Application of factorial design to analyse the effect of brick powder size on biogas purification results

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Abstract. This study aims to know the effect of differences in grain size variation in fine and coarse bricks in the biogas purification process by reducing carbon dioxide gas levels using the adsorption method, so as to obtain the most suitable size to produce the most optimal purification results. In this research, the biogas purification process uses brick powder as an adsorbent, utilizing the alumina and silica content in bricks to absorb carbon dioxide gas. Testing the purified gas content in biogas using Gas Chromatography - Mass Spectrometry (GCMS). The data processing method used in this study was a factorial design method $2^3$. The variations used in this study were the size of fine and coarse bricks, the purification time variation of 5 minutes and 20 minutes and the flow rate variation was 1 Liter minute$^{-1}$ and 2 Liter minute$^{-1}$. Fine size variation, a flow rate of 1 L Min$^{-1}$ and a purification time of 20 minutes give the best results with the percentage level of carbon dioxide falling reaching 10.319%.

Keywords: brick powder, biogas purification and CO$_2$ adsorption.

1. Introduction
Biogas is an alternative of problem in increasing energy needs. Biogas has advantages in sources that are easily available as well as simple installation tools. This energy is produced from animal waste through anaerobic process in about 7 days. This process produces a gas-shaped fuel containing methane (CH$_4$) of 55-75%, carbon dioxide (CO$_2$) of 25-45%, nitrogen (N$_2$) of 0-0.3%, hydrogen (H$_2$) of 1-5% hydrogen sulfide (H$_2$S) of 0-3% and oxygen (O$_2$) of 0.1-0.5% [1]. The purity of biogas can be seen from the higher percentage of methane gas because it causes the biogas calorific value to increase. But in addition to methane gas which can increase the calorific value there is carbon dioxide in the biogas content where the carbon dioxide (CO$_2$) content influences the combustion energy value [2]. Carbon dioxide (CO$_2$) is an inhibitory gas or impurities in combustion because the gas inhibits the collision reaction between hydrocarbons and air molecules. So that we need a solution to overcome these problems which can reduce the percentage of carbon dioxide to be able to increase the calorific value so that biogas with good quality is obtained, a solution to the problem that is purification.
Purification in biogas is a solution that aims to reduce carbon dioxide \( (\text{CO}_2) \) gas thus increasing the percentage of methane gas \( (\text{CH}_4) \). The biogas calorific value can be known from the carbon dioxide \( (\text{CO}_2) \) percentage which, the higher the percentage of methane gas \( (\text{CH}_4) \), causing the combustion process to be less optimal and the lower the percentage of carbon dioxide \( (\text{CO}_2) \) gas in the biogas, the higher the percentage of methane gas \( (\text{CH}_4) \) so as to improve the quality of biogas and the combustion process is more optimal.

Purification process can improve the quality of biogas with adsorbents that contain \( \text{SiO}_2 \) and \( \text{Al}_2\text{O}_3 \) because it is able to absorb \( \text{CO}_2 \) [3]. This study will further study the use of brick adsorbents containing \( \text{SiO}_2 \) and \( \text{Al}_2\text{O}_3 \) with the aim of obtaining the optimum grain size in the biogas purification process so as to reduce \( \text{CO}_2 \) gas content and increase the biogas calorific value. This research is expected that brick dust can be used as an easy and inexpensive solution for biogas management because it is able to be an alternative adsorbent for biogas for the wider community and especially people who have livestock manure waste and brick waste as a substitute for fossil fuels.

2. Research methods

In this research, red brick powder is used as an adsorbent with fine and coarse sizes. The biogas used was taken from cattle farmers in South Malang, namely from Mr. Sulis and Mr. Syamsi and the bricks used were from South Malang, after XRF testing, the composition of bricks in table 1.

| No | elements | Percentage | Major |
|----|----------|------------|-------|
| 1  | \( \text{SiO}_2 \) | 43.5%      | Si    |
| 2  | \( \text{Fe}_2\text{O}_3 \) | 28.74%     | Fe    |
| 3  | \( \text{Al}_2\text{O}_3 \) | 15%        | Al    |
| 4  | \( \text{CaO} \) | 8.85%      | Ca    |
| 5  | \( \text{TiO}_2 \) | 2.03%      | Ti    |
| 6  | \( \text{K}_2\text{O} \) | 0.61%      | K     |
| 7  | \( \text{MnO} \) | 0.51%      | Mn    |

The equipments used: rotap sifter and rotap rocker to obtain fine and coarse brick powder, digital balance, and flowmeter to regulate flow rate, stopwatch, and tedlar bag to collect gas samples. Gas Chromatography - Mass Spectrometry (GCMS) for analyzing gas purification. The gas identified by GCMS is the percentages of carbon dioxide \( (\text{CO}_2) \) and methane \( (\text{CH}_4) \) gases.

