

Pengaruh Ukuran Partikel Biochar Terhadap Produksi Biogas Menggunakan Substrat Limbah Roti

The Effect of Biochar Particle Size on Biogas Production Using Bread Waste Substrate

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ABSTRAK

Tujuan dari penelitian ini adalah menginvestasi pengaruh penambahan ukuran biochar dengan variasi ukuran partikel pada produksi biogas dengan *two stage anaerobic digestion* menggunakan limbah roti tawar. Penelitian ini dilakukan dalam skala laboratorium dengan bioreaktor 100 mL. Penelitian ini menambahkan variasi biochar dengan ukuran partikel <500 µm, <250 µm, <149 µm, <125 µm, dan <63 µm dan penambahan variasi konsentrasi biochar 5 g/L, 15 g/L, dan 25 g/L. Kultur campuran diinkubasi pada suhu 35 °C selama 40 hari. Penelitian ini mengukur volume gas harian, pH dan *Chemical Oxygen Demand* (COD). Hasil penelitian menunjukkan bahwa ukuran partikel biochar <500 µm dengan konsentrasi biochar 5 g/L meningkatkan produksi biogas sebesar 60% dan menghasilkan akumulasi biogas sebesar 4,240.03 mL/L. Biochar terbukti dapat meningkatkan pH pada proses *anaerobic digestion*. Konsentrasi COD menurun dari 9,670 mg/L menjadi 1,640 mg/L.

ABSTRACT

The effect of biochar supplementation with different particle size on biogas production in two-stage anaerobic digestion of white bread waste was carried out on a laboratory scale using a 100 mL bioreactor. The supplementation of biochar variations with particle sizes <63 µm, <125 µm, <149 µm, <250 µm, and <500 µm and the addition of different biochar concentration of 5 g/L, 15 g/L, and 25 g/L were investigated to obtain optimal concentrations. Mixed cultures were incubated at 35 °C for 40 days. Daily gas production, pH and Chemical Oxygen Demand (COD) were measured using the daily water displacement method. The results showed that the particle size of biochar < 500 µm with a biochar concentration of 5 g/L increased biogas production by 60% and resulted in gas accumulation of 4,240.03 mL/L. CH₄. It has proven that biochar increased pH in the mixture. COD concentration decreased from 9,670 mg/L to 1,640 mg/L.

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1. Introduction

The food industry is increasing, especially the bakery industry, in various countries. Bread is frequently consumed since it is a source of carbohydrates. Therefore, as the production of bread has expanded, so has the amount of wasted bread. According to estimates, Sweden produces 80,410 tons of wasted bread each year [1]. Additionally, according to data from the National Waste Management Information System (SIPSN) for 2021, food waste in Indonesia reached 40.26% of the total waste production, which is a result of the food industry's expansion, including the bakery industry, which grows yearly. Malang City is one of the cities in Indonesia with many white bread industries, with the number of industries from the Central Statistics Agency (BPS) data for 2021 reaching 981 industries [3]. Many existing white bread industries are directly correlated to the waste produced. If waste produced is not properly managed, it will harm the ecosystem, accelerating eutrophication [1] and global warming [4]. This problem requires reasonable waste management solutions, such as processing and utilizing bread waste. Anaerobic digestion is one technique that may be used for managing bread waste. Waste treatment using an anaerobic process has several benefits, including producing energy and reducing pollution. Anaerobic processing can reduce greenhouse gases by 0.55 kg CO₂e/kg food waste [5]. In the anaerobic process, there are three primary phases involving microorganisms in the biogas production process: hydrolysis, acidogenesis, and methanogenesis [6]. Biogas contains CH₄ gas (50-70%), CO₂ (30-50%), and other gases that can replace natural gas [7]. Biogas can be used as fuel for producing power and heat, and these gases can be utilized as renewable and sustainable energy sources, so it supports the Indonesian government in achieving the EBT target of 23% in 2025 [8] and is also encouraged by the Regulation of the Minister of Energy and Mineral Resources of the Republic of Indonesia Number 39 of 2017 concerning Implementation of Physical Activities for Utilization of New, Renewable Energy and Energy Conservation. Indonesia has started developing biogas power generators by converting renewable resources through the production of biogas from animal feces, which could lead to the development of sustainable energy in the country [9]. The utilization of bread waste into renewable energy in the form of biogas by utilizing the anaerobic digestion process produces 48% methane (CH₄) and 52% carbon dioxide (CO₂) [10]. Previous studies on processing bread waste into biogas discovered that using two phases of anaerobic digestion to break down the waste may more effectively and efficiently create hydrogen and methane than using potato starch and sugarcane bagasse fermentation residue [11]. Hydrogen production occurs in the first phase, and methane production peaks in the second phase of anaerobic digestion.

