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Key words: *biogas purification, adsorbent of red brick powder, 2^3 factorial design*

Cite article:
Slamet, W., Firman, N. E., & Ahmat, F. [2021]. The effects of adsorbent mass using red brick powder on the results of biogas purification. *Journal of Applied Engineering Science*, 19(1), 17 - 23. DOI:10.5937/jaes0-27726

Online aceess *of full paper is available at:* www.engineeringscience.rs/browse-issues

Original Scientific Paper

Istraživanja i projektovanja za privredu
Journal of Applied Engineering Science

doi:10.5937/jaes0-27726 Paper number: 19(2021)1, 758, 17-23

THE EFFECTS OF ADSORBENT MASS USING RED BRICK POWDER ON THE RESULTS OF BIOGAS PURIFICATION

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Biogas is produced by the digestion of organic waste by anaerobic bacteria. However, the application of raw biogas is not effective because it consists of impurities such as carbon dioxide (CO_2), hydrogen sulfide (H_2S), water vapor (H_2O), and other impurity gases. Physical Adsorption is the simplest method of immobilization of biomolecules such as CO_2 which is attached to the surface through the weak bonds like van der Waals forces. One of the physical adsorption means to reduce CO_2 levels in biogas is to use brick powder. Increasing of brick powder adsorbent mass caused the decrease of CO_2 concentration in the biogas purification. Brick is a porous material containing SiO_2 – Al_2O_3, so it has the ability to form Van Der Waals bonding forces with CO_2. The goal of this research is to determine the efficiency of brick powder as a purification adsorbent to reduce of CO_2 levels using the 2^3 factorial design method. The application of 200 and 400 grams brick powder adsorbents, with the biogas flow rate of 1 and 2 liters/minute, was researched at intervals of 5 and 20 minutes, for the CO_2 concentration data and heating values of biogas. Gas Chromatography (GC) was used to determine the concentration of adsorption gases, especially CO_2 and CH_4. The results showed that the biggest efficiency reduction in CO_2 concentration is 59.28 %.

Key words: biogas purification, adsorbent of red brick powder, 2^3 factorial design

INTRODUCTION

Renewable energy production is a big issue in the world. With different technologies, a lot of renewable energy can be obtained, such as: solar energy, wind energy, geothermal energy, and biomass energy [1]. Biomass is the main energy resource for biogas as a renewable energy, being mostly found in agricultural residues, livestock manure, and others [2]. Organic households, restaurants, parks, and wastes from industry [3], [4]. Biogas is produced from anaerobic bacterial digestion of degraded organic waste [5]. The composition of biogas consists of 35-75 % methane, 25-65 % carbon dioxide, 1-5 % hydrogen, and tracer of water vapor, ammonia, and hydrogen sulfide. Poor biogas quality (CH_4 / CO_2 < 1) exacerbates damage to the atmosphere [6]. Purification techniques are used to clean and enhance biogas into biomethane which can replace natural gas [7].

Purification of biogas aims to improve its quality by separating non-methane gases, specifically carbon dioxide, which can reduce the heating value and the combustion efficiency [8]. Decreasing the concentration of carbon dioxide can be done by physical adsorption, which is the simplest method to immobilize of biomolecules such as CO_2 which are bounded to the surface through weak bonds such as van der Waals forces [9]. There are three types of adsorbents, including elements that contain carbon (activated carbon and graphite), polymer elements (porous polymer matrix), and elements that contain oxides (silica gel and zeolites) [10].

XRF testing is carried out to find out the elements that exist in a material such as clay by bombarding the material with high energy X-rays or gamma rays so that the waveforms of the clay-forming elements are detected. [11]. Red brick is one of the ceramic products made from clay whose particles are shaped like a sheet that has a special surface with Al_2O_3 and SiO_2 content in it [12]. Clay has high adsorption ability, it has very strong compressive power, it wrinkes when dried, and it has fine-grained granules so that it can be categorized as montmorillonite [13]. A mixture of tile powder and zeolite can reduce CO_2 concentrations by 34.56 % [14].

RESEARCH METHODS

Red brick powder, which came from the village of Urek - Urek Gondanglegi, Malang, Indonesian. The red brick adsorbent had fine structure, with particle sizez between 0.0625-0.4 mm. Each adsorbtion tube was filled with 200 and 400 g of adsorbent. Tests were conducted at biogas flow rates of 1 and 2 $Lmin^{-1}$ and purification times of 5 and 20 min. Biogas is the gaseous result of digesting the chicken and cow manure in anaerobic conditions.

