



Effect of Thermal Pretreatment of Pineapple Peel Waste in Biogas Production using Response Surface Methodology

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Abstract. This study aims to determine the effect of thermal pretreatment from pineapple peel waste on biogas production using a batch anaerobic digestion process. The experimental process was carried out on various variables, including observation time (30 days), operating temperature (25 - 35 °C), the ratio of starter and sample (1:1), also applied by two treatments, namely the anaerobic digester process without pretreatment and pretreatment using a hot water bath with a temperature of 60 °C, 80 °C, and 100 °C, with a time duration of 25, 45, and 65 minutes. The results showed that the thermal pretreatment given to pineapple peel waste accelerated the biogas production process and reduced the lag phase in the anaerobic digestion process. The highest biogas production volume was obtained from pineapple peel waste, which was 616.33 mL (357.190 mL/g volatile solids), pretreated for 25 minutes at 60 °C (variable B3). The lowest biogas production was obtained from pineapple peel waste without pretreatment (variable A), which was 384.33 mL or 219.619 mL/g of volatile solids. The optimum % yield value of CH₄ gas content reached 67.27%, which was achieved in the pineapple peel hot water bath pretreatment at a temperature of 100 °C with a water bath time of 25 minutes. Meanwhile, pineapple peel waste without a pretreatment hot water bath obtained a % CH₄ yield of 60.19%. The lignocellulose analysis results with the highest hemicellulose and cellulose content were found in pineapple peel waste, pretreated for 45 minutes at a temperature of 80 °C (B9 & B10), with 22.1% and 55.2%, respectively. The B9 and B10 samples obtained the lowest lignin content of 0.41% for both samples.

Keywords: Anaerobic digestion; Biogas; Pineapple peel waste; Thermal pretreatment

1. Introduction

A Indonesia is one of the largest pineapple producers in the world, producing around 1,396,153 million tons per year (Widowati, 2019). A large amount of fruit peels biomass from the pineapples mostly ends up as wastes in most production areas. Eventually, the waste will build up and become a source of concern for the environment if it is not controlled. Making value-added products out of pineapple waste is one approach to deal with these wastes in an environmentally friendly manner (Hamzah et al., 2020; Lun et al., 2014; Maneintr et al., 2018). Peel of pineapple waste can be utilized as the raw materials for producing biogas, however the amount of nitrogen is insufficient (Lun et al., 2014).

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Animal waste includes a large amount of nitrogen (N) and phosphorus (P), causing nutrient imbalance and environmental degradation if not effectively managed (Chakravarty, 2016; Deressa et al., 2015; Hamzah et al., 2020). Therefore, combining pineapple peel waste and animal waste could produce biogas with high content of carbohydrates and methane gas while also be the solution for the environmental issues. The gas composition in biogas fuel has a generally higher percentage of CH₄ than CO₂, N₂, O₂, and H₂S. According to (Alvarez & Lidén, 2009), cow dung and the mixture of agricultural waste yielded CH₄ fertilizer in 47% and 47 – 55%, respectively. Thermal pretreatment is one of the processes to increase biogas production. It is used to help the compound contained in plants be easily broken so that microorganisms can easily convert polymers in cellulose and hemicellulose into biogas (Budiyono et al., 2017; Darwin et al., 2016). A method that can be used to produce biogas is the anaerobic fermentation method using a biogas reactor (biodigester). Biogas is a gas mixture consisting mainly of methane and carbon dioxide. Biogas is produced anaerobically through the following three stages: hydrolysis, acidogenesis, and methanogenesis (Biarnes, 2016; Prasetyo et al., 2017). Various kinds of organic wastes such as animal manure, municipal solid waste, agricultural residues, and industrial waste can be used as a substrate in biogas production. Other substrates are kitchen, garden, cow dung, and domestic waste. Different biomass sources (waste) will produce different quantity of biogas, e.g., two liters tapioca waste water could produce 2458 mL of biogas (Budiyono et al., 2018a; Nwokolo et al., 2020; Sumardiono et al., 2015). The biogas production system has several advantages, such as: reducing the effect of greenhouse gases, reducing unpleasant odor pollution, as fertilizer also producing power and heat, creates a healthier environment by converting waste to biofuel, also compost sludge and liquid fertilizer can be made from biogas (Koopmans & Consultation, 1999; Pertiwinigrum et al., 2017).