In order to increase biogas production, it is necessary to add additives. Some additives that can be added are metal compounds, fungi, enzymes, activated carbon, and biochar [12]. Based on some of the additives that have been studied, biochar has the potential to be developed in the anaerobic digestion process. Biochar is an additive in anaerobic digestion because it can form biofilms and reduce ammonia inhibition [11]. Adding biochar to biogas can accelerate the formation of granules in sludge, reducing loading rates in the sludge activation process. The biochar production process can be done through high-temperature heating (>500 °C). The materials used for the manufacture of biochar are agricultural and forestry wastes. Biochar has pores that bacteria can use as a place to attach.

Previous research has investigated biochar's physical and chemical characteristics [2], but only a few references have investigated the particle size of biochar in biogas production. The biochar particles will affect the pores used for the microorganism media to attach and grow. Porous biochar will provide a relatively higher surface area [5]. The smaller the particle size of the biochar, the larger the pores [3]. So, this research was conducted to investigate the effect of biochar particle size on biogas production by an anaerobic digestion process using bread waste as a substrate. Knowing that the right biochar particle size is predicted to give a high surface area may optimize the granule formation process and increase biogas production.

2. Research Methods

a. Materials

The inoculum is an organic residue (digestate) obtained from an anaerobic digester of cow feces in Biogas Sanan Village, Malang City. The bread waste was obtained from the Sukun area, Malang City.

b. Sludge Preparation

The sludge was obtained from Kampung Biogas Sanan, Malang City. The equipment used in making this sludge is a filter, funnel, and jig. Before entering the tube, the inoculum is filtered to remove any large particles. [11].

c. Preparation of Bread Solid Waste

Unsuccessful bread batches from the Aries Malang Bread Factory are the main ingredients of solid waste for plain bread. The bread was first chopped and sliced into little pieces. During the production of biogas, bread solid waste is utilized as the bacteria's food source [11].

d. Phases of Research

The Two-Phase Anaerobic Digestion (TPAD) research was conducted using the batch method with a flow chart of the research procedure in **Figure 1**. This research used a Completely Randomized Design (CRD) with two treatment factors: variations in the size of biochar and variations in the concentration of added biochar. Then, the research method used was experimental with a quantitative descriptive approach. The experiment consisted of 15 treatments (**Table 1**). There were variations in the size of the biochar <500 μm , <250 μm , <149 μm , <125 μm , and <63 μm with variations in the concentration of the addition of biochar which are 5 g/L, 15 g/L, and 25 g/L and the addition of bread as much as 15.3 gVS/L.

Table 1. Research design

<i>Biochar Size (μm)</i>	<i>Biochar Doses (g/L)</i>		
	B₁	B₂	B₃
U ₁	U ₁ B ₁	U ₁ B ₂	U ₁ B ₃
U ₂	U ₂ B ₁	U ₂ B ₂	U ₂ B ₃
U ₃	U ₃ B ₁	U ₃ B ₂	U ₃ B ₃
U ₄	U ₄ B ₁	U ₄ B ₂	U ₄ B ₃
U ₅	U ₅ B ₁	U ₅ B ₂	U ₅ B ₃

Description: U₁ : <500 μm , U₂ : <250 μm , U₃ : <149 μm , U₄ : <125 μm , U₅ : <63 μm ; B₁ : 5 g/L, B₂ : 15 g/L, B₃ : 25 g/L