The composition of the red brick content based on XRF test consisted of 55.2 % SiO_2, 17.1 % Al_2O_3, 13.1 % CaO, 4.2 % MnO, 3.2% Na_2O, 3.14 % TiO_2, and 2.4 % BaO.

The following parameters were followed : M – adsorbent mass (g), Q – biogas flow rate ($Lmin^{-1}$) and t – purification time (min). The equipment used in this research are Gas Chromatography (GC) at Greenhouse Gas Laboratory (Balingtan, Pati, Center Java), rotap rocker as a sieve to adjust grain size, digital balance to measure the mass of adsorbent, tedlar bag to accommodate post-purification gas samples, flowmeter to regulate flow rate, stopwatch to measure purification time, polyurethane hose for biogas distribution lines, distribution tubes for storing pressurized biogas, purifying equipment and biogas compression for transferring biogas from digesters

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to distribution tubes. The percentage of CO_2 and CH_4 concentration per treatment after purification and the effectiveness of adsorption for each treatment.

Tools and materials were prepared according to Figure 1. Biogas flows through tubes (no. 6 and 7) which have been filled with adsorbents into the sample bag.

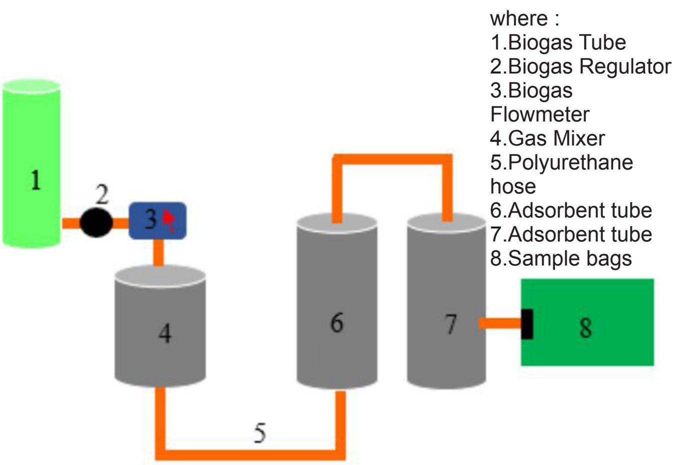

Figure 1: The scheme biogas purification equipment

Sampling done at time variations of 5 and 20 minutes, the sample bag was fully loaded (2 liters). Two repetitions were made for each variation. After the data obtained, an analysis was carried out using factorial design method 2^3 to determine the effect of each treatment and the interaction between treatments on the binding of CO_2 to biogas purification.

RESULTS AND DISCUSSION CO_2 CONCENTRATION

The percentage data of the results of CO_2 binding using red brick powder adsorbents obtained from Gas Chromatography (GC) are shown in Table 1.

Based on the data in Table 1 obtained a combination of factorial design data 2^3 as shown in Figure 2.

The use of factorial design 2^3 was carried out to determine the effect response of each adsorbent mass factor (M), flow rate (Q) and purification time (t) to the percentage CO_2 produced by biogas. High level (+) show high treatment value an low level (-1) shows low treatment value on each factor [15][16]. The matrix from in Figure 3 was obtained:

Table 1: 2^3 factorial design scheme and % CO_2 after biogas purification

Coding			M (g)	Q (L min^{-1})	t (min)	(%) CO_2	(%) CO_2	$\bar{y}$ (%) CO_2
-	-	-	200	1	5	17.25	17.13	17.189 (1)
+	-	-	400	1	5	10.32	11.84	11.082 a
-	+	-	200	2	5	20.44	21.84	21.141 c
+	+	-	400	2	5	12.67	10.18	11.422 ac
-	-	+	200	1	20	17.18	17.13	17.154b
+	-	+	400	1	20	10.33	10.31	10.319 ab
-	+	+	200	2	20	20.84	20.16	20.497 bc
+	+	+	400	2	20	10.28	11.73	11 abc
Without treatment						25.34		

