 10⁷ and 10², respectively. A greater Ka value indicates easier dissociation, so Cl⁻ has a greater ability to bind to Al³⁺ compared to SO₄²⁻ (Sulistyowati & Darmawan, 2018). The bond energy of Al-Cl is 119 kcal/mol, while the bond energy of Al-SO₄ is 79 kcal/mol. This implies that the bond formed between Al-Cl is stronger than that between Al-SO₄ (Side et al., 2023).

3.2. Characterization of Red Mud Pores

Data obtained from the red mud GSA analysis before and after activation are presented at Table

2.

Pore	Red Mud			
Characteristics –	Without	Activated	Activated with HCl	
	Activation	with H_2SO_4		
Surface area	23.994	63.078	67.081	
(m ² /g)				
Total pore	0.08428	0.1566	0.1624	
volume (cm³/g)				
Pore radius (Å)	70.2524	49.6525	48.4242	

 Table 2. Characteristics of red mud pores

Red mud surface area that has been activated by dealumination and calcination process is increased compared to the non-activated red mud, as shown in Table 2. The highest increase was observed in red mud dealuminated by HCl, which was $67.081 \text{ m}^2/\text{g}$. Acidic solutions are capable of enhancing adsorbent surface area. Treatment with acid solutions leads to characteristic changes in the material (Minh, 2017). The ease of material in adsorbing a substance is affected by the characteristic changes in the material (Ismettulloh et al., 2019).

Furthermore, the dealumination method can enhance the total pore volume of red mud. In particular, red mud treated with HCl gave the highest increase in total pore volume, which was 0.1624 cm₃/g. The total pore volume represents the total amount of adsorbate that can be adsorbed. An enlargement of the total pore volume increases the adsorbate that can be adsorbed in the adsorbent pores (Maleiva et al., 2015).

Table 2. show that the average of the red mud pore radius becomes smaller as the surface area and total pore volume increase. The HCl activared red mud gave the smallest average pore radius of 48.4242 Å. A correlation was observed between the surface area and the total pore volume, and a decrease in the pore flat radius was observed, which makes the red mud suitable as a gas adsorbent (Widyastuti et al., 2013). The dealumination and calcination method of red mud activation can produce adsorbents with porous structures that can remove impurity gases like H2S, CO2, H2O, and N2 from biogas.

3.3. The Effect of Adsorbent Column Length and Contact Time on CO₂ Removal

The test results of CO₂ content before and after purification shown in Figure 2. and Figure 3.



Figure 2. Graph of CO₂ content value after purification

Figure 2. shows an increase in CO_2 in the column variations of 10 cm with a purification time of 10 minutes. The CO_2 gas levels rose from 343624.34 ppm to 347150.07 ppm. The rise in CO_2 content occurs due to the release of CO_2 gas from the surface of the red mud adsorbent. An adsorbate can detach from the adsorbent as the van der Waals bonding affects the adsorption process. The process is reversible because the bond is weak and easy to desorb (Nurfitriyani et al., 2013).

Figure 3. demonstrates that biogas CO₂ content tends to decrease with an increase in adsorbent column length (Gantina and Farhani, 2020). The greatest CO₂ decrease was observed for a 30 cm column and 20-minute purification time, with CO₂ content decreasing from 361754.20 ppm before purification to 292742.30 ppm, resulting in a removal efficiency of 19.08%.



Figure 3. Graph of CO₂ removal after purification

The reduction in CO_2 content is a result of CO_2 gas adsorption in the red mud adsorbent treated with HCl. The CO_2 gas can be adsorbed by the red mud adsorbent as CO_2 can bind to the SiO₂ surface. Furthermore, the CO_2 content is reduced by increasing the length of the adsorbent column. The increased length of the adsorbent column during the adsorption process results in a wider contact surface between the adsorbent and impurities gas, enabling greater adsorption of CO_2 gas by the red mud adsorbent (Zulkefli et al., 2017). Additionally, CO_2 gas has the ability to bind to the surface of Fe_2O_3 compounds. The bond energy of CO_2 and SiO_2 is 108 kcal/mol, while the bond energy of CO_2 and Fe_2O_3 is 14.98 kcal/mol.

Table 3. and **Figure 2.** demonstrate how the duration of the purification process can impact the CO_2 gas concentration in biogas. The findings reveal that an increase of purification time led to a reduction of the CO_2 content, with the most significant decrease occurring after a 20-minute variation. This is due to the CO_2 gas taking much longer to adsorb on the adsorbent and therefore more CO_2 gas is adsorbed on the adsorbent surface (Ritonga, Masruhi, et al., 2021). The adsorption of CO_2 gas onto the surface of the red mud adsorbent results in the reduction of CO_2 gas in the biogas.