The first phase (acidogenesis) will produce hydrogen (H₂), and the second phase (methanogenesis) will produce methane (CH₄). 10 mL of sludge and 50 mL of distilled water were added to each 100 mL bottle with a working volume of 60 mL. Furthermore, the filtered white bread waste and biochar were mixed into the sample bottles according to each treatment. The next step is to neutralize the pH in the bottle to 7. Before experimenting, the serum bottle is treated with high-purity nitrogen gas for 15 seconds and closed using a rubber stopper and aluminum [13]. The serum bottles were then incubated for 40 days at 35 °C. Three times each set of experiments were conducted under identical circumstances. After 15 days, the remaining culture in the reactor was used as feed for methane production in the second phase by adding 10 mL of sludge. Then, the pH is neutralized back to 7 at 35 °C. A gas sample is taken every week, and the gas volume is measured using the water volume displacement technique daily.

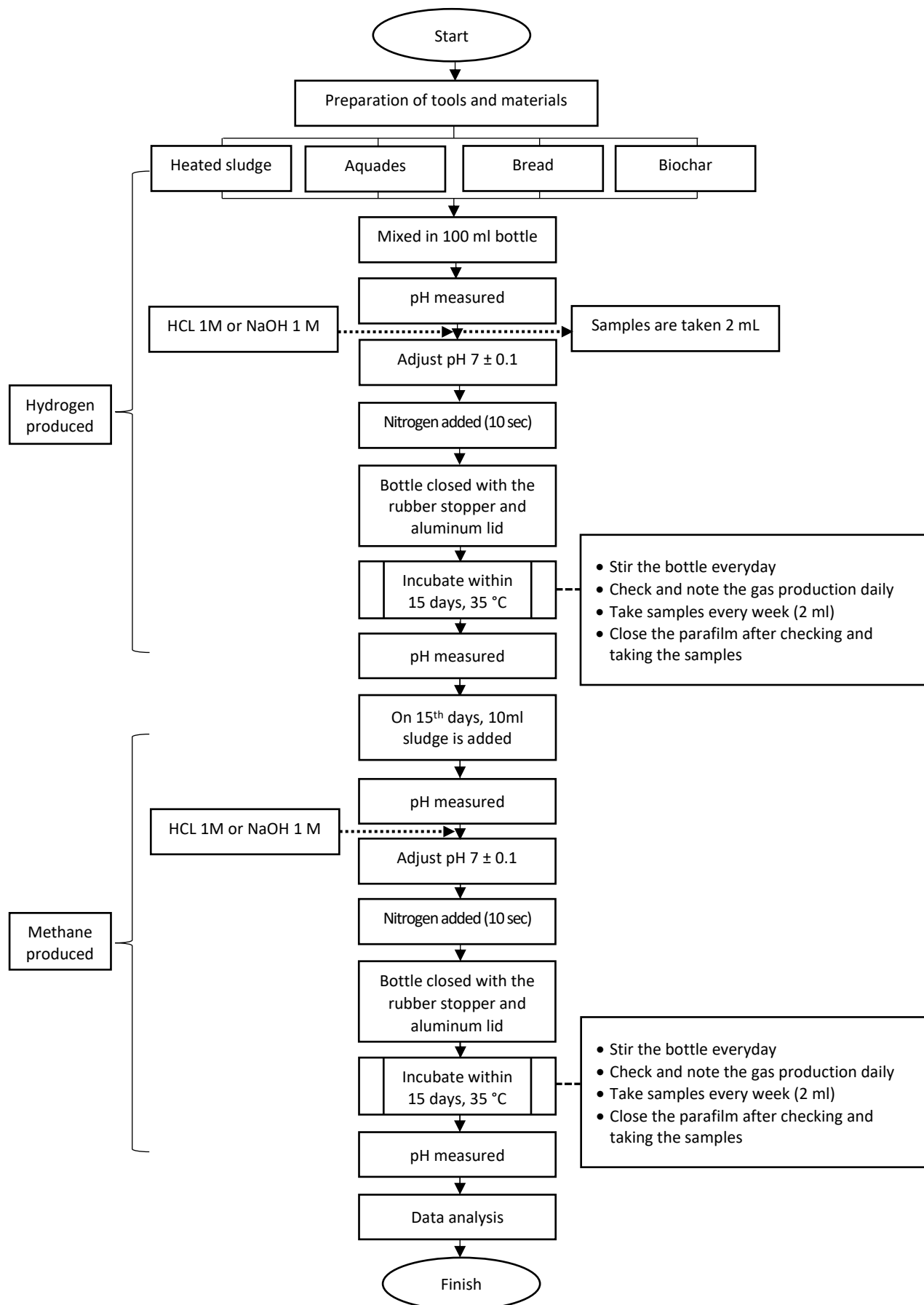


Figure 1. Anaerobic digestion process

The parameters measured in this research were daily gas content, water content, Total Solid (TS) content, Volatile Solid (VS) content, pH, and Chemical Oxygen Demand (COD). pH values were measured on day 1, day 15 and day 40. The pH measurement was done by a digital pH meter using the electrometric method. Moisture content, TS content, ash content, and VS content were measured using the gravimetric method according to SNI 01-2891-1992. In order to measure COD, a DR 900 that had been heated using a DRB 200 was used in combination with the closed reflux technique, and under STP conditions (pressure 273 °K and 1 atm), daily biogas output was calculated using the water displacement technique [3] and converted to volumetric biogas production using the law of ideal gases. Calculation of CH₄ content use the Bushwell equation (**Equation 1**).

$$\text{Bushwell : } C_c H_h O_o N_n S_s + \frac{1}{4}(4c-h-2o+3n+2s)H_2O = \frac{1}{8}(4c+h-2o-3n-2s)CH_4 + \frac{1}{8}(4c-h+2o+3n+2s)CO_2 + nNH_3 + sH_2S \quad (1)$$

Calculation of the Buswell equation uses the chemical composition of organic matter in the form of C, H, O, N, and S [6].

3. Result and Discussion

Characterization of raw materials for biogas generation is required to determine their organic matter content, the large volume of biogas production obtained from the strong organic content inside. Based on the ultimate test results in **Table 2**, it can be seen that the water content in white bread waste was 36.32%, and biochar was 1.64%. The water content affects the microorganisms that will work in making biogas. High