

Purification of biogas from tofu waste using zeolite and activated carbon (AC) as adsorbent

Budihardjo M.A.^{1*}, Syafrudin¹, Samadikun B.P.¹, Wardhana I.W.¹, Ramadan B.S.² and Humaira N.G.²

¹Department of Environmental Engineering, Faculty of Engineering, Universitas Diponegoro, Semarang-50275, Indonesia

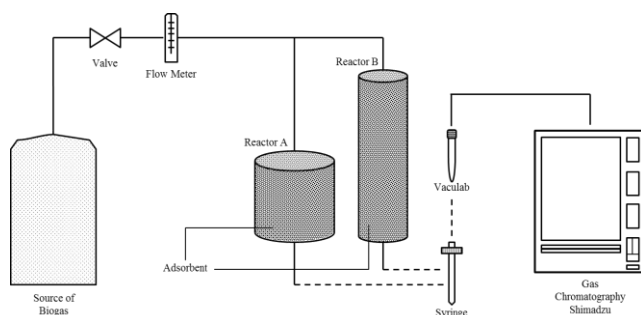
²Environmental Sustainability Research Group, Department of Environmental Engineering, Faculty of Engineering, Universitas Diponegoro, Semarang-50275, Indonesia

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*to whom all correspondence should be addressed: e-mail: m.budihardjo@ft.undip.ac.id

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Graphical abstract



Abstract

Substitution of fossil fuel into alternative energy sources, such as biogas, needs to be applied to prevent various problems in the environment. Biogas purification can be an alternative technology to improve the quality of biogas, which increases the heat value by reducing CO₂. Purification can be carried out by the adsorption method using solid adsorbents, for example natural zeolite and activated carbon. This study used 12 reactors from PVC, with 2 different diameters, 3 different mesh size (20,50,80) and was given both zeolite (A1-A3; B1-B3) and activated carbon (A4-A6; B4-B6). Biogas purification with the addition of zeolite has an optimal compound content in reactor A3 which has an increase in CH₄ (63.63%) and decrease in CO₂ composition (25.07%), which is better than other reactors. Meanwhile, the results of measurements of gas content on the addition of activated carbon showed optimal performance in reactor A6, which was increased to 76.86% for CH₄ levels and CO₂ levels dropped to 19.79%.

Keywords: Adsorption, activated carbon, biogas composition, biogas purification, zeolite.

1. Introduction

The increase in CO₂ gas emissions in 1970-2004 was recorded at 80%, with the amount of CO₂ emitted by 35.65 million metric tons in 2011 (Boden *et al.*, 2015;

Stern and Stern, 2007). The steps to reduce CO₂ emissions are very important to reduce the risk of global warming. Governments from various countries have taken concrete steps to achieve these goals, including the creation of the Kyoto Protocol at the 2012 and 2015 COP 21 Conference in Paris (Ferella *et al.*, 2017; Townsend, 2013). On the other hand, a lot of research has been done as an effort to mitigate global warming and safeguard the availability of fossil fuels, one of which is the use of biogas as an alternative to fossil fuels (Meyer-Aurich *et al.*, 2012; Shen *et al.*, 2018; Yousef *et al.*, 2018).

Biogas is a product generated by anaerobic digestion from a variety of biodegradable materials, such as agricultural waste, animal waste, waste in human activities, and industrial waste (Aghbashlo *et al.*, 2019; Zain *et al.*, 2018). One of the industrial wastes that have the potential to produce biogas is the waste from the tofu industry. The tofu industry in Indonesia produces waste in the form of solid and liquid waste, which is 1.024 million m³ per year (solid waste) and 20 million m³ per year (liquid waste) (Faisal *et al.*, 2016). The number of organic compounds contained in it can produce biogas which can be recovered due to its anaerobic treatment. Biogas that is produced by 1 kg of tofu waste contains (Lay *et al.*, 2013).