The pineapple peel is the outer part of the pineapple fruit (*Ananas comosus*), it's also the biomass source that is usually thrown away (Sianipar, 2006). Characterization of pineapple peel waste with C and N content according to (Fu et al., 2016), namely C in the amount of 41.02% and N in the amount of 0.79%. The composition between the C and N content in the pineapple peel waste was reported to affect the biogas CH₄ production (Arifan et al., 2018; Laopaiboon et al., 2010). The pre-treatment and hydrolysis processes are essential processes that affect the yield of biogas. Large organic molecules cannot be directly absorbed and used by microorganisms as a substrate source and yield methane (Schnurer & Jarvis, 2010; Verma, 2002). The pretreatment process is carried out to condition lignocellulosic materials both in terms of structure and size. The pre-treatment process directly affects biogas production by breaking down the lignin content (Ghatak & Mahanta, 2017; Sumardiono et al., 2017). Research conducted by (Basaria & Priadi, 2016) and (Arifan et al., 2021a) shows that the pretreatment process can improve the performance of anaerobic digestion (AD) by increasing the contact between the substrate and microorganisms resulted in the higher amount of methane yields produced of 0.229 L CH₄/g VS. According to (Casabar et al., 2019), the purpose of pretreatment is to open the lignocellulose structure and to make the cellulose more accessible to enzymes that break down saccharide polymers into sugar monomers. Pre-treatment provides easier access to the enzymes to increase glucose and xylose yield. According to (Harmsen et al., 2010) the pretreatment process in which hemicellulose hydrolysis may occur, includes physical pretreatment (heated, crushed, milled, sheared), chemical pretreatment (hydrolysis of weak acids, strong acids, alkalis), a combination of physical and chemical pretreatments (steam explosion, CO₂ explosion, ammonia fiber explosion (AFEX)), and biological pretreatment (enzymes and microorganisms).

An advantage of thermal pretreatment is its cost-effectiveness, as it dissolves the biomass waste's high lignin concentration without the use of sodium hydroxide, which is typically required for other pretreatment methods. The research objective is to determine pineapple peel waste thermal effect on biogas production using a batch anaerobic digestion process. As a result, this research will be used to the problem of finding solutions for renewable energy.

2. Methods

2.1. Materials

The research materials were pineapple peel waste and cow dung. The pineapple peel waste was obtained from fruit traders of Banyumanik market, Semarang, and a starter (cow dung) was obtained from the Faculty of Agriculture and Animal Science (FPP), Diponegoro University (UNDIP). The anaerobic digestion process was carried out in mesophilic conditions (at ambient temperatures) in the range of 25 – 35°C, where the shearing process is carried out for 30 days with a system batch. The series of biodigester tools can be seen in Figure 1.

2.2. Methods

2.2.1. Preparation of the Starter

The starter is the digester output, derived from the fermentation of cow dung from the Department of Animal Science, Diponegoro University. The starter was previously filtered using a filter cloth, and the amount required was 200 g.

2.2.2. Sample Preparation

Pineapple peel waste was physically pretreated. Then, thermal pretreatment was carried out at varying temperatures and time durations in the hot water bath by blending for 30 seconds. Then, it was put in a hot water bath (Memmert WNB14 Ring Waterbath 14 L) according to the temperature and time variables. For control variables (without pretreatment), the pineapple peel was crushed in a blender for 30 seconds. The sample was fed into the digester. The number of samples depends on the volatile solid (VS) sample and VS starter content.

2.2.3. Biogas Production

The biogas production starts with pouring the starter in 200 g and sample to the glass bottle corked with rubber and connected to a rubber hose with another bottle filled with water and clogged with hollow rubber (for water outlet). The amount of produced biogas was measured by quantifying the water displaced due to formed biogas pressure. The biogas production from the sample is calculated by subtracting the starter of biogas production with biogas produced from a starter and samples. The experiment's observation will be repeated three times in order to provide a more accurate estimate of experimental error (Sutaryo, 2017; Budiyo et al., 2018b).