water content facilitates simple substrate breakdown by microorganism. The C/N ratio of the final analysis results influences biogas production. The value of the C/N ratio obtained for white bread waste was 18.56%, while for biochar was 145.06%. The C/N ratio in biogas production is used to characterize the elements in organic matter and evaluate anaerobic stability [9]. Because methane bacteria rapidly utilize nitrogen, methane output will be low if the C/N ratio is relatively high [8]. However, since nitrogen can quickly react to generate ammonia (NH₄), the low C/N ratio would kill the methane bacterium.

In addition, the ash content contained in the white bread waste was 8.12%, and biochar was 79.17%. The ash contains inorganic materials such as sodium, potassium, and calcium, which can affect the degradation process. The sample's ash content value relates to TS and VS. TS is the number of dry solids from samples that have undergone an oven or drying process for a certain period. White bread waste was identified as 63.68% of the research's total solid value, whereas biochar totaled 98.36%. The anaerobic process will not be running completely if the TS concentration is low due to the unavailability of a source of microbial nutrition. The research results of bread waste VS are 91.88% and biochar 20.83%. The VS content significantly affects the amount of biogas accumulation and the resulting methane gas content. Sugiarto explained that high VS content could increase the substrate requirements that microbes need [13].

Tabel 2. Ultimate analysis of bread and biochar waste content

Parameter	Unit	Bread Waste	Biochar
Water content	%	36.32	1.64
Ash content	%	8.12	79.17
C	%	42.70	84.14
H	%	9.10	1.57
O	%	45.6	13.70
N	%	2.30	0.58
C/N	%	18.56	145.06
Total Solids	%	63.68	98.36
Volatile Solids	%	91.88	20.83

Based on **Table 3**, the results of the proximate test showed that the protein content in white bread waste was 11.03%, while biochar was 3.32%. The value of protein content in bread waste was 4.96% and biochar 0.27%. Bread has a high-fat content; if it is low, this may be due to the nature of the fat, which is hard to dissolve in water and extract. The carbohydrate content obtained from the proximate analysis was 35.94%, whereas, in biochar, there was no carbohydrate content. The high carbohydrate content in bread indicates that the organic matter content, which can be hydrolyzed in the formation of biogas, will have a high impact so that the methane produced will be maximized. Sunyoto stated that the organic matter contained in bread also makes bread a suitable substrate for biogas or a source of nutrition for microorganisms [11].