3.4. Effect of Adsorbent Column Length and Contact Time on Increased CH₄ in Biogas

Based on the data presented in **Figure 4.**, there was a decrease in CH_4 content by 17474.08 ppm, from 262377.74 ppm to 244903.66 ppm, when the column length varied by 10 cm in the 10th minute. The decrease in CH_4 was due to the increase in CO_2 gas content by 17474.08 ppm, from 262377.74 ppm to 244903.66 ppm, when the column length varied by 10 cm in the 10th minute. However, with an increase in column length adsorption, the CH_4 gas content showed a tendency to increase. The highest CH_4 content was obtained in the adsorption column with a length of 30 cm, which increased from biogas before purification by 11153.82 ppm to 378232.69 ppm.

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Figure 4. Graph of CH₄ content value after purification

The increase in CH₄ is due to the adsorption column with a length of 30 cm contains more red mud adsorbents activated with HCl. **Figure 5.** presents the efficiency of increasing CH₄ at different column lengths.



Figure 5. Graph of CH₄ increasing efficiency after purification

A large number of adsorbents will produce larger active surface areas. The greater the active surface area, the more biogas impurities adsorbed by the adsorbent. As a result, the CH_4 content of the biogas can be increased (Adisasmito et al., 2020).

Moreover, the adsorbent column length can also influence the increase in CH_4 gas. By increasing the length of the column, adsorbents have an extended contact time to remove biogas impurities (Simanjuntak et al., 2023). Therefore, a column with a height of 30 cm provides a longer contact time for impurities to be adsorbed compared to columns with heights of 10 and 20 cm.

Table 4 and **Figure 3** demonstrate a positive correlation between purification time and CH_4 content, with the highest increase occurred in the 20th minute. This shows that in addition to the influence of the length of the adsorbent column, the increase in CH_4 is also influenced by the length of the purification time. The longer the purification time, the longer the contact time between biogas and adsorbents, resulting in more impurities in biogas being adsorbed. The adsorption of impurity gases has a major influence on the CH_4 levels produced. By lowering the impurity gases in biogas, the CH_4 content can be increased (Abdullah et al., 2015)). However, a prolonged biogas purification process can lead to reduced adsorbate amounts due to saturation of adsorbents (Dewi et al., 2019).

The CH₄ content test indicate a significant increase of the CH₄ in each sample, specifically up to 240.28% in the 30 cm column variation with a purification time of 20 minutes. Another thing that can cause an increase in CH₄ content is the molecular weight. Impurities in biogas such as CO₂ have a molecular weight of 44 g/mol, H₂S 34.1 g/mol, N₂ 28 g/mol, H₂O 18 g/mol, and CH₄ gas 16 g/mol. This shows that CH₄ gas is much lighter than other biogas impurities. The molecular weight can impact the rate of diffusion within the pore and consequently impact the adsorption process. Methane gas with a

very low molecular weight can diffuse more rapidly through the adsorbent surface without contact than gases with a higher molecular weight (Iriani and Heryadi, 2014).

3.5. Flame Test

The flame test in this research was conducted by burning biogas coming out of the gas discharge hose. The results of the flame color test presented in **Figure 6**.



Figure 6. Biogas flame test results (a) before purification (b) purification with 10 cm column (c) purification with 20 cm column (d) purification with 30 cm column

Flame tests conducted on biogas before and after purification at varying column lengths showed that the gas could ignite when brought close to a fire source. This observation confirms the presence of methane in both before and after purification biogas. Biogas containing methane will also burn when brought close to the source of the fire (Yahya et al., 2018).

When biogas before purification was subjected to a flame test, the colour of the flame produced was reddish. This particular reddish flame colour indicates that the biogas contains CO_2 gas (Ritonga, Masrukhi, et al., 2021) After the purification process using adsorbent columns of 10, 20 and 30 cm, the colour of the biogas flame produced has changed, where the colour of the flame tends to be blue and slightly reddish. The results indicate a decrease in CO_2 gas content in biogas due to the adsorption of CO_2 gas by red mud adsorbents. Furthermore, the change in colour demonstrate an increase in CH_4 gas content of the purified biogas using 10, 20, and 30 cm adsorbent columns. When the biogas experiences an increase in methane content and a decrease in carbon dioxide, the resulting fire tends to be blue (Abdullah et al., 2015).

4. Conclusions

The type of acid used in the dealumination process can affect the red mud adsorbents characteristics, which can increase the Si/Al ratio, surface area, and total pore volume. Optimal dealumination is achieved through the use of HCl, wherein the Si/Al ratio, total pore volume, surface area, and pore radius are respectively 1.697, 67.081 m2/g, 0.1624 cm3/g, and 48.4242 Å. This study demonstrates how the length of the column interferes with the efficiency of CO₂ reduction. By increasing the column length, the adsorbents have a longer contact time to adsorb CO₂. The optimal length for the column is 30cm, which results in a 292742.30 ppm reduction in CO₂ content with an efficiency of 19.08%. Additionally, the amount of CH₄ increased by 378232.69 ppm with a percentage increase of 240.28%. Other



gas parameters, particularly H₂S gas, were not tested in this study, therefore the effect of high Fe_2O_3 content on Redmud was not known hence in future studies it will be necessary to test other gas parameters besides CH_4 gas and CO_2 gas, mainly H₂S.

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