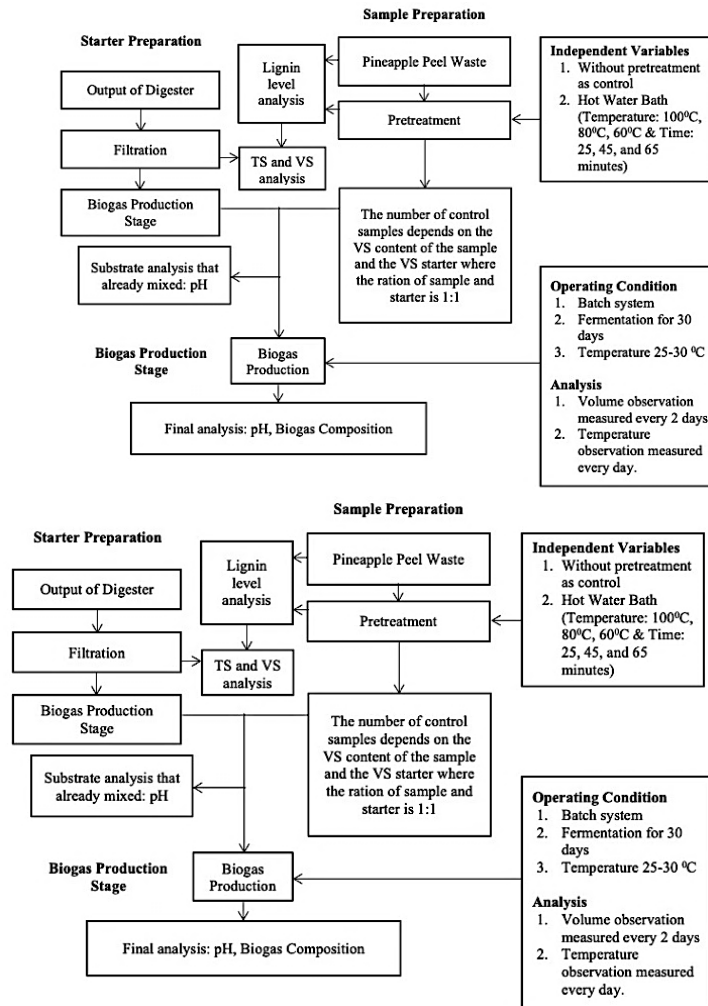


Figure 1 Experimental Procedure

2.2.4. Analysis and Observation

The Hanna Digital HI 98107 pH Meter will be used to record a sample of the biogas production every two days for a period of 30 days, during which time the pH will be measured both initially and ultimately. The environmental temperature measurements to determine the exact temperature of the digestion process, TS, VS, for the material included in the biodigester. Lignocystic analysis using spectrophotometric methods and analysis of the composition of methane and carbon dioxide gas, by using Gas Chromatography. Thermal Conductivity Detector (GC-TCD) Hewlett Packard 6890 FID TCD. Data interpretation was used to obtain data which useful in determining the most influential process variables using Response Surface Methodology (RSM).

3. Results and Discussion

3.1. Characterization of Raw Material

The raw material used in the production of biogas was pineapple peel waste. Pineapple peel contains lignocellulose which consists of lignin, cellulose, and hemicellulose. Lignin is one of the very complex compounds to break down, so it needs pretreatment to break down lignin so that cellulose is easily accessible. Cellulose plays a significant function in converting into biogas. This cellulose will be turned into glucose and utilized as food for methanogenesis bacteria to transform the complicated substances into methane.. For this reason, it is necessary to characterize pineapple peel waste to determine how much lignin,

cellulose, and hemicellulose content is available. The following is the result of the characterization of pineapple peel waste in Table 1.

Table 1 Characterization of Raw Materials

Parameter	Value
Lignin	1.14%
Hemicellulose	17.4%
Cellulose	51.1%

3.2. Total Solid (TS), Volatile Solid (VS), pH, and Temperature

Analysis TS and VS are carried out to calculate substrate requirements of pineapple peel waste needed in the digester. Materials tested included starter, pineapple peel waste with and without pretreatment. Following are the results of the TS and VS analysis. The sample used in the TS and VS analysis used were samples weighing 4 g.

Table 2 Measurement of TS and VS Weight to Determine the Amount of Substrate that Entered the Digester

Treatment	Cup	TS	Ash	% TS	% Ash	% VS	Pineapple Peel (g)	Total Substrate (g)
Starter	1.27	1.32	1.29	1.35	0.475	0.875	-	-
A	209.25	2.04	1.28	19.55	0.62	18.93	9.25	1.25
B1	1.26	2.07	1.29	20.34	0.69	19.65	8.91	208.91
B2	1.26	2.09	1.29	20.58	0.72	19.85	8.82	208.82
B3	1.27	2.07	1.29	20.14	0.69	19.45	9.00	209.00
B4	1.25	2.07	1.28	20.41	0.81	19.60	8.93	208.93
B5	1.27	2.08	1.30	20.34	0.71	19.63	8.92	208.92
B6	1.26	2.03	1.28	19.20	0.64	18.56	9.43	209.43
B7	1.26	2.07	1.29	20.13	0.60	19.53	8.96	208.96
B8	1.25	2.09	1.28	20.89	0.78	20.11	8.70	208.70
B9	1.26	2.06	1.29	20.08	0.84	19.24	9.10	209.10
B10	1.26	2.07	1.29	20.39	0.74	19.65	8.91	208.91