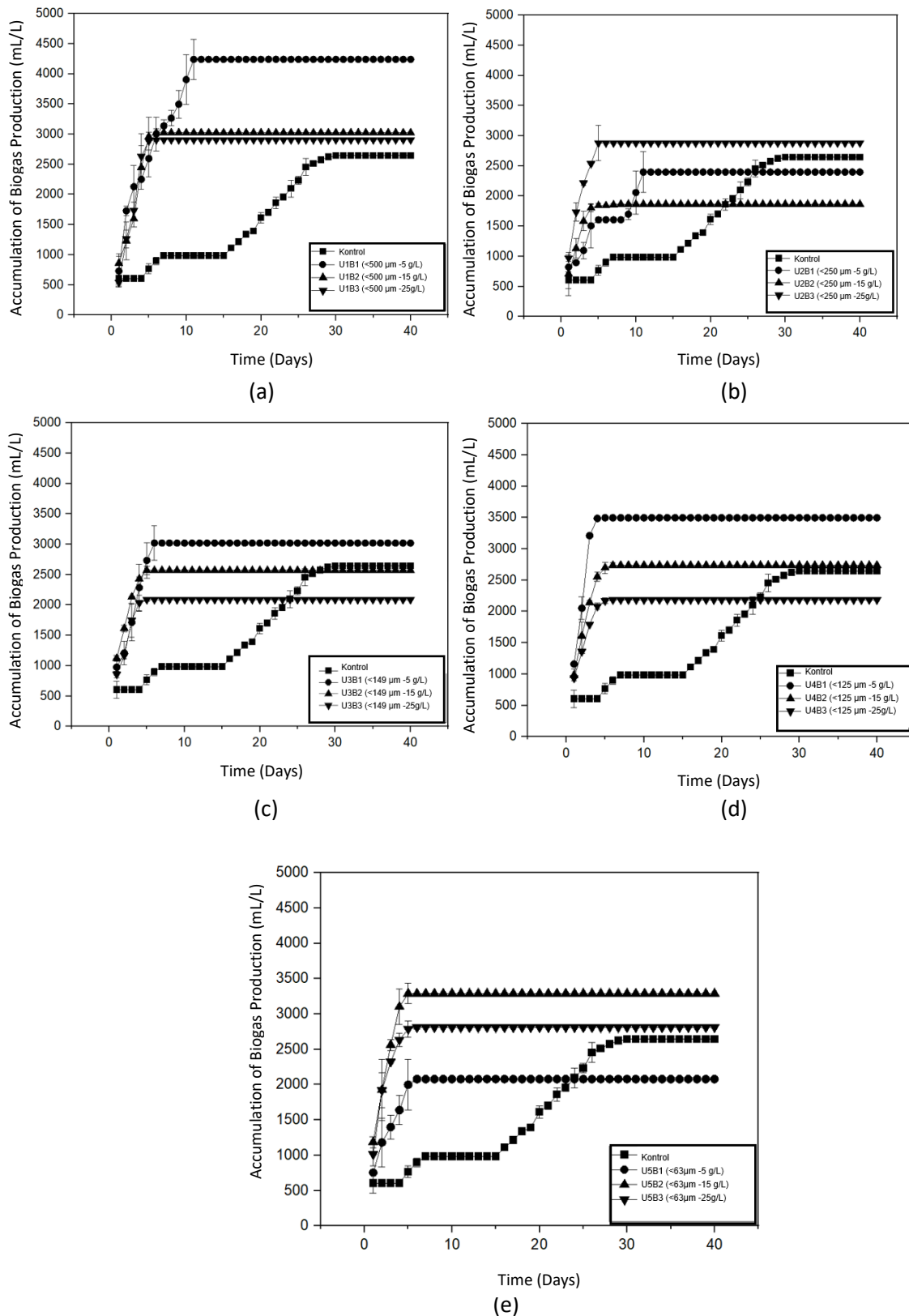
Tabel 3. Proximate analysis of bread and biochar waste content