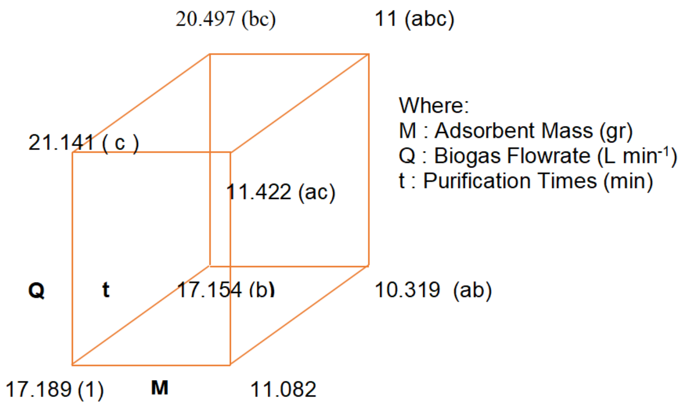

Figure 2: Combinatioan of CO_2 data on factorial design 2^3

With the Gauss Siedel iteration method, the completion of matrix on Figure 3 is obtained:

$$y = 14.976 - 4.0198M + 1.039Q - 0.233.t - 0.784.MQ - - 0.063.MT - 0.0335.Qt + 0.119.MQt \tag{1}$$

Where:

y: The percentage of CO_2 after biogas purification (%)

IM: The Mass Factor of Brick Powder Adsorbent (g)

IQ: The Biogas Flowrate Factor (L min^{-1})

It: The Purification Time Factor (min)

$$\begin{bmatrix} 1 & -1 & -1 & -1 & 1 & 1 & 1 & -1 \\ 1 & 1 & -1 & -1 & -1 & -1 & 1 & 1 \\ 1 & -1 & 1 & -1 & -1 & 1 & -1 & 1 \\ 1 & 1 & 1 & -1 & 1 & -1 & -1 & -1 \\ 1 & -1 & -1 & 1 & 1 & -1 & -1 & 1 \\ 1 & 1 & -1 & 1 & -1 & 1 & -1 & -1 \\ 1 & -1 & 1 & 1 & -1 & -1 & 1 & -1 \\ 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 \end{bmatrix} \begin{bmatrix} I0 \\ IM \\ IQ \\ It \\ IMQ \\ IMt \\ IQt \\ IMQt \end{bmatrix} = \begin{bmatrix} 17.189 \\ 11.082 \\ 21.141 \\ 11.422 \\ 17.154 \\ 10.319 \\ 20.497 \\ 11 \end{bmatrix}$$

Figure 3: Factorial Design 2^3 CO_2 Data Matrix

IMQ: The Interaction between Adsorbent Mass and Biogas Flowrate

IMt: The Interaction between Adsorbent Mass and Purification time

IQt: The Interaction between Biogas Flowrate and Purification time

IMQt: The Interaction between Adsorbent Mass, Biogas Flowrate, and Purification Time

Equation (1) explains that the greatest effect (≥ ± 0.5) occurs on the mass factor of adsorbent red brick powder, biogas flow rate, and mass interaction - flow rate on CO_2 levels. A negative value on the M coefficient means that there is an effect of increasing mass on CO_2 levels, CO_2 concentration on 200 grams of the adsorbent mass is higher than 400 grams of the adsorbent mass, which means that the use of the adsorbent mass is more effective with higher levels. Likewise, mass - flow rate interactions have an effect on decreasing CO_2 levels after purification even in small amounts. However, a positive value on the flow rate coefficient indicates that increasing the flow rate does not increase the binding of CO_2 so that the flow rate of 1 $Lmin^{-1}$ is better for binding carbon dioxide compared to 2 $Lmin^{-1}$. This will be proven in the analysis of each treatment variable.

Analysis of the effect of the adsorbent mass on CO_2 concentration in biogas with variable flow rates and time constant be seen in Figure 4.

Figure 4 shows that a mass of 200 grams has a higher CO_2 concentration than a mass of 400 grams. The increase in the mass of the adsorbent will reduce the value of CO_2 concentrations as a result of biogas purification. Increasing the mass of the adsorbent means that the volume also increases which will increase the area of the adsorbent. This will result in the increase of the contact area between the CO_2 gas and the adsorbent, so the CO_2 content will continue to decrease along with the increase in the mass of the adsorbent. These results are in line with previous studies [17], [18], [19], [20].

Analysis of the biogas flowrates effect on CO_2 concentrations in biogas with mass and time variables can be seen in Figure 5.