Biogas usually has a CH₄ content of 55-65%; CO₂ of 35-45%; and a small portion of traces consisting of ammonia (NH₃), nitrogen (N₂), oxygen (O₂), carbon monoxide (CO), and hydrogen sulfide (H₂S), with concentrations of less than 1% (Abdeshahian *et al.*, 2016; Belaissaoui *et al.*, 2018; Remy *et al.*, 2014). Biogas can be used as fuel for vehicles and sources to produce heat and electricity, with the advantage of being a versatile, clean, and inexpensive fuel (Aghbashlo *et al.*, 2019; Tabatabaei *et al.*, 2019). However, the CO₂ contained in biogas becomes a contaminant that can reduce the value of heat, is corrosive, and damages the pipeline from biogas (Ahmad *et al.*, 2017; Duran *et al.*, 2018; Jiang *et al.*, 2018a). Therefore, removal of these contaminants is needed to obtain greater methane content in biogas, where the heating value of biogas with 60% (v/v) methane content reaches 5,000-6,000 kcal/N.m³, while higher methane

content (96-97%) can increase the heating value up to 8,000 kcal/n.m³ (Ryckebosch *et al.*, 2011).

Various methods of CO₂ removal contained in biogas from different activities are available. Adsorption, absorption, membrane separation, and cryogenic distillation are the most common method (Pettersson and WeLLInGer, 2009; Rufford *et al.*, 2012; ; Songolzadeh *et al.*, 2014). Adsorption is a technology that has advantages in economic aspects, both cost and energy use, but has high efficiency (Duran *et al.*, 2018; Zhou *et al.*, 2017). Zeolites, activated carbons, and metal-organic framework (MOF) are microporous materials that are often used in the adsorption process because of their ability to remove CO₂ (Hedin *et al.*, 2013; Li *et al.*, 2013; Möller *et al.*, 2017).

Table 1 Composition of Biogas (%v/v) CH₄ and CO₂

Reactor	CH ₄ (%v/v)					CO ₂ (%v/v)				
	0 min	30 min	60 min	90 min	120 min	0 min	30 min	60 min	90 min	120 min
A0	0	43.90	43.90	43.90	43.90	0	37.30	43.90	43.90	43.90
B0	0	44.60	136,587	142,977	148,720	0	44.60	136,587	142,977	148,720

Table 2 Adsorbents Properties

Reactor	Dimension	Adsorbent (mesh)	
		Zeolite	Activated Carbon
A1	2 inch 80 cm	20	-
A2		50	-
A3		80	-
A4	2 inch 80 cm	-	20
A5		-	50
A6		-	80
B1	4 inch 40 cm	20	-
B2		50	-
B3		80	-
B4	4 inch 40 cm	-	20
B5		-	50
B6		-	80

In addition to zeolite, activated carbon is also widely used in CO₂ removal due to the high adsorption capacity of ambient air, low regeneration cost, long-term stability, and fast kinetic. The material needed to produce activated carbon has an economical price and is relatively easy to obtain because it generally comes from the rest of agriculture and forests. This material is usually found in the form of wood, starch, coconut shells, or empty fruit bunch (EFB) (Lee *et al.*, 2013; Li *et al.*, 2018; Vilella *et al.*, 2017). Activation of these materials can be done both physically and chemically. In contrast to zeolites, activated carbon has a better tolerance to water because it has low affinity and it is a hydrophilic material so that it is able to maintain its adsorption capacity in high humidity (González *et al.*, 2013). However, activated also has a disadvantage because it has a low CO₂ selectivity compared to CH₄ (Bacsik *et al.*, 2016).

The main objective of this study is to determine the trend of using filters with zeolites and activated carbons to purify biogas, eliminate contaminants in the form of CO₂, and obtain more CH₄ content from the tofu waste treatment. In addition, according to Ogunwande (2017), the reactor surface area is significantly affected by biogas

Zeolites are three-dimensional crystalline aluminum silicates, derived from alkali and alkaline earth cations (Turan and Ergun, 2009). Zeolites widely used as adsorbents because of their removal ability for various chemicals, high thermal and chemical stability, and their uniqueness in carrying out molecular sifting (Ferella *et al.*, 2017; Loureiro and Kartel, 2006; Zhou *et al.*, 2017). Zeolites can increase removal of CO₂ because of their higher selectivity than CH₄. However, zeolite's affinity for water is very high, where the water contained in biogas reaches 6% of its volume (Bacsik *et al.*, 2016; Ferella *et al.*, 2017). Therefore, it is recommended to reduce the water content that will pass through the adsorbent to improve the CO₂ removal performance.

production, so this study used two-reactor with different surface areas to determine the relationship between reactor dimensions and biogas purification performance.