Parameter	Unit	Bread Waste	Biochar
Protein content	%	11.03	3.32
Fat content	%	4.96	0.27
Carbohydrates	%	35.94	0.00

The different sizes of biochar are used to determine the effect on the production of biogas and the microbial media in anaerobic digestion. The biomass used was 0.5 g/L, 15 g/L, and 25 g/L. The volume of gas produced is measured based on the pressure generated daily using the water volume displacement method.

Based on **Figure 2**, the best variation of the size and concentration of biochar added to biogas production is the U₁B₁ and U₄B₁ variations with adding both sample concentrations of 5 g/L. It is known that adding biochar can increase biogas production compared to the control (substrate 15.3 gVS/L), for the control biogas production results (without adding biochar), which accumulated from 40 days, was 2,642.841 mL/L. In contrast, the U₁B₁ sample (adding 5 g/L biochar) resulted in a gas accumulation of 4,240.037 mL/L, the U₅B₂ sample (adding 15 g/L biochar) was 3,289.599 mL/L, and the U₂B₃ sample (adding 25 g/L biochar) it was 2,880.861 mL/L. Adding biochar to biogas production can increase biogas production by an average of 31% so that the larger the biochar used, the more biogas produced, but the smaller the biochar used, the lower the biogas produced.

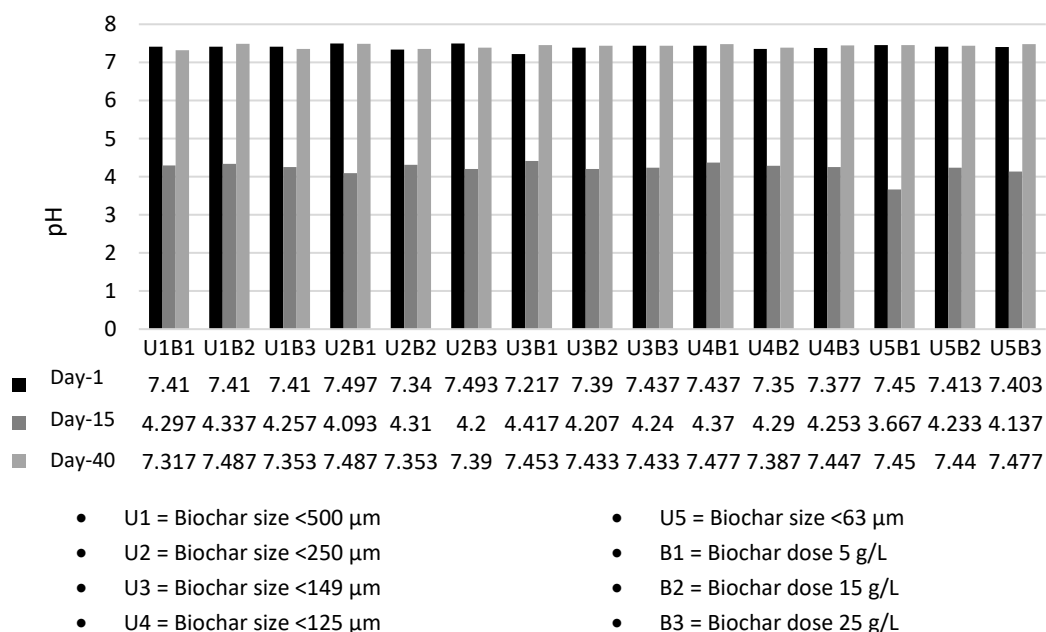
Biochar in biogas production can increase biofilm formation [16] and provide temporary nutrition for microbial growth [11]. Adding biochar can improve the process of anaerobic digestion because it can facilitate microbes in the formation of CH₄ through various stages that occur in anaerobic digestion, including the stages of hydrolysis, acidogenesis, acetogenesis, and methanogenesis. The increase in biogas occurred from day 1 to day 15, whereas the control sample showed a slow increase in biogas production from day 4 to day 7 and was stuck until there was an increase again from day 15 to day 30. So, the production of biogas without adding biochar with two-phase anaerobic digestion takes quite a long time compared to adding biochar.