Variations in this study are fine and coarse brick powder adsorbents, flow rates of 1 L Minute\(^{-1}\) and 2 L min\(^{-1}\), with a mass of 400 grams. The research method used in this research is an experimental research method. This research is conducted at the Metal Casting Laboratory and Motor Fuel Laboratory Laboratory of the Engineering Faculty, Brawijaya University of Malang, Central Mineral & Advanced Materials Laboratory, Faculty of Mathematics and Natural Sciences, State University of Malang, Greenhouse Gas Laboratory, Research Institute for Agricultural Environment, Pati, and Middle Java.
After preparation of the installation and materials as shown in Figure 1, the data collection starts from the APK biogas tube (no. 1). Opening the valve (no. 2) on the APK tube, biogas flows out through a hose that has been connected to the flowmeter (no. 3) with the openings that have been set on the flowmeter ie discharge 1 and 2 Liters minute\(^{-1}\). At the installation of the purification tube (no. 4 and 5) has been filled with red brick powder with a variation of coarse size of 400 grams and then close the lid so that no gas is leaked.

The process of extracting the purified gas using a time variation of 5 and 20 minutes then the sample is put in a sample bag (7). After the sample for each variation in size, flow rate, and time have been repeated, repeat the step by first closing the valve in the Biogas APK tube then replacing the brick powder in the purification tube with different variations at a mass of 400 grams. Gas Chromatography - Mass Spectrometry (GCMS) will then analyze the results of purification where 5 ml will be taken in each gas bag that has been taken to be injected into a GCMS device which is seen the percentage of gas content in the sample.

### Table 2. Percentage of CO\(_2\) and CH\(_4\) gas after purifying

| No | Size  | Q (L min\(^{-1}\)) | t (min) | CH\(_4\) (%) | CH\(_4\) (%) | ̅ (±) | CO\(_2\) (%) | CO\(_2\) (%) | (±)  |
|----|-------|-------------------|--------|-------------|-------------|------|-------------|-------------|------|
| 1  | Course| 1                 | 5      | 48.36       | 46.04       | 47.203 | 12.71       | 16.36       | 14.538 |
| 2  | Fine  | 1                 | 5      | 58.68       | 67.13       | 62.904 | 10.32       | 11.84       | 11.082 |
| 3  | Course| 2                 | 5      | 66.09       | 50.10       | 58.095 | 20.35       | 24.57       | 22.457 |
| 4  | Fine  | 2                 | 5      | 66.44       | 65.00       | 65.717 | 12.67       | 10.18       | 11.422 |
| 5  | Course| 1                 | 20     | 55.88       | 55.71       | 55.797 | 20.64       | 21.76       | 21.199 |
| 6  | Fine  | 1                 | 20     | 67.42       | 66.44       | 66.929 | 10.33       | 10.31       | 10.319 |
| 7  | Course| 2                 | 20     | 65.30       | 57.64       | 61.471 | 14.36       | 17.36       | 15.860 |
| 8  | Fine  | 2                 | 20     | 69.04       | 67.76       | 68.402 | 10.28       | 11.73       | 11.00  |
|    | Untreatment |           |       | 36.83       | 25.34       |      |             |             |      |
The following is the percentage of carbon dioxide (CO2) absorption and the percentage of methane (CH4) gas after purification using brick powder adsorbents obtained from Gas Chromatography - Mass Spectrometry (GCMS), shown in Table 2. The carbon dioxide (CO2) data in Table 2 is processed into a factorial design scheme where this design can be used to investigate the parameters involved in the purification process to increase the percentage of methane gas [4], following the factorial design scheme in Table 3.

Table 3. Scheme of factorial design 2$^3$ of CO$_2$ after biogas purification

| No | Coding | Size | Q (L minute$^{-1}$) | t (minute) | (%) CO$_2$ | (%) CO$_2$ | $\bar{y}$ |
|----|--------|------|-------------------|-----------|------------|------------|----------|
| 1  | - - -  | Course | 1                 | 5         | 12.71      | 16.36      | 14.538 (1) |
| 2  | + - -  | Fine   | 1                 | 5         | 10.32      | 11.84      | 11.082 a  |
| 3  | - + -  | Course | 2                 | 5         | 20.35      | 24.57      | 22.457 c  |
| 4  | + + -  | Fine   | 2                 | 5         | 12.67      | 10.18      | 11.422 ac |
| 5  | - - +  | Course | 1                 | 20        | 20.64      | 21.76      | 21.199 b  |
| 6  | + - +  | Fine   | 1                 | 20        | 10.33      | 10.31      | 10.319 b  |
| 7  | - + +  | Course | 2                 | 20        | 14.36      | 17.36      | 15.860 ab |
| 8  | + + +  | Fine   | 2                 | 20        | 10.28      | 11.73      | 11.00 abc |

Note: Data from Table 3 is then made into a combination of cubes as shown in Figure 2.