The initial and final pH measurements were carried out in the anaerobic process. Temperature measurement is carried out to determine the ambient temperature used during anaerobic. A is pineapple peel waste without pre-treatment, while B is pineapple peel waste using a pre-treatment hot water bath with different temperature and time variations. Figure 2 shows that the temperature measurement is obtained between 30-32°C, where the temperature falls into the mesophilic range. While the pH obtained is shown in Table 3 also for the initial pH obtained, which is 6, while after the anaerobic digester process, the pH is 8 for the whole treatment. The optimum pH conditions for producing biogas are in the range of 6.8 – 7.2 (Arifan et al., 2021b).

Table 3 Measurement of pH

Std. Run	Initial pH	Final pH
A (non-pretreatment)	6	8
B1	6	8
B2	6	8
B3	6	8
B4	6	8
B5	6	8
B6	6	8
B7	6	8
B8	6	8

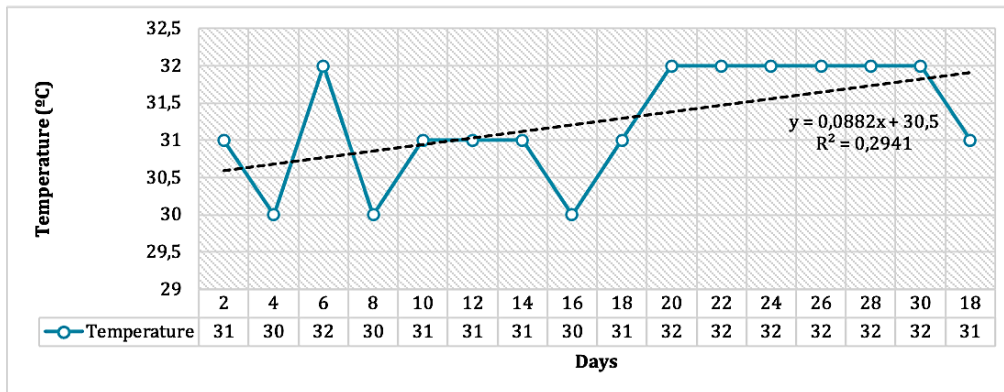


Figure 2 Measurement of Temperature

3.2. Biogas Production

Production of biogas from pineapple peel using a batch biodigester was conducted through 10 experiments using the independent variables, namely pretreatment time and temperature. Total production of biogas from waste pineapple peel is performed for 30 days. Pineapple peel waste without pretreatment (A) produced a smaller volume than pretreatment using a hot water bath.

Figure 3 shows that the highest total biogas production for 30 days of observation pretreated for 25 minutes at 60 °C (variable B3) was 352.190 mL/g VS. Meanwhile, the lowest biogas production without pretreatment (variable A), was 219.619 mL/g VS. This observed due to the high lignin content in pineapple peel waste without pretreatment so that microorganisms are very difficult to degrade the substrate. The lignin content is still high because there is no pretreatment. That thermal pretreatment can be utilized to increase biogas production has been demonstrated through the work of (Saragih et al., 2019) and (Schwede et al., 2013) that pretreatment time also had an impact on biogas yield. Samples pretreated for 8 hours produced more gas than samples pretreated for 2 hours.