2. Materials and methods

2.1. Material characteristics

Biogas generated from waste treatment of tofu industry was used in this study. The biogas was obtained from waste processing of Wismilak Tofu Industry, Mangkang, Semarang City. Before the study began, a preliminary test of raw biogas generation was conducted using 2 PVC pipe with 2-inch and 4-inch diameter. The test determined the initial composition of methane (CH₄) and carbon dioxide (CO₂) from the raw biogas, which are shown in Table 1. Biogas purification was carried out by the adsorption method using adsorbents (solids), which are natural zeolite and activated carbon. Purification process of biogas from tofu waste using both adsorbents then started to investigate the optimum condition that can improve the biogas quality.

In this study, natural zeolite is used because in addition to being easily obtained, the market price is quite cheap, and

natural zeolite is reactive to CO₂. Natural zeolite serves to increase CH₄ levels and eliminate CO₂ levels in the biogas purification process (Jiang *et al.*, 2018b). The use of natural zeolite as an adsorbent requires an activation process to enhance the special nature of zeolite and remove impurities. Activation of natural zeolite can be carried out by chemical and physical methods with the aim of cleaning pore surfaces, removing impurities and rearranging the exchanged atoms. Chemical activation is carried out through immersion using a NaOH solution with a concentration of 5% for 24 hours, previously it is necessary to wash zeolite using distilled water (Nikolov *et al.*, 2017). Afterwards, zeolite is washed with distilled water until it is neutral and then dried. After drying, natural zeolite is physically activated by heating at 250°C for 2 hours in a furnace (Wibowo *et al.*, 2017).

In addition to natural zeolite, activated carbon can also be used to carry out biogas purification. Activated carbon is a material with high porosity that has a large adsorption capacity and is widely used as an adsorbent for purification (Kim and Pui, 2015). Activated carbon can adsorb certain gases and chemical compounds where the adsorption properties depend on the size or volume of pores and surface area (Sethia and Sayari, 2015). The detailed adsorbent properties is shown in Table 2.

2.2. Biogas purification reactor

There were 12 reactors that were made from PVC pipes with 2 variations in size, which were 2-inch tubes with 80 cm height (A1-A6) and 4-inch tubes with 40 cm height (B1-B6). All of these reactors were planned to have the same volume with a different diameter that determined from the results of preliminary study. Figure 1 illustrates the series of measurement tools and test positional schemes in this study. Both zeolite and activated carbon will be inserted in the reactor with 3 different mesh (20, 50, and 80), with the weight of 300 grams. The mesh size was specified to obtain the most suitable dimension of adsorbent during the biogas purification process. The higher the mesh size the smaller the screen opening and the smaller the particle that will pass through.

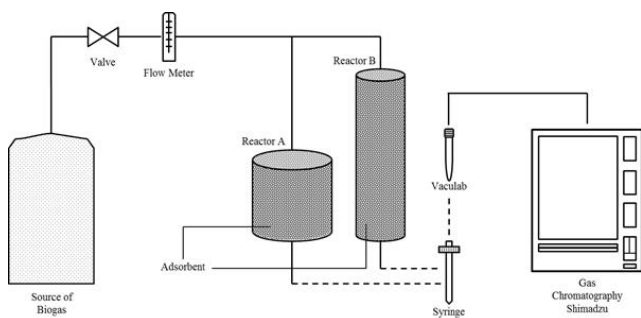


Figure 1. Scheme of biogas purification.

2.3. Measurement test

On biogas purification test, the collected gas flowed from the source into the reactor using a hose and tap with flow meter installed to control the biogas flow rate. The flow rate was maintained in 10 liter/min, which adjusted from the flow of gas generation at tofu waste treatment. The addition of adsorbent, zeolite and activated carbon, was

carried out by inserting it into the biogas purification tube of tofu waste through the reactor insulator for the adsorption process to the outlet. The time spent on each purification variation was 3 hours each. Samples of biogas from all reactors were collected every 30 minutes using an injection (syringe) and stored in a vaculab or vacuum tube. The measurement of CH₄ and CO₂ contents was conducted using Gas Chromatography Shimadzu-14 A (Liu *et al.*, 2015b), which is able to detect gaseous components in the form of methane, carbon dioxide and nitrogen based on ASTM D1945-14 (International, 2014), at Pati and Research Center Laboratory of Pati Regency.