Gambar 2. Biogas cumulative production with biochar concentration variations <500 μm (a); <250 μm (b); <149 μm (c); <125 μm (d); <63 μm (e).

The first phase of biogas production (day 1 to day 15) will produce hydrogen and volatile fatty acids (VFA). The dominant fatty acids are butyric acid and acetic acid [11]. Biogas production on day 15 for U₁B₁ (biochar size <500 µm and added biochar concentration of 5 g/L) increased and entered the methanogenesis phase after adding sludge, this is under research conducted by [11], knowing that the addition of untreated sludge has an effect where there will be many microorganisms, namely methanogenic bacteria, which play a role in the production of CH₄ by changing the Volatile Fatty Acid (VFA) which accumulates in the early stages in adding of feedstocks. The bacteria contained in the process of methanogenesis or the formation of CH₄ are Methanobrevibacterium, Methanospirillum, and Methanobrevibacter [13]. Variations in the size of the biochar used have varying pore sizes, namely micropores, mesoporous, and macropores. Micropores are responsible for biochar's overall surface area and high adsorption capacity. Mesopores are important for liquid-solid adsorption mechanics. Macropores provide important aeration for enhanced catalytic reactions while also providing hydrology and other more critical factors for biochar applications.

The addition of a biochar dose of 5 g/L can increase biogas production, but this does not apply to certain size variations, namely <250 µm and <63 µm. Therefore, adding biochar to biogas production with excessive doses cannot provide a further enhancing effect for the microbial community in the fixed reactor chamber but can absorb more methane-rich biogas due to the high adsorption capacity of biochar. In the methane production process, the porous biochar structure can support biofilm development and accommodate a variety of methanogens and functional bacteria [11]. However, the immobilization of the microbial community in the anaerobic digestion digester is limited due to poor mass transfer caused by excessive biochar dosage in the limited reactor chamber [13]. In general, biochar can play different roles, such as acting as an adsorbent to reduce the effect of inhibitors, a carrier for microbial colonization, and a buffer for pH to improve anaerobic digestion performance [14]. The processes and mechanisms involved in enhancing AD performance are to solve challenges associated with enhancing microbial metabolism.



Gambar 3. pH data on biogas production

Based on **Figure 3**, the initial pH value of the samples ranged from 7. All samples were conditioned at a neutral pH (pH 7). The pH value was measured at the beginning and end of the biogas production process on the 1st,

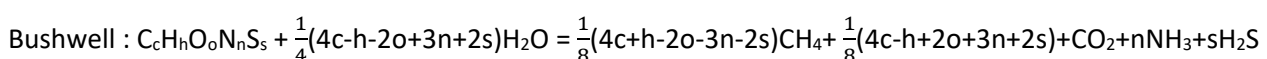
15th, and 40th days (measurement on the 15th day was done after adding sludge to each biogas sample). The pH factor plays a role in anaerobic decomposition. If the pH is not appropriate, the microbes cannot grow to the maximum, which causes microbial death. A suitable pH in the biogas production process is in the range of 6-8. All biogas samples experienced a decrease in pH value so that on the 15th day, the pH value of all samples was in the pH range of 3-5 or tended to be acidic. After the 15th day, the biogas production process occurs (phase 2: methanogenesis). The methanogenesis phase begins with adding sludge as an inoculum as a bacterial starter for microbes that have died or are absent from acidic conditions. The decrease in pH value in phase 1 is due to the formation of organic acids during the acidogenesis process in the form of volatile fatty acids. The accumulation of these organic acids will reduce the pH value and affect the stability of anaerobic fermentation due to acidification [15].

Then in phase 2 (day 15), there was an increase in the pH value caused by methanogenic bacteria, which convert acid and produce nitrogen during protein degradation, thus making the pH alkaline in the reactor [16]; this corresponds to the biogas production phase where there are acidogenesis and acetogenesis phases where volatile fatty acids are formed which will lower the pH value in the biogas reactor. Biogas production on the last day or the 40th day has a pH value of around 7. pH value. It can be seen in one of the highest biogas production data, namely the U₁B₁ variation. The pH data on the last day showed it was in optimum condition, namely 7.47; this indicates that microorganisms have dominated to form methane gas. Methane gas is formed in the optimum range of pH conditions.