Figure 2. Data combination in factorial design 2$^3$

Figure 3. Factorial design matrix 2$^3$
The factorial design $2^3$ functions to show the effect response of each factor, Size (U), Flow rate (Q) and purification time (t) to the percentage of carbon dioxide (CO$_2$) purified by biogas, to obtain the following matrix form. Through the Gauss Seidel iteration method, the following matrix is obtained:

$$y = 14.734 – 3.7789U + 0.4503Q – 0.1401t – 0.1948UQ – 0.156Ut – 1.6145Qt + 1.7UQt$$

(1)

Information:
- $y$: Percentage of CO$_2$ Biogas Purification
- M: Adsorbent Size Factor
- Q: Biogas Flow Rate Factor (L min$^{-1}$)
- t: Purification Time Factor (minutes)
- UQ: Interaction of Adsorbent Size and Biogas Flow Rate
- Ut: Interaction of Adsorbent Size and Purification Time
- Qt: Interaction of Flow Rate and Purification Time
- UQt: Interaction of Adsorbent Size, Flow Rate, and Purification Time

Equation 1 can be seen that the Q value does not reach 1 or -1 explains that in this study the flow rate factor has a small effect on the absorption of carbon dioxide, this is because the difference between the flow rate variables used is quite small 1L min$^{-1}$. U value explains that in this study the size factor of adsorbent powder has a fairly large influence on the absorption of carbon dioxide because it has a value 3.7. The value of t explains that in this study the duration of the purification process has a small effect on the absorption of carbon dioxide because it does not reach 1 or -1. UQ value is the value of the interaction factor between the type of size of the adsorbent and the flow rate and Ut is the value of the interaction factor between the purification time and the size of the adsorbent, both of these factors have little effect on the absorption of carbon dioxide gas because it has a value that does not reach 1 or -1. Qt is the value of the interaction factor between discharge and purification time, this interaction factor has an influence on the process of carbon dioxide absorption because it has a value that reaches -1. UQt is the value of the interaction factor between the type of adsorbent size, flow rate, and purification time explained that in this study the interaction of these three factors has a significant effect because it
has a value that reaches 1. To find out the percentage of responses from the factors that influence carbon dioxide (CO$_2$) absorption in the biogas purification process can be seen through the diagram shown in Figure 4.

The decrease in the percentage of carbon dioxide gas (CO$_2$) is influenced by the ability of adsorption by factors of type of adsorbent, amount of adsorbent, size of adsorbent [5], type of size of adsorbent [6]. The smaller the size of the adsorbent, the more surface area of the adsorber that comes in contact with the adsorbate so that the adsorption value will also increase [7,8]. This is because bricks have alumina silicate compounds. Bricks are porous so they have the ability to adsorb so they can absorb carbon dioxide gas to the fullest.

Furthermore, to determine the effectiveness of carbon dioxide gas absorption which can be seen through the diagram shown in Figure 5.

![Figure 5. Effectiveness of CO$_2$ gas absorption](image)

The effectiveness in this study shows how much the ability of adsorbents to absorb CO$_2$ in the biogas purification process. The use of brick powder as an adsorbent in the purification process has the effectiveness of reducing CO$_2$ levels up to 59.28%. Figure 5 shows that the effect of contact surface area on the ability to absorb carbon dioxide explains the effectiveness of adsorbs [9]. The methane gas (CH$_4$) data in table 2 is then processed into a factorial design scheme as in table 4.

**Table 4. Scheme of factorial design $2^3$ and % CH$_4$ After Biogas Purification**

| No  | Coding | Size | Q (L min$^{-1}$) | t (minute) | CH$_4$ (%) | CH$_4$ (%) | $\bar{x}$ (%) |
|-----|--------|------|-----------------|-----------|------------|------------|---------------|
| 1   | - - -  | Course | 1              | 5         | 48.36      | 46.04      | 47.203 (1)    |
| 2   | + - -  | Fine  | 1              | 5         | 58.68      | 67.13      | 62.904 a      |
| 3   | - + -  | Course | 2              | 5         | 66.09      | 50.10      | 58.095 c      |
| 4   | + + -  | Fine  | 2              | 5         | 66.44      | 65.00      | 65.717 ac     |
| 5   | - - +  | Course | 1              | 20        | 55.88      | 55.71      | 55.797 b      |
| 6   | + - +  | Fine  | 1              | 20        | 67.42      | 66.44      | 66.929 b      |
| 7   | - + +  | Course | 2              | 20        | 65.30      | 57.64      | 61.471 bc     |
| 8   | + + +  | Fine  | 2              | 20        | 69.04      | 67.76      | 68.402 abc    |