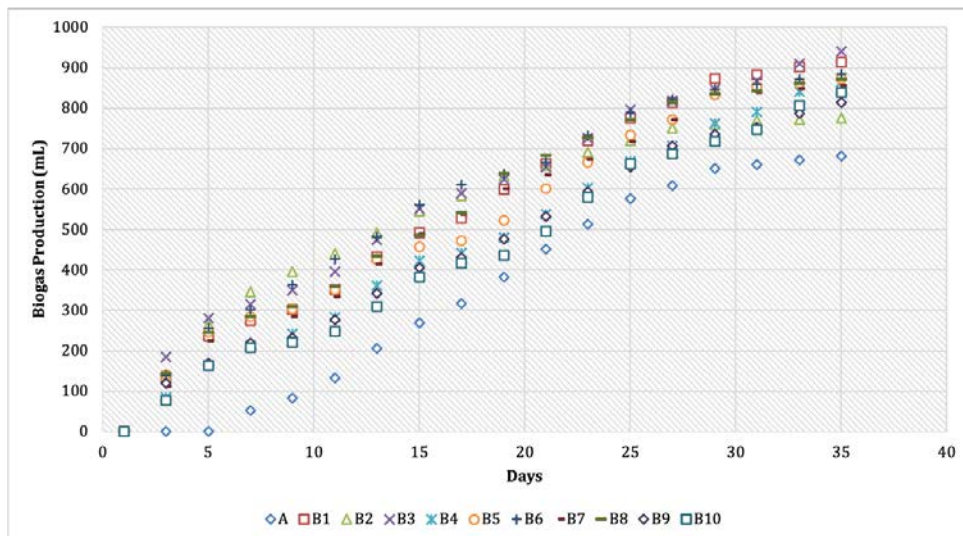


Figure 3 Biogas production with thermal pretreatment (B) and without pretreatment (A)

Figure 3 shows that the substrate that was not pre-treated (A) has a more extended time lag phase than the substrate that has been given thermal pretreatment (B1 – B10). This is also in accordance with the research conducted by (Bougrier et al., 2008), which states that the application of thermal pretreatment to the substrate in the form of activated sludge in the anaerobic digestion process can increase biogas production. The increase in

biogas production volume is related to the rise in the amount of organic matter easily dissolved in water to facilitate the conversion process of organic waste into biogas. The highest yield of biogas was in variable B3 (temperature 60 °C, time 25 minutes), which was 367.905 mL/g VS, then followed by B1 (temperature 100 °C, time 25 minutes), which was 352.190 mL/g VS. Then followed by variable B7 (temperature 80 °C, time 16.72 minutes) amounting to 362.476 mL/g VS. The increase in biogas production indicates that cellulose is completely degraded and the hydrolysis process is going well. It is stated by (Penghe et al., 2020) that high hydrolysis or dissolving rate can be achieved well by conducting thermal pretreatment using a short temperature and time duration or low temperature with a longer duration pretreatment. Due to the high rate of hydrolysis on thermally pretreated substrates compared to untreated substrates, biogas production is higher at the beginning of the anaerobic process. This is in accordance with research conducted by (Wang & Schmidt, 2010) which states that pretreatment of lignocellulosic biomass waste causes the lignocellulosic biomass structure to be easily broken down so that anaerobic microorganisms easily access the components of biomass containing cellulose. The B2 variable (temperature 100 °C, time 65 minutes) produced the lowest biogas yield than other pretreatment variables. However, the yield is still high when compared to variable A (without pretreatment), namely 273.619 mL/g VS. This can happen due to several factors such as the formation of toxic compounds such as phenolic and furfural. This toxic compounds affects the performance of microorganisms and cause the microorganisms fails to break down the compounds, which reduces the yield of biogas production. It is stated by (Ramos, 2003) that phenolic compounds may have a toxic effect on bacteria, especially methanogens, which affect the anaerobic digestion process. In some cases, other inhibiting compounds for anaerobic bacteria, namely vanillin, furfural, and hydroxymethylfurfural, can also affect the anaerobic digestion process.

In the anaerobic digestion process, it can be seen in Figure 3 that the samples of B1-B10 only need three days to produce biogas in a specific volume. In contrast, sample A (without pretreatment) needs nine days to increase the production volume from 0 to 52 mL. It can be seen from the increase in volume that occurred on day 12. Applying thermal pretreatment to pineapple peel waste increases the efficiency of the anaerobic digestion process, causing bacteria to efficiently convert cellulose into biogas. These results are in accordance with research conducted by (Lei et al., 2013), which states that applying thermal pretreatment using hot water baths on lignocellulosic biomass will increase the rate of hydrolysis process to convert cellulose into simple components in the form of sugar. The hydrolysis products will be converted into fermentation products such as alcohol and volatile fatty acids, then converted into methane gas at the methanogenesis stage. Specifically, anaerobic digestion involves a series of chemical processes, one of which is hydrolysis, the next of these is fermentation and the final of it is methanogenesis (the bacterial conversion of organic acids into methane and carbon dioxide). Methanogens convert acetic acid, carbon dioxide, and hydrogen to biogas in the methanogenesis stage (Metcalf & Eddy, 2003; United States Environmental Protection Agency, 2015).