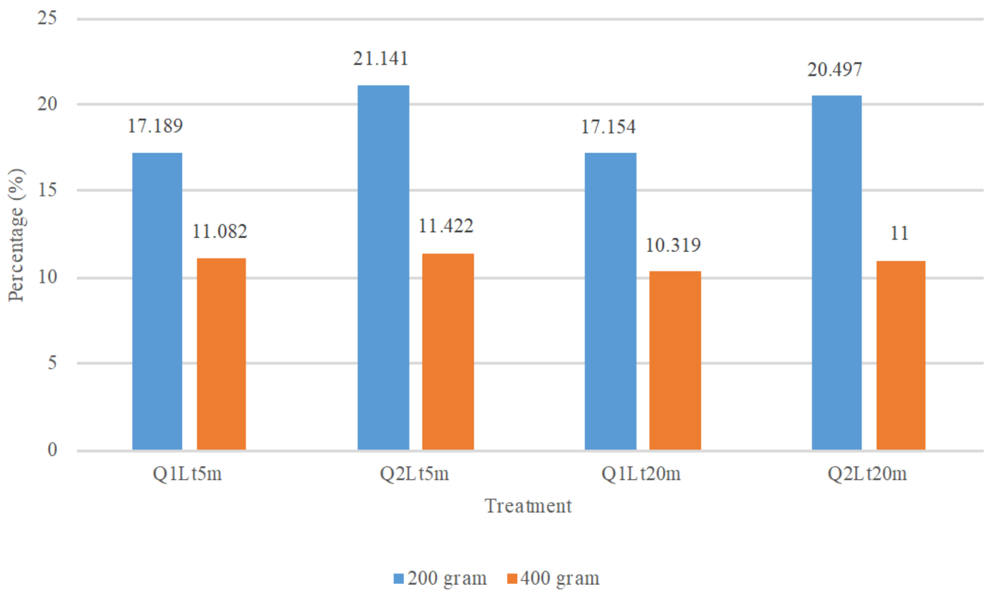

Figure 4: Adsorbent mass effect on CO_2 concentrations

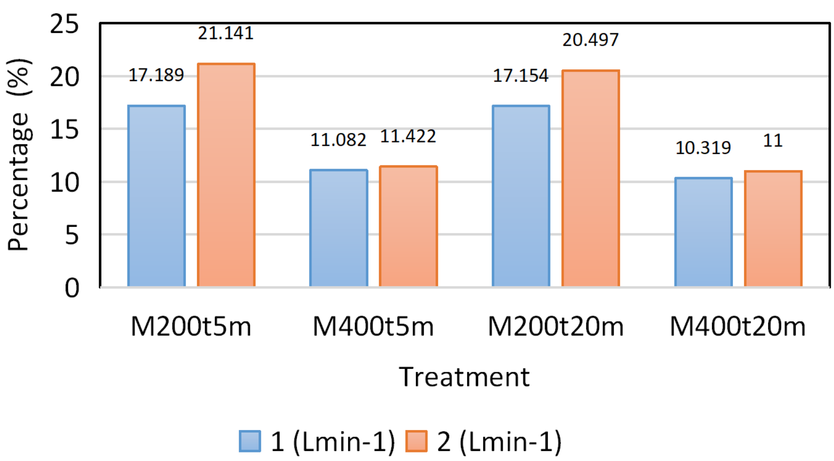

Figure 5: The diagram of biogas flowrates effect on CO_2 concentrations

Figure 5 shows that the flow rate of 2 $Lmin^{-1}$ has a higher CO_2 concentration than the flow rate of 1 $Lmin^{-1}$. These results are in accordance with previous studies [21], [22] that high flow rates can cause a lot of biogas to be wasted because it was not adsorbed beforehand. Significant differences in CO_2 concentrations between the flow rates 1 and 2 $Lmin^{-1}$ occur at a mass of 200 grams with a delta of around 3%. This happens because the adsorbent is in small amount but the flow rate is too fast so that a lot of biogas is wasted without being bounded into the pores of the adsorbent.

The calculation of the efficiency of CO_2 binding is done to find out the best treatment, using the formula [23]:

$$\eta = \left(1 - \frac{CO_2 out}{CO_2 in}\right) x100\% \quad (2)$$

where:

η: The Efficiency of CO_2 Adsorption (%)

$CO_{2,out}$: The CO_2 Concentration After Purification (%)

$CO_{2,in}$: The CO_2 Concentration Before Purification (%)

The results of calculating the effectivity of CO_2 adsorption are shown in Figure 6.