The effectiveness in this study shows how much the ability of adsorbents to absorb CO$_2$ in the biogas purification process. The use of brick powder as an adsorbent in the purification process has the effectiveness of reducing CO$_2$ levels up to 59.28%. Figure 5 shows that the effect of contact surface area on the ability to absorb carbon dioxide explains the effectiveness of adsorbs [9]. The methane gas (CH$_4$) data in table 2 is then processed into a factorial design scheme as in table 4.
Data from Table 3 is made into a combination of cubes as shown in Figure 6. Figure 6 is a factorial design $2^3$ of methane gas that serves to determine the interaction between Size (U), Flow rate (Q) and purification time (t) to the percentage of methane gas (CH$_4$) resulting from biogas purification, so that the matrix form is obtained as follows:

$$
\begin{bmatrix}
1 & -1 & -1 & 1 & 1 & 1 & -1 \\
1 & 1 & -1 & -1 & -1 & 1 & 1 \\
1 & 1 & 1 & 1 & -1 & -1 & -1 \\
1 & -1 & -1 & -1 & 1 & -1 & 1 \\
1 & 1 & -1 & 1 & -1 & -1 & 1 \\
1 & -1 & 1 & 1 & -1 & 1 & -1 \\
1 & 1 & 1 & 1 & 1 & 1 & 1
\end{bmatrix}
\begin{bmatrix}
X0 \\
XU \\
XQ \\
Xt \\
XUQ \\
XUt \\
XQt
\end{bmatrix} =
\begin{bmatrix}
14.538 \\
11.082 \\
22.457 \\
11.422 \\
21.199 \\
10.319 \\
15.860 \\
11.00
\end{bmatrix}
$$

Figure 7. Design factorial matrix $2^3$

Through the Gauss Seidel iteration method, the following matrix is obtained:

$y = 60,8116 + 5,1733.U + 2,6064.Q + 2,3352.t - 1,535.UQ - 0,6575.Ut - 0,8199Qt + 0,4847.UQt$ (2)

Information:
y: Percentage of CO$_2$ Biogas Purification
M: Adsorbent Size Factor
Q: Biogas Flow Rate Factor (L min$^{-1}$)
t: Purification Time Factor (minutes)
UQ: Interaction of Adsorbent Size and Biogas Flow Rate
Ut: Interaction of Adsorbent Size and Purification Time
Qt: Interaction of Biogas Flow Rate and Purification Time
UQt: Interaction of Adsorbent Size, Biogas Flow Rate, and Purification Time
Equation 2 explains that the positive value of Q explains that in this study the flow rate factor has a small effect on the increase in the percentage of methane gas, this is because the difference between the flow rate variables used is quite small namely 1 L min\(^{-1}\). U has negative value explains that in this study the size factor of adsorbent powder has a fairly large influence on the increase in the percentage of methane gas because it has a large minus value. t has positive value explains that in this study the duration of the purification process has a small effect on the absorption of carbon dioxide because it has a positive value. UQ value is the value of the interaction factor between the type of adsorbent size and flow rate, Qt is the value of the interaction factor between...
discharge and purification time, Ut is the value of the interaction factor between purification time and the adsorbent size, these three interaction factors have an influence on the increase in the percentage of methane. UQt is the value of the interaction factor between the type of adsorbent and the adsorbent size, these three interaction factors have an influence on the increase in the quality of biogas because it has a higher heating value obtained from the higher value of methane gas, which causes better combustion [10].

Furthermore, to find out the percentage of methane gas, it can be seen through the diagram shown in figure 8. The decrease in the percentage of carbon dioxide (CO₂) causes an increase of the percentage of methane (CH₄). The less percentage of carbon dioxide (CO₂) gas, the higher the quality of biogas because it has a higher heating value obtained from the higher value of methane gas, which causes better combustion [10].

The biogas calor value is obtained from the percentage of methane gas (CH₄) compared to the percentage of total biogas that is 100% multiplied by the calorific value (CH₄) multiplied by the density of methane gas (CH₄). Variation of the flow rate of 2 L min⁻¹, fine powder size, and purification time of 20 minutes have the highest heating value of 22910.7 kJ m⁻³.

4. Conclusion
The conclusions that have been made in this study are as follows:

- Fine size can reduce CO₂ percentage better than coarse size because the smaller the grain size with the same mass, the more the number of grains so that the surface area of contact with the adsorbate increases, the better the adsorption means the better the adsorption so that the quality of the biogas will increase.
- The lowest percentage of CO₂ gas is in the variation of fine size, flow rate of 1 L minute⁻¹, and 20 minutes of purification time is 10.319% with the highest effective CO₂ absorption value is 59.28%.

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