A response fitted surface can be created from the experimental results in Figure 4(a) that shows the relationship between temperature and time to the biogas production. By applying multiple regression analysis to the experimental data, a second level polynomial equation is obtained to represent the total biogas production obtained as follows:

$$Y = 598.84849588083 - 4.5698390113643 X_1 + 0.031830327967252 X_1^2 \\ - 3.6170407363812 X_2 + 0.048080384517114 X_2^2 \\ - 0.022142857142857 X_1 X_2$$

Biogas production optimization process from pineapple peel waste was carried out through 10 experiments using temperature and time as independent variables, and were pretreated using a hot water bath. This model's validity can be seen from the coefficient of determination (R^2) value. The value of R^2 provides a measure of how experimental variables and their interactions can explain different of the variability in the values of the observed responses. Correlation coefficient R^2 was used to evaluate the model's quality. High correlation between the model's predicted value and the observed response is reported to be indicated by R^2 values more than 0.90 (significantly closer to 1) (Wang et al., 2015; Yulianto et al., 2018). In this case, the value of the coefficient of determination ($R^2 = 0.8236$) indicated that 82.36% of the variability in response can be explained by the model (Table 4).

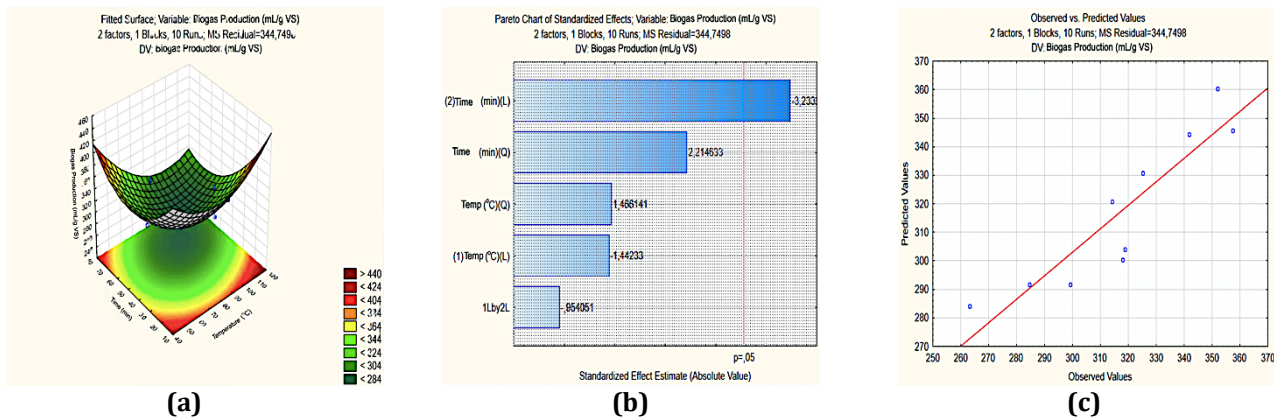


Figure 4 (a) Response Fitted Surface of Temperature and Time Variables to Biogas Production; (b) Pareto Chart of Standardized Effects of Variables on Biogas Production (mL/g VS); (c) Comparison of Experimental Data and Estimates of Biogas Production (mL/g VS)

The proximity of the estimated values from the model is close to the values obtained from the experimental data presented in Figure 4(c). The graph's plot values show a satisfactory correlation between the observed and estimated values due to the closeness in values between the experimental and estimated values to the linear line. The Response Surface Methodology (RSM) optimization also provides the most influential variables. (Yulianto et al., 2020). From the Pareto diagram in Figure 4(b), it appears that the most influential independent variable in making biogas from pineapple peel waste is the duration of time used for the pretreatment of the pineapple peel when is in a hot water bath. The average of each variable pretreated in a short time duration resulted in a higher biogas volume than the variable pretreated in a long time duration. As stated by (Olatunji et al., 2021), the hydrolysis of substrate is regarded as the very important stage in biogas. This means that pretreatment duration in the hydrolysis stage is more decisive than temperature.

According to (Penghe et al., 2020), high hydrolysis or dissolution rates is achieved well by performing thermal pretreatment using a low temperature with a longer duration pretreatment. According to (Acharjee et al., 2011), the high water content of lignocellulosic biomass corresponding to the moisture level of lignocellulosic biomass also impacts the pretreatment time required, which takes longer to be pretreated using a hot water bath. Optimization parameters of biogas production from pineapple peel waste against temperature and time of pretreatment were determined by the critical value in Table 4. Thus, the critical value for optimization of biogas production from pineapple peel is reached when the temperature is 92.257°C for 58.858 minutes with biogas production in the amount 292.0785 mL/g VS.

Table 4 Predictive Value of Optimum Biogas Production at Critical Values of Temperature and Time Pretreatment of Pineapple Peel

Factor	Minimum Value of Treatment	Value of Critical	Maximum Value of Treatment
Temperature (°C)	51.716	92.257	108.284
Time (min)	16.716	58.858	73.284
The approximate value of biogas production (mL / g VS)		292.079	

The second level response surface model is appropriate in the form of Analysis of Variance (ANOVA), given in Table 5. The Fisher ratio of variance, the value of $F (= S^2_r / S^2_e)$, is a statistically valid measure of how well the factors explain variation in data about the mean and the effect of the predicted real factors. The greater the F value, the more uniform it is. The ANOVA of the regression model shows that this model indicates there was a correlation, as evidenced by the F value of the Fisher test (F model = 20.498).

Table 5 Analysis of Variants of the Polynomial Equation Model for Biogas Production

Factor	SS	df	MS	F
(1) Temperature(°C) (L)	717.191	1	717.191	2.080
Temperature (°C) (Q)	741.064	1	741.064	2.150
(2) Time (min) (L)	3603.901	1	3603.901	10.454
Time (min) (Q)	1690.860	1	1690.860	4.905
1L by 2L	313.796	1	313.796	0.910
Error	1378.999	4	344.750	
Total	7817.633	9		20.498

3.3. Percent Yield of CH₄ in Biogas

Testing of the biogas composition, namely CH₄ and CO₂ gas, was carried out at the Chemical Engineering Laboratory, Gadjah Mada University, Yogyakarta. Measurement of CH₄ and CO₂ composition was carried out using GC-TCD.

In Figure 5, it is shown that the value of CH₄ gas content in the variables with pretreatment hot water bath (B1 – B10) resulted in a higher % CH₄ compared to those without pretreatment (A). The optimum result of % CH₄ reached 67.27%, which was achieved in the hot water bath of pretreated pineapple peel waste at a temperature of 100 °C for 25 minutes. Meanwhile, pineapple peel waste that was not pretreated in the hot water bath gives 60.19% CH₄. This indicates that the hot water bath pretreatment of pineapple peel waste affects the amount of CH₄ content in biogas. It can also be seen in Figure 5 that the highest % CH₄ results are, on average, produced by variables with temperature variations at the shorter pretreatment time compared to pretreatment with a long time.

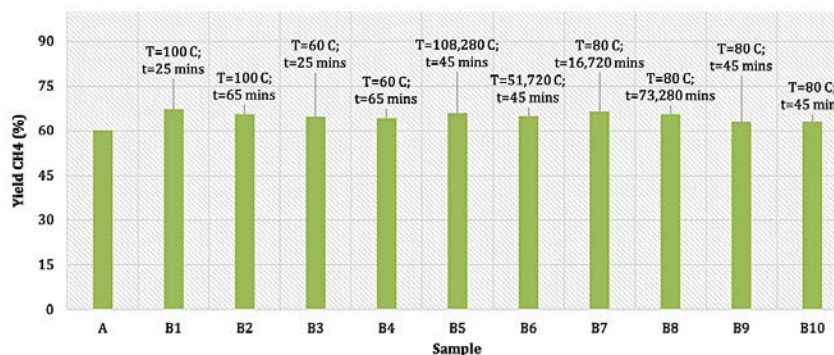


Figure 5 Content of CH₄ (%)

The % yield of CH₄ results was identical to the research conducted by (Gonzalez et al., 2014) on sugar cane peel waste by pretreatment hot water bath at 150°C for 20 minutes with the mesophilic batch process, which yielded 63% of CH₄.

In Figure 5, variable A (without pretreatment), shows the % yield of CH₄ is 60.19%, while in variable B1 (temperature 100 °C, time 25 minutes), the % yield for CH₄ is 67.27%. The lignin content contained in pineapple peel waste was high due to a lack of pretreatment in variant A, which causes less degradation of cellulose by microorganisms.

Meanwhile, the lowest % yield of CH₄ was generated among variables pretreated outside the control variables, namely variables B9 and B10 (temperature 80 °C, time 45 minutes) of 62.94%. This result was lower than the variable B6 (temperature 51.72 °C, time 45 minutes), which was 64.84%. The variation is due to the presence of toxic compounds formed during thermal pretreatment of biogas production. Based on (Rafique et al., 2010), the increasing the temperature will dramatically reduce the methane yield in some cases. This may be due to the formation of complex and toxic compounds such as phenolic and furfural acids.

3.4. Analysis of Lignin, Cellulose, and Hemicellulose

Pineapple peel waste was thermally pretreated to dissolve the lignin so as to assist anaerobic microorganisms responsible for converting polymers in the form of cellulose and hemicellulose into cellulose biogas. The results show that the lignin content was high for variable A (without pretreatment), while variable B1 – B10 with pretreatment showed low lignin content. This indicates that the pretreatment process reduces lignin content, which it helps the microbes to access the cellulose and hemicellulose in the anaerobic digestion.

According to (Rafique et al., 2010), the determination of optimal temperature depending on the medium and the method that varies from about 100-250 °C. However, the drastic increase in temperature reduces the methane yields. This may be due to the formation of complex and toxic compounds such as phenolic acid.

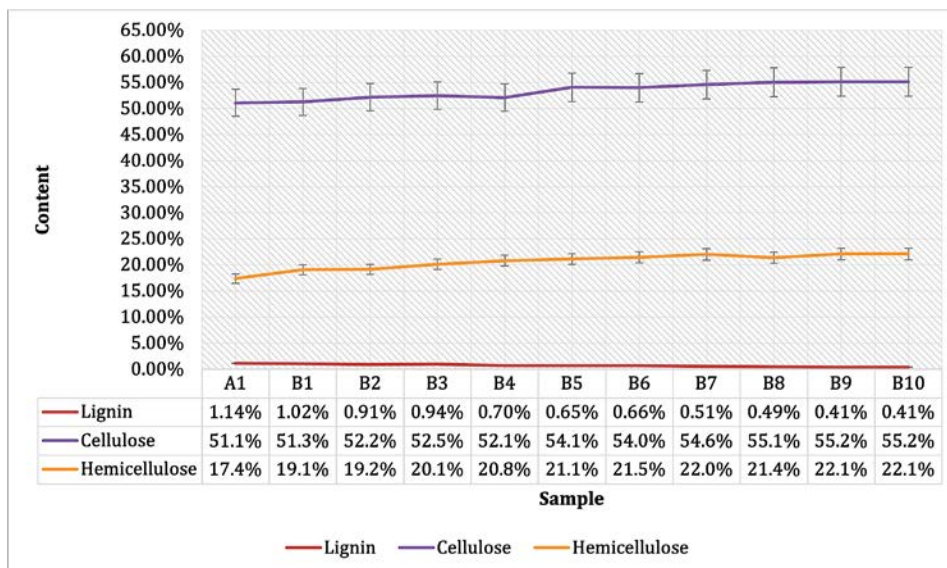


Figure 6 Level of Lignin, Hemicellulose, and Cellulose

In Figure 6, the lowest lignin content was in variables B9 and B10 (temperature 80 °C at 45 minutes), which was 0.41% for both samples, compared to variable B1 (temperature 100 °C at 25 minutes) which is higher in the amount of 1.02% but still lower than the lignin content in variable A (without pretreatment). Higher pretreatment temperature results in higher lignin breakdown, resulting in easy accessibility of cellulose and hemicellulose (Matsushita et al., 2004) reported that an increase in lignin levels occurs due to its

structure, which causes its molecule to condense and settle partially. The molecular weight of lignin increases as a result of the accumulation of lignin molecules.

The analysis showed a decrease of 17.4% hemicellulose in lignin levels of variable A (without pretreatment), while variables B9 and B10 had the highest hemicellulose content of 22.1%, with the mean of the pretreatment variable increasing.

Cellulose analysis results showed that cellulose increase with a decrease in lignin levels with the average value of the differences in the amount of 52.66%. Variable A (without pretreatment) contained 51.1% cellulose while variables B9 and B10 had the highest cellulose content, which was 55.2% for both samples. The increase in cellulose was due to the pretreatment of lignin, which increases the surface area for bacteria to act upon it.

4. Conclusions

This study used thermal pretreatment using a hot water bath with pineapple peel waste as the raw material. The results indicate that pretreatment of pineapple peel waste with hot water bath significantly