Figure 6, the highest effectivity occurred in the M400Q1Lt20m treatment at 59.28%. The lower the concentration of CO_2 after purification, the value of the effectivity of CO_2 increases [14], [24]. It happens because the interaction of the variables makes the contact area larger between the red brick powder adsorbent and CO_2 for the 400 gram mass variable, the binding of CO_2 with the red brick powder adsorbent is more evenly distributed and minimal biogas is wasted for variable flow rates of 1 $Lmin^{-1}$, and the binding of CO_2 is more maximal and evenly distributed before the saturated red brick adsorbent allows no biogas to be wasted for a 20 minute purification time variable. The interaction of these three variables makes the effectivity of the CO_2 adsorption with adsorbent red brick powder to be high.

CH_4 CONCENTRATION

Percentage data of post-purification CH_4 results using red brick powder adsorbents obtained from Gas Chromatography as shown in Table 2

Data from Table 2 is converted to a combination of data models as shown in Figure 7.

Table 2: 2^3 factorial design scheme and % CH_4 after biogas purification

Coding			M (g)	Q (L min^{-1})	t (min)	(%) CH_4	(%) CH_4	(%) $\bar{x}$
-	-	-	200	1	5	47.53	48.35	47.936 (1)
+	-	-	400	1	5	58.68	67.13	62.904 a
-	+	-	200	2	5	66.32	56.98	61.648 c
+	+	-	400	2	5	66.44	65.00	68.402 ac
-	-	+	200	1	20	69.09	45.10	57.096 b
+	-	+	400	1	20	67.42	66.44	66.929 ab
-	+	+	200	2	20	47.33	59.10	53.213 bc
+	+	+	400	2	20	69.04	67.76	65.717 abc
Without treatment						36.83		

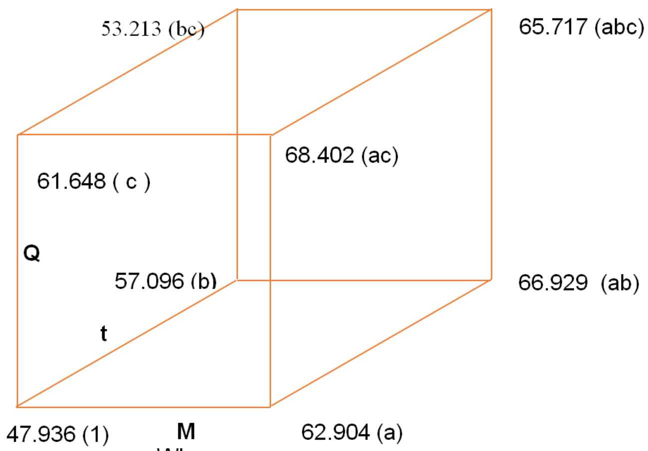

Figure 7: Combination of CH_4 Data on Factorial Design 2^3

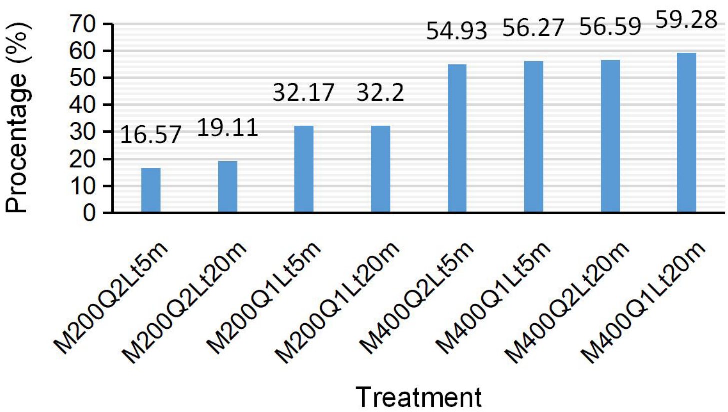

Figure 6: The Effectivity of CO_2 Adsorption

The use of factorial design 2^3 was carried out to determine the effect response of each adsorbent Mass (M), Flowrate (Q) and Purification Time (t) to the % CH_4 of biogas purification. High level (+1) shows high treatment value and low level (-1) shows low treatment value on each factor [15]. The matrix form is obtained in Figure 8;