Tabel 4. COD content in U₁B₁

Day	Biogas accumulation in U1B1 (mL/L)	COD (mg/L)
1	728.83	9670
7	3136.94	9240
14	4240.03	8760
21	4240.03	8570
28	4240.03	6240
35	4240.03	1800
40	4240.03	1640

COD is another factor that affects biogas production. COD was measured using a DR 900 by taking 2 ml of sample and mixing it with the COD High Range (COD HR) reagent and then heating it for 2 hours at 150 °C in the Digital Reactor Block 200 (DRB 200). Testing the COD content in this research was limited because the costs incurred to test the entire sample were very large, so they were selected from the best variation that produced the highest biogas. The highest biogas sample was in the U₁B₁ sample, where the sample was a variation of the size of 500 µm biochar with an added biochar dose of 5 g/L with 15.3 gVS/L of plain bread substrate. It can be seen from the results of COD measurements for the U₁B₁ sample on days 1, 7, 14, 21, 28, 35, and 40th that there was a decrease in the value of the reduced COD content in the sample; this indicates degradation of organic matter in biogas samples or a reduction in pollutant load from any substrate during the processing, and this is by research conducted by [17]. The COD decrease in the U₁B₁ sample was 83% or 8030 mg/L. Organic matter in the substrate is converted into biogas by bacterial activity, including hydrolysis, acidogenesis, acetogenesis, and methanogenesis. The amount of COD value that is degraded by bacteria is called COD removal. In the process of anaerobic digestion, adding biochar in the process of forming biogas plays a role as a medium that can increase the reduction of COD and reduce the lag phase of methanogenesis.



Tabel 5. Calculation of mol each component C, H, O, N, and S

Bread waste			
Components	Atomic weights	Ultimate analysis (%wt)	n (mol)
C	12	42.7	3.55
H	1	9.10	9.10
O	16	45.6	2.85
N	14	2.30	0.16
S	32	0.30	0.009
Total		100	

Tabel 6. Buswell calculation results for gas potential in biogas production

Bread waste			
Components	Bushwell	% biogas	% biogas without N and S
CH ₄	2.14	57.13	60.14
CO ₂	1.40	37.85	39.85
H ₂ S	0.01	0.25	
NH ₃	0.16	4.38	
H ₂ O	0.01	0.36	
Total	3.74		

The potential for methane in biogas production was obtained from Bushwell calculations (**Equation 1**) based on biogas production in this research [6]. The content of white bread waste that will be used in calculating the potential gas produced from biogas production in this research can be seen in **Table 5**. Buswell's calculations are based on the number of moles of each component, with the results shown in **Table 6**. The gas produced in this research includes CH₄, CO₂, H₂S, NH₃, and H₂O. The gas production resulting from the methane gas bush well equation calculation is 60.14% assuming the biogas constituent components are CH₄ and CO₂. Methane levels in biogas generally range from 55-78% from TPAD experiments [11].

4. Conclusion

Adding biochar as an additive to biogas production using bread waste as a two-phase anaerobic substrate can increase biogas production compared to biogas production without biochar. Adding bread waste as a feedstock helps the biogas production process run optimally in phase 1, which produces hydrogen gas (H₂), and phase 2, which produces methane gas (CH₄). These organic materials, such as carbohydrates 35.94%, fat 4.96%, and protein 11.03% from bread waste, can be a source of microbial nutrition. Effect of biochar particle size on biogas production through two-phase anaerobic digestion using bread waste substrate with different size variations and different biochar doses, namely <500 µm, <250 µm, <149 µm, <125 µm, and <63 µm and a dose concentration of 5 g/L, 15 g/L, and 25 g/L can increase biogas production. Adding biochar with a concentration of 5 g/L with a size of <500 µm had the highest effect on increasing biogas by 60% and produced biogas accumulation of 4240.037 mL/L.

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