3. Results

3.1. CO₂ reduction

CO₂ content in the biogas decreases until the end of the study. Figure 2a showed the reduction of CO₂ in reactor A. Reactor A1 had the highest CO₂ content of 32.00% in the 30th minute, while A3 had the lowest CO₂ content of 29.90%. Significant reduction occurred in the A3 reactor with final content of 25.07% in the 120th minute. This was lower than the other two reactors, A1 and A2, which are at 27.08% and 26.37% respectively. Meanwhile, in the activated carbon reactor (A4), CO₂ content decreased gradually from 24.37% to 21.61% in the end of the study. Reactor A5 experienced a fall until 120th minute, which decreased from 23.91% to 20,38%. Reactor A6 also decreased significantly starting from 60 minutes with 19.79% in the final composition, which also showed that it has the lowest CO₂ composition in the end of the study.

CO₂ composition in reactor B (Figure 2b) shows the same pattern with those in reactor A. Reactor B1 had the highest CO₂ content of zeolite reactor in the 120th minute with 29.75% while reactor B3 had the lowest CO₂ value of 27.69%. In reactor B4, CO₂ was eliminated from 28.96% in the first 30 minutes into 24.64% in the final content of biogas. On the other hand, Reactor B6 showed the most decrease compared to other activated carbon reactors, which fell from 26.67% to 22.19%. These results showed that activated carbon could decrease carbon dioxide composition of biogas more than zeolite in both reactor A and B. All of reactors that added with adsorbent had significant improvement of CO₂ composition, which was reduced until less than 30% v/v.

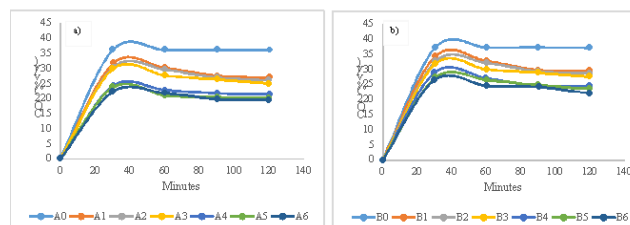


Figure 2. (a) Changes of carbon dioxide in reactor a, (b) changes of carbon dioxide in reactor B.

3.2. CH₄ generation

In this biogas purification test, it is known that the composition of CH₄ gases subsequently increased until it reached the highest content in the 120th minute. Figure 3a

and 3b shows the level of CH₄ in reactor A and B during the study. The final composition of CH₄ in A1 – A3 was ranged between 61% – 64%. The early CH₄ value in A1 reactor is the lowest, which was 55.70% in the 30th minute, while the CH₄ content in A3 reactor is the highest value (58.90%). Reactor A3 produced the highest methane after 2 hours generation, which was 63.63% and the lowest was reactor A1 with CH₄ content of 61.30%.

On the other hand, CH₄ in reactor A4 – A6 was ranged from 71% to 77%. Reactor A6 increased significantly compared to A4 and A5, which reached 76.86% methane content in the 120th minutes, which was also higher than A3. Meanwhile, A4 had the lowest methane content with 73.86% methane in the end of the study.

Reactor B3 had the highest CH₄ level in the 30th minute of 57.61% while the reactor B1 is the lowest with a value of 54.51%. The CH₄ level in the 120th minute on the reactor B3 became the highest with a value of 61.96% and B1 is the lowest with a value of 59.72%. Reactor B using activated carbon had higher CH₄ content compared to zeolite. Reactor B6 experienced greater increment than the other activated carbon reactor with the final methane content of 71.27% while B4 had about 5% less methane content, which is the least amount in the other two.

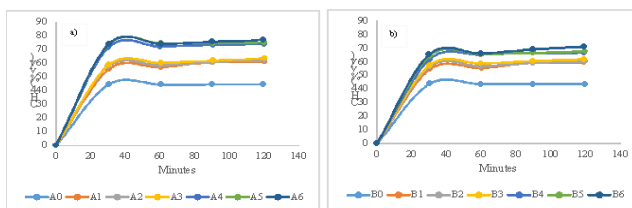


Figure 3. (a) Changes of Methane gas in reactor A, (b) changes of methane gas in reactor B.

4. Discussion

Compared to the preliminary study results, both zeolite and activated carbon reactor had better biogas composition. This means the addition of both adsorbents increases CH₄ and reduces CO₂ content. Although zeolite has higher CO₂-CH₄ selectivity, activated carbon improved the biogas composition better than zeolite, which was shown in higher methane and lower carbon dioxide.