Method of the Gauss Siedel iteration was used the completion of matrix on Figure 8 is obtained:

$$\begin{bmatrix} 1 & -1 & -1 & -1 & 1 & 1 & 1 & -1 \\ 1 & 1 & -1 & -1 & -1 & -1 & 1 & 1 \\ 1 & -1 & 1 & -1 & -1 & 1 & -1 & 1 \\ 1 & 1 & 1 & -1 & 1 & -1 & -1 & -1 \\ 1 & -1 & -1 & 1 & 1 & -1 & -1 & 1 \\ 1 & 1 & -1 & 1 & -1 & 1 & -1 & -1 \\ 1 & -1 & 1 & 1 & -1 & -1 & 1 & -1 \\ 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 \end{bmatrix} \begin{bmatrix} F0 \\ FM \\ FQ \\ Ft \\ FMQ \\ FMt \\ FQt \\ FMQt \end{bmatrix} = \begin{bmatrix} 47.936 \\ 62.904 \\ 61.648 \\ 68.402 \\ 57.096 \\ 66.929 \\ 53.213 \\ 65.717 \end{bmatrix}$$

Figure 8: Factorial Design 2^3 CH_4 Data Matrix

where:

x: The percentage of CH_4 after biogas purification (%)

FM: The Mass Factor of Brick Powder Adsorbent (g)

FQ: The Biogas Flowrate Factor (L min^{-1})

Ft: The Purification Time Factor (min)

FMQ: The Interaction between Adsorbent Mass and Biogas Flowrate

FMt: The Interaction between Adsorbent Mass and Purification time

FQt: The Interaction between Biogas Flowrate and Purification time

FMQt: The Interaction between Adsorbent Mass, Biogas Flowrate, and Purification time

Equation (3) explains that the greatest effect (≥0.5) occurs in the mass factor of the adsorbent red brick powder, biogas Flowrate, Mass interaction - Flowrate, Flowrate - Purification Time Interaction, and Mass - Flow rate - Purification Time Influence CH_4 levels. A positive value on the M coefficient means that there is an effect of increasing Mass on CH_4 levels [17], [19], [20]. Increasing the mass of the adsorbent will increase the percentage of CH_4 because a lot of CO_2 is bound to the adsorbent, thereby increasing the ratio between CH_4 and CO_2.

The Q value is positive, which means an increase in flow rate, increases the concentration of CH_4 after purification [25], [26]. This is because the addition of the flow rate to the red brick adsorbent containing a lot of silica-alumina will trap or bind CO_2 to the pores of the adsorbent due to van der Waals force and also the size of CO_2 which is larger than CH_4, at that time also smaller CH_4 will pass through the adsorbent without being bound by the adsorbent, so the faster the biogas flow rate, the more CH_4 levels are obtained.

The Mass Interaction Factor and the Flowrate (MQ) is negative, which means that there is an influence of interaction between variables on the decrease in CH_4 although it is relatively small. This happens because the Mass interaction and Flowrate are inversely proportional. Increased Mass will potentially increase CH_4 levels because CO_2 levels become reduced [17], [21], [22]. While an increase in Flowrate has the potential to reduce the ratio between CO_2 and CH_4 because although a lot of CH_4 is obtained, there is also a lot of CO_2 that has not been adsorbed so that the ratio decreases. [21], [22].

The interaction factor of Flowrate and Purification Time (Qt) is negative, which means there is an influence of interaction between variables on the decrease in CH_4, because an increase in Flowrate has the potential to reduce the ratio between CO_2 and CH_4 because although a lot of CH_4 is obtained, there is also a lot of CO_2 that has not been adsorbed so the ratio decreases [21], [22]. While an increase in purification time will make the CO_2 adsorption increase to the optimum point, then decrease thereby reducing the ratio of CO_2 and CH_4 levels [19], [27].

The adsorbent Mass Interaction Factor, biogas Flowrate, and Purification Time (MQt) are positive, which means there is an influence of interaction between variables on the increase in CH_4. This happens because each factor has its own influence, and the influence of Mass has the biggest role so that the interaction of MQt is positive so there is an increase in CH_4 in biogas purification.

CONCLUSION

Conclusions in this research, as follows:

1. The Mass factor of red brick powder adsorbent has the most influence in the results of biogas purification.
2. Red brick powder as an adsorbent in the biogas purification process has an influence on reducing CO_2 levels due to high SiO_2 and Al_2O_3 content which makes red brick a good adsorbent because more CO_2 is bounded due to van der Waals forces on the red brick surface.
3. Red brick that has a lot of CO_2 adsorption increases the ratio of CH_4 to CO_2.

ACKNOWLEDGMENTS

This research was funded by Faculty of Engineering, Brawijaya University, Malang, Indonesia with a contract agreement number: 19/UN10.F07/PN/2020

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Paper submitted: 30.07.2020.
Paper accepted: 11.09.2020.