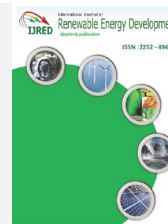








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Research Article

# Effect of Pretreatment and C/N Ratio in Anaerobic Digestion on Biogas Production from Coffee Grounds and Rice Husk Mixtures

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**Abstract.** Indonesia has great potential in producing large quantities of renewable energy sources, such as biomass. Biogas is a renewable energy source produced from biomass. It can be developed in agricultural countries producing rice and coffee, where a large amount of waste is produced in the form of rice husks and coffee grounds. This study examined the effect of physiochemical pretreatment and the C/N ratio on biogas production using coffee grounds and rice husk mixtures. Physical pretreatment was conducted by grinding the mixture up to 50 mesh size, followed by chemical pretreatment by soaking the mixture in 3% KOH; moreover, the variation in the C/N ratio was set at 25 and 30. Anaerobic bacteria were acquired from rumen fluid. The ratio of the coffee ground material, rice husks, and rumen fluid was 1:1:1. This research was conducted in duplicate under batch conditions at ambient temperature (25–35 °C) with a digester volume of 1.5 L. Biogas productivity was measured every 2 d for 60 d. The experimental results indicated that biogas production with a C/N ratio of 30 was 13.3–66.5% higher than that with a C/N ratio of 25. The inclusion of physical pretreatment at a C/N ratio of 30 increased biogas production by up to 31.3%. Moreover, the inclusion of a chemical pretreatment at a C/N ratio of 30 resulted in 30.3% higher biogas production. The kinetics model of biogas production showed that a C/N ratio of 30 with physical and alkaline pretreatment can produce maximum biogas yields of 6,619 mL and 6,570 mL, respectively. Overall, both pretreatments sequentially increased the biogas production significantly.

**Keywords:** Biogas, coffee grounds, rice husks, physical pretreatment, chemical pretreatment, C/N ratio.



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Received: 30<sup>th</sup> Sept 2022; Revised: 28<sup>th</sup> Nov 2022; Accepted: 19<sup>th</sup> Dec 2022; Available online: 1<sup>st</sup> Jan 2023

## 1. Introduction

Biogas is a combustible gas (CH<sub>4</sub> and CO<sub>2</sub>) produced by the decomposition of organic compounds by anaerobic microorganisms. The energy produced by biogas is environmentally friendly (non-polluting) and can be used for various purposes. Biogas is generally produced in an airtight (anaerobic) reactor and contains 55–57% CH<sub>4</sub>, 25–45% CO<sub>2</sub>, 0–0.03% H<sub>2</sub>, 0–3% H<sub>2</sub>S, 0.1–0.5% O<sub>2</sub>, and water vapor (Ryckebosch *et al.*, 2011; Ullah Khan *et al.*, 2017; Zheng *et al.*, 2014). The fermentation stages in biogas production include (i) hydrolysis of polymer substrates into monomers, (ii) acidogenesis to convert monomers into volatile fatty acids, CO<sub>2</sub>, and H<sub>2</sub>, (iii) acetogenesis to produce acetate from metabolic intermediates, and (iv) methanogenesis to convert acetate and CO<sub>2</sub> into CH<sub>4</sub> (Bruni, Jensen, & Angelidaki, 2010). Biogas production requires organic materials with a C/N ratio of approximately 20–30. Therefore, plantation waste can be utilized for this process (Budyono *et al.*, 2021; Matin & Hadiyanto, 2018; Sumardiono *et al.*, 2022; Syafrudin *et al.*, 2020). Coffee is one of Indonesia's largest plantation products. It can be found in almost all regions in Indonesia. However, coffee

processing produces waste, such as coffee grounds, which originate from the brewing processes.

Indonesia was the fourth largest coffee-producing country in the world in 2020, with 11.9 million sacks weighing 60 kg (International Coffee Organization, 2021). Coffee consumption in Indonesia has increased by approximately 7% annually (Limantara *et al.*, 2019). This increase was accompanied by an increase in the number of coffee grounds produced. Unfortunately, the use of coffee grounds is still limited in scope; one example is their use as a nutritional enhancer for plants. Therefore, in this study, we developed a process to utilize coffee grounds in biogas production. Coffee grounds have a C/N ratio of 23.3; therefore, they can be used for biogas production (Kim *et al.*, 2017).

In addition to coffee grounds, rice husks are also an example of agricultural wastes that can be used as biogas. Indonesia has an abundant generation of rice husk waste as it is an agricultural country that produces rice, as observed from the rice production in Indonesia that reached 54.42 million tons in 2021. During rice processing, approximately 20–30% of rice husk waste is produced (Matin *et al.*, 2020). Therefore, processing

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rice husks to avoid environmental pollution is necessary. Thus, rice husks can be processed into biogas as they have a C/N ratio of 85 (Thiyageshwari *et al.*, 2018), which is extremely high. This indicates that mixing other substrates is necessary to reduce the C/N ratio. Mixing coffee grounds and rice husks can result in an optimal C/N ratio. The mixture is processed into biogas via a fermentation process utilizing microorganisms in an anaerobic bioreactor. Anaerobic digestion (AD) is commonly used in biogas production. It is a complex biological process wherein anaerobic bacteria decompose organic matter in an anaerobic environment (Brown & Li, 2013; Budiyono *et al.*, 2021). Compared with biological and thermochemical conversion processes, such as those using cellulosic ethanol, AD technology for CH<sub>4</sub> production is a relatively more efficient method for energy generation from biomass. Anaerobic treatment was selected because it produces large amounts of CH<sub>4</sub> and CO<sub>2</sub> (biogas). Treating raw materials is important for obtaining optimal biogas; additionally, pretreating coffee grounds is important (Ahmad *et al.*, 2018; Dwi Nugraha *et al.*, 2018; Mirtsou-Xanthopoulou *et al.*, 2014). Pretreatment was conducted to decompose the complex internal structure of lignocellulose. A mixture of coffee grounds and rice husks has high lignocellulose content.

Currently, the use of rice husks and coffee grounds is limited. The utilization of coffee grounds as biogas sources remains minimal. Coffee grounds have great potential for use as biogas sources. This study aimed to utilize coffee grounds and rice husk as alternative energy sources. Biogas production using a mixture of coffee grounds and rice husks has never been

achieved; therefore, a new development process should be designed for their utilization. Accordingly, this study examined biogas production using a mixture of coffee grounds and rice husk waste considering physical pretreatments, such as grinding, and chemical treatments using KOH (alkaline), considering variations in the C/N ratio.

## 2. Materials and Methods

### 2.1 Study Period and Location

The research was conducted in 2021–2022 at the Waste Treatment Laboratory, Chemical Engineering, Diponegoro University.

### 2.2 Materials and Experimental Set Up

Coffee grounds acquired from a coffee shop in Semarang City, and rice husks acquired from a rice mill house in Tembalang were used as substrates. Cow rumen fluid from a slaughterhouse in Penggaron was used as a source of anaerobic bacteria. An alkaline pretreatment was conducted using KOH and HCl obtained from a local shop in Semarang for substrate neutralization. A blender was used for the physical pretreatment process. A plastic laboratory-scale 15-L biogas digester was used. The research scheme followed in this study is illustrated in Figure 1.

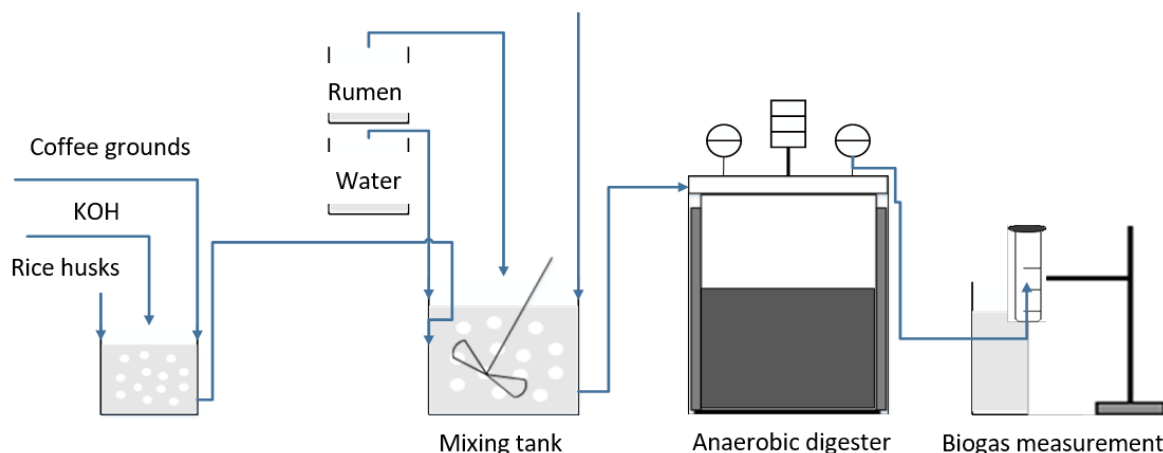


Fig. 1 Laboratory-scale research scheme

**Table 1**  
Experimental design for biogas production

Run	Pretreatment	C/N Ratio	Coffee Grounds: Rice Husks: Rumen Fluid	Yields (mL)	
				Experimental Values	Model Maximum Values
1	-	-	1:1:1	2,262	2,581
2	-	25	1:1:1	2,744	3,026
3	-	30	1:1:1	4,263	5,040
4	Physical	-	1:1:1	3,277	3,684
5	Physical	25	1:1:1	4,841	5,843
6	Physical	30	1:1:1	5,292	6,619
7	Alkaline	-	1:1:1	3,373	3,618
8	Alkaline	25	1:1:1	4,943	5,684
9	Alkaline	30	1:1:1	5,749	6,570

### 2.3 Biogas Production

This study was conducted on a laboratory scale in batches using the anaerobic digestion method under mesophilic conditions, namely at ambient temperature (25–35 °C). Prior to the pretreatment process, an analysis was conducted to calculate the values of total solids and water content in the coffee grounds and rice husks. Physical pretreatment was performed by grinding the material to a size of 50 mesh. Subsequently, an alkaline pretreatment was conducted (by soaking the substrates in 3% KOH solution), and the pH was adjusted to a neutral value using HCl. After a neutral substrate was acquired, it was mixed with rumen fluid at a predetermined ratio. The C/N ratio was varied from 25 to 30. Before entering the anaerobic digester, all the materials were stirred in a mixed tank according to their respective variables. The run and research variables are presented in Table 1. The finished sample was placed into the digester, tightly closed to obtain anaerobic conditions, and the operation was initiated. The amount of biogas produced was measured using the water displacement method, namely, by flowing gas into a measuring cup filled with water and then observing the difference in the water level in the measuring cup; accordingly, the biogas volume was obtained in millimeters. The biogas formation process was conducted for 60 d (until the biogas productivity decreased), and the volume was checked every 2 d.

### 2.4 Kinetic Model

Biogas technology is an active technology that uses anaerobic microorganisms. Therefore, the rate of biogas production kinetics can be ascertained in line with the growth rate of microorganisms in the digester, which is in accordance with the Gompertz equation model (Budiyo et al., 2021; Chouaibi et al., 2020).

$$Y = A \cdot \exp \left\{ -\exp \left[ \frac{U \cdot e}{A} (\lambda - t) + 1 \right] \right\} \quad (1)$$

where Y = cumulative biogas production per unit time days (mL), A = maximum biogas production that can be produced (mL), U = biogas production growth rate (mL/day),  $\lambda$  = the required lag phase time before the formation of CH<sub>4</sub> (days), t = cumulative time for biogas production (days), and e = Euler's number (e = 2.71828).

## 3. Results and Discussion

### 3.1 Effect of the two C/N Ratios (25 and 30) on Biogas Production

Biogas production at C/N ratios of 25 and 30 was compared without pretreatment and it was also compared with the biogas production without any treatment (variable control). It was found that the biogas production at C/N ratios of 25 and 30 was 2,744 mL and 4,263 mL, respectively, while the biogas production in the control variable reached 2,269 mL (Figure 2). For this control variable without pretreatment, kinetic tests were conducted using the Gompertz equation (Eq. 1), at C/N ratios of 25 and 30, and for control. The maximum biogas production (A), specific growth rate (U), and lag time ( $\lambda$ ) under these three conditions were 3,026 mL, 102.6 mL/d, and 16 d; 5,040 mL, 120 mL/d, and 15 d; and 2,581 mL; 81 mL/d, and 17 d, respectively. These results showed that the variable with a C/N ratio of 30 exhibited the highest biogas production, followed by a C/N ratio of 25 and the control variable.

The variable with a C/N ratio of 30 had a higher biogas production (66.5 %) than the variable with a C/N ratio of 25, and the control variable had the lowest biogas productivity. This was because at a C/N ratio of 25, the N amount cannot be possibly assimilated, and is lost through volatilization as ammonia (denitrified). This ammonia can be toxic to bacteria, causing their mortality; notably, the death phase is faster than under usual conditions. According to Budiyo et al., 2018, who examined the productivity of biogas produced from biomass in the form of cassava, a C/N ratio of 30 was an optimum ratio for anaerobic fermentation using biomass as the substrate because the amount of N produced, which is an energy source for methanogenic bacteria, was sufficient; thus, the death phase was not reached too quickly and the bacteria produced biogas for a longer time.

### 3.2 Effect of the two C/N Ratios (25 and 30) with Physical Pretreatment on Biogas Production

The biogas production at C/N ratios of 25 and 30 with physical pretreatment by grinding with a size of 50 mesh was compared. Moreover, the variables obtained without setting the C/N ratio were also compared. The biogas production at C/N ratios of 25 and 30 were 4,841 mL and 5,292 mL, respectively, while biogas production with physical pretreatment without setting the C/N ratio (C/N ratio 38) reached only 3,277 mL (Figure 3). Furthermore, a kinetic test was performed using the Gompertz equation. The values of the variable with physical pretreatment at a C/N ratio of 25, a C/N ratio of 30, and a variable without a C/N ratio setting were: A: 5,843 mL, U: 132 mL/d, and  $\lambda$ : 14 d; A: 6,619 mL, U: 142 mL/d, and  $\lambda$ : 15 d; and A: 3,684 mL, U: 108 mL/d, and  $\lambda$ : 13 d, respectively. These results indicated that the physical pretreatment at a C/N ratio of 30 exhibited the best biogas production compared to the physical pretreatment variable at a C/N ratio of 25 and without setting the C/N ratio.

Based on the three variables, the results with physical pretreatment at a C/N ratio of 30 had a 13.3% higher biogas production than the variable with a C/N ratio of 25, while the variable with physical pretreatment without setting the C/N ratio had the lowest biogas productivity. Compared with the variables with and without physical pretreatment when the C/N ratio was 30, the variable with physical pretreatment had 31.3% higher biogas productivity than the variable without physical pretreatment. This was in accordance with the results of previous studies (Lim et al., 2012; Sumardiono et al., 2022; Syafrudin et al., 2020), which reported that the presence of physical pretreatment increases the surface area of the substrate, which is in direct contact with anaerobic bacteria, thus, increasing the amount of biogas due to the effect of bacteria in the substrate conversion process.

### 3.3 Effect of the two C/N Ratios (25 and 30) with Alkaline Pretreatment on Biogas Production

In this section, we discuss the comparison between biogas production at C/N ratios of 25 and 30 with alkaline pretreatment using KOH. Moreover, the variables obtained without setting the C/N ratio were compared. Biogas production at C/N ratios of 25 and 30 was 4,943 mL and 5,749 mL, respectively, whereas biogas production with alkaline pretreatment without setting the C/N ratio (C/N ratio 38) only reached 3,373 mL. Figure 4 shows the biogas productivity. A kinetics test of biogas production based on the Gompertz equation was conducted for this variable with alkaline pretreatment. The results of the kinetics calculations for a C/N ratio of 25, C/N ratio of 30, and the variable without setting the C/N ratio were obtained as follows: A: 5,684 mL, U: 146 mL/d,

and  $\lambda$ : 14 d; A: 6,570 mL, U: 168 mL/d, and  $\lambda$ : 13 d; and A: 3,618 mL, U: 113 mL/d, and  $\lambda$ : 10 d. Based on the kinetic data, we found that in the variable with alkaline pretreatment, the highest biogas production was at a C/N ratio of 30, followed by a C/N ratio of 25 and a variable without a C/N setting.

Based on the above results, the variable with alkaline pretreatment at a C/N ratio of 30 showed 15.5% higher biogas production than the variable with a C/N ratio of 25, while the variable with alkaline pretreatment without setting the C/N ratio showed the lowest biogas productivity. When the biogas productivity under a C/N ratio of 30 was compared between variables with and without alkaline pretreatment, it was found that the variable with alkaline pretreatment had 30.3% higher biogas productivity than the variable without alkaline pretreatment. This was because pretreatment using alkali can separate lignin, hemicellulose, and/or cellulose, thus, promoting easy decomposition of lignocellulosic biomass by microbes. Additionally, the pretreatment can also reduce the degree of polymerization and crystallinity and can damage the chain between lignin and other polymers (Liew et al., 2011;

Mancini et al., 2018; Monlau et al., 2012; Syafrudin et al., 2018; Taherdanak & Zilouei, 2014; Zhu et al., 2010). Scanning electron microscopy analysis conducted by Matin & Hadiyanto, 2018 showed that the pretreated rice husks became crushed and irregular. This can be explained by the strong bonds in alkali that can break the ester bond between lignin, hemicellulose, and cellulose (Salehian et al., 2013). KOH molecules enter a substrate and break down the lignin structure such that lignin is more soluble, consequently, decreasing the lignin levels (Chandra et al., 2012). KOH is useful in destroying the lignin structure; that is, it promotes delignification (Jha et al., 2011; Sivagurunathan et al., 2017). Moreover, KOH pretreatment can increase cellulose decomposition and sugar degradation more significantly than acid pretreatment; however, its application is constrained by its high cost. The pretreatment of lignocellulosic materials with KOH solution causes swelling, increased internal surface area, reduced degree of polymerization, reduced crystallinity, separation of structural bonds between lignin and carbohydrates, and disruption of lignin structure (Karimi & Taherzadeh, 2016; Taherzadeh & Karimi, 2008).

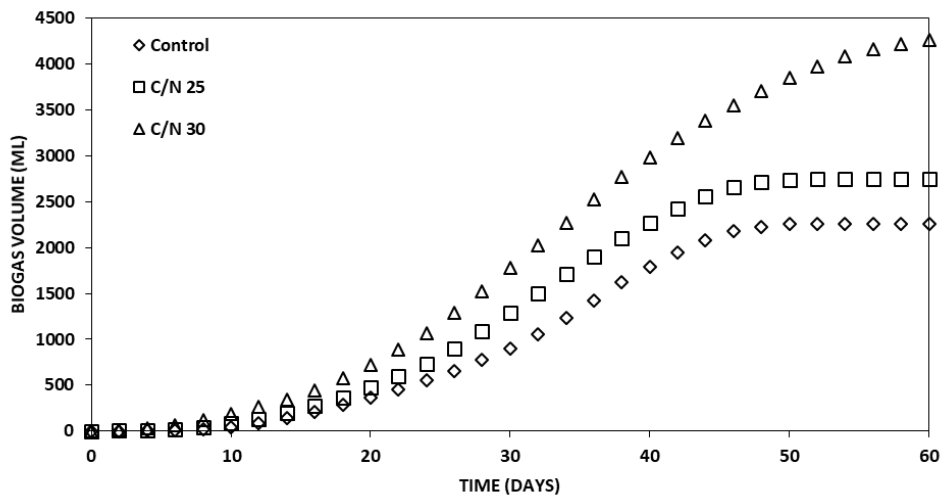


Fig. 2 Total Biogas Production at C/N Ratios of 25 and 30

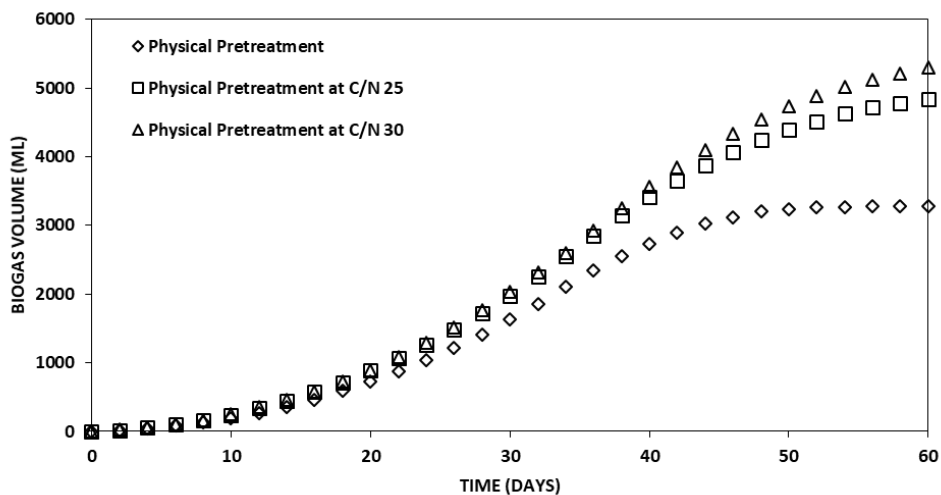


Fig. 3 Total Biogas Production at C/N Ratios of 25 and 30 with Physical Pretreatment

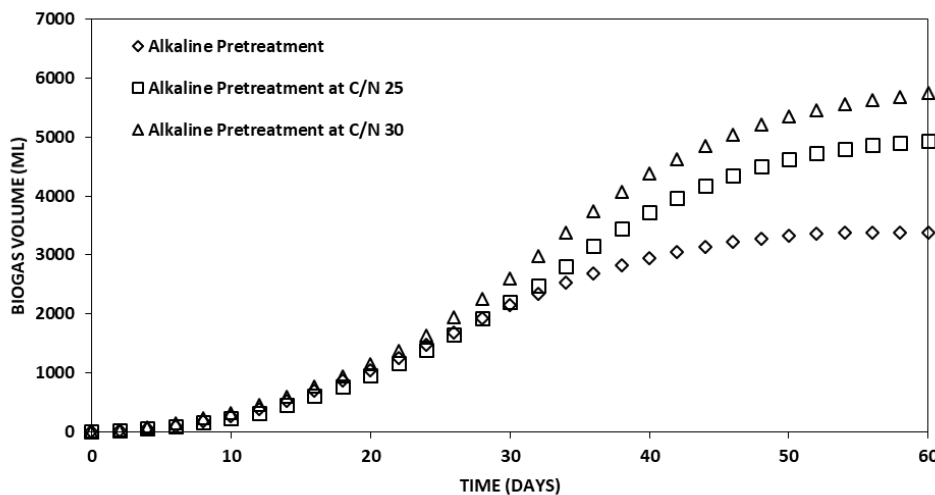


Fig. 4 Total Biogas Production at C/N Ratios 25 and 30 with Alkali Pretreatment

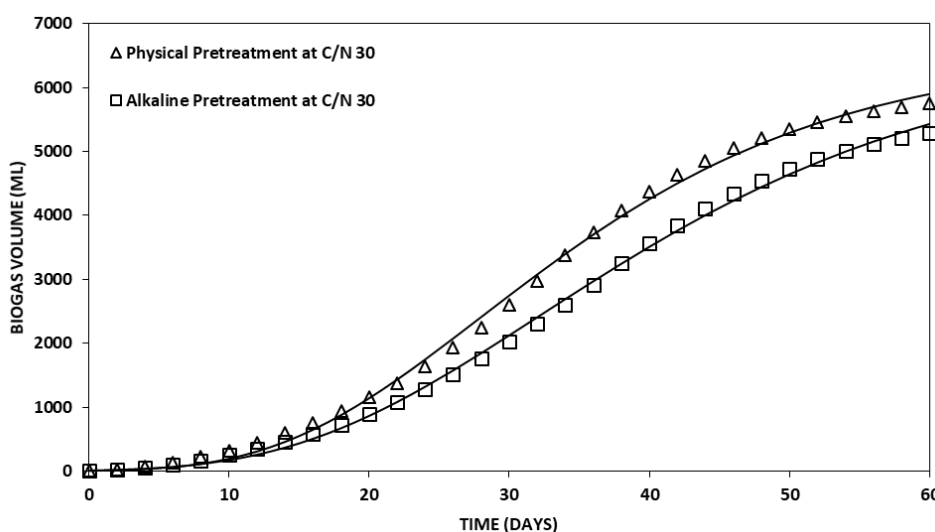


Fig. 5 Relationship between Experimental Data and Model, with Physical and Alkaline Pretreatments at a C/N Ratio 30

### 3.4 Study of Physical and Alkaline Pretreatment with C/N Ratios of 25 and 30 in Biogas Production

This study compared two pretreatments, namely, physical treatment with grinding and alkalinity with KOH at C/N ratios of 25 and 30. As shown in Table 1, the maximum biogas production (A) with physical pretreatment at C/N ratios of 25 and 30 was 5,843 mL and 6,619 mL, respectively, while the biogas production under the alkaline pretreatment at C/N ratios of 25 and 30 was 5,684 mL and 6,570 mL, respectively. The comparison between the physical and alkaline pretreatments shows that physical pretreatment had higher biogas productivity than alkaline pretreatment, both at C/N ratios of 25 and 30.

However, the difference (0.7–2.7%) was not significant. This is because both pretreatments could increase the biogas production. Research related to the physical pretreatment was conducted by Mustafa *et al.*, 2017, who stated that the physical pretreatment of milling can increase the production of biogas from rice straw. Studies on biogas production from rice husk (Syafudin *et al.*, 2020) and corn stalk (Sumardiono *et al.*, 2022) have been conducted to understand the effect of grinding on

biogas production using each substrate and the results indicated that physical pretreatment resulted in relatively high biogas productivity. Similar to physical pretreatment, alkaline pretreatment also contributes to biogas productivity. Alkaline pretreatment is widely used to break down lignin content, increase buffer capacity, and increase methanogenic activity and process stability (Ficara & Malpei, 2011; Liew *et al.*, 2011; Matin & Hadiyanto, 2018; Nugraha *et al.*, 2020; Syafudin *et al.*, 2020). Liang *et al.* (2016) stated that the order of comparison of the effectiveness of alkaline pretreatment was  $\text{KOH} > \text{Mg}(\text{OH})_2 > \text{Ca}(\text{OH})_2$  where alkaline pretreatment was highly effective in dissolving slurry and increasing biogas production. Both these pretreatments positively contributed to biogas productivity and thus, they can be used simultaneously to obtain better biogas yields.

## 4. Conclusion

In this study, biogas production from coffee grounds and rice husk mixtures was assessed considering differences in the C/N ratio. It was found that biogas production with a C/N ratio of 30

was 13.3–66.5% higher than that with a C/N ratio of 25. The physical pretreatment at a C/N ratio of 30 increased biogas production by up to 31.3%. Moreover, KOH pretreatment at a C/N ratio of 30 resulted in 30.3% higher biogas production. The kinetics model of biogas production showed that a C/N ratio of 30 with physical and alkaline pretreatment can produce maximum biogas yields of 6,619 mL and 6,570 mL, respectively. Thus, the sequential use of the two pretreatments will certainly significantly increase biogas production from coffee grounds and rice husks. This biogas energy based on coffee grounds and rice husks has the potential to reduce and substitute fossil energy, the availability of which is increasingly depleting.

## Acknowledgments

We acknowledge Diponegoro University for the World Class Research University Program 2021 for supporting this research.

**Author Contributions:** Budiyono: Conceptualization, methodology, writing—review and editing, project administration, validation; Hashfi Hawali Abdul Matin: supervision, writing—review and editing, project administration, validation; Ihzani Yulistra Yasmin and Iwang Septo Priogo: Conceptualization, methodology, formal analysis, writing—original draft. All the authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the World Class Research University Program, 2021, of the Diponegoro University.

**Conflicts of Interest:** This manuscript has not been published previously and is not under consideration for publication elsewhere. We have no conflicts of interest.

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