CH₄ is an inert material towards the adsorption on activated carbon (Liu *et al.*, 2015a). Inactosite methane molecules are completely neutral (neither acidic nor basic). Methane has a regular tetrahedral structure, which favors incompletely non-polar structure. In contrary, CO₂ is acidic and polar, which makes chemical reactions in basic interaction with activated carbon. Based on Mamun (Al Mamun *et al.*, 2016), activated carbon, which has high porosity, is able to adsorb carbon dioxide and separate it from a certain gas mixture. The use of activated carbon is more beneficial because it can in generation while zeolite does. (Hauchhum and Mahanta, 2014). CO₂ can form a permanent polarity because there are transformations of CO₂ molecules that perform stretching motion of asymmetric bond at normal pressure and temperature. The van der Waals force in CO₂ molecule and the

framework of carbon determine the adsorption of CO₂ takes place, which is between single or multilayer of activated carbon. Moreover, the adsorption of CO₂ performed differently based on the level of interaction between quadrupole of CO₂, which is higher than CH₄, and gradient of electric field. This interaction is determined by the ratio of Si/Al in the adsorbent (Jiang *et al.*, 2018a; Li *et al.*, 2013).

Fernández (Juárez *et al.*, 2018) has conducted research about purification of biogas using biomass ash. The purified methane gas increased significantly more than crude CH₄. It also applied on purified CO₂ and crude CO₂. The reaction of CO₂ occurred rapidly because of high alkalinity and the ash humidity. CO₂ broke through the filter in several hours after most of alkaline mineral and reactive hydroxides were consumed. Durán *et al.* (Duran *et al.*, 2018), who used activated carbon from pine sawdust, stated that CH₄ broke faster than CO₂ but CH₄ will be replaced later because of roll-up effect. The gas mixture affected the adsorptive performance of the activated carbon due to the decrease of partial pressure when mixing components.

Besides the type of adsorbent, the mesh size also affects the performance of the biogas purification process. It can be seen that the reactor with the size of 80 mesh has the lowest CO₂ content compared to other mesh sizes. This is because the smallest adsorbent size is at 80 mesh size, which causes a larger contact area and increases the adsorption rate. The finer adsorbent can absorb more CO₂ than the coarser one because of the smaller fraction size, thus increasing the adsorption process. (Jafari *et al.*, 2017; Mastalerz *et al.*, 2017). Furthermore, gas adsorption capacity is strongly related to the specific surface area and micropore volume (Lee and Park, 2011). Usually, gas molecules in the gas-solid interaction (adsorption) potential are driven by strong van der Waals forces. In particular, the gas adsorption potential is highest in the sub-nanometer pores as they begin to overlap with decreasing pore size, leading to much higher binding energy come from the creation of deep potential wells (Romanos *et al.*, 2011). Therefore, it is crucial to maximizing the number of sub-nanometer pores by increasing the specific surface area.

In this study, reactors A and B have the same volume but have different results. The composition of biogas in reactor A showed a better improvement than reactor B. Reactor A produced less CO₂ and more CH₄ content than reactor B. It happens because the diameter/surface area in reactor A is wider than in reactor B. According to Ogunwande (2017) study, the reactor surface area is significantly affected by biogas production. Biogas yield increases when the surface area increases. Furthermore, specific surface area, porosity, surface roughness, pore size, and orientation of the packing material were found to play an important role in anaerobic reactor performance (Singh and Prerna, 2009).

5. Conclusion

This study discusses the effect of adding size variations of adsorbents-zeolite and activated carbon-on biogas

purification. Both of these materials have a variety of advantages, such as high adsorption that can increase CH₄ levels and eliminate CO₂ levels in the biogas purification process. Measurement tests carried out on the level of CH₄ increase and CO₂ adsorption using the Gas Chromatography Shimadzu-14 A.

In conclusion, choosing activated carbon as an adsorbent is more appropriate than zeolite. This is due to activated carbon could eliminate the most CO₂ contaminants and obtain the most CH₄ content. Furthermore, using the smallest adsorbent size and reactor with largest surface area has been identified to be able to increase the CH₄ content and reduce the CO₂ gas content. There are still interesting things to do to improve the performance of biogas purification. Measuring the capacity of two adsorbents and measuring the purification process using a mixture of two ingredients can be alternative research in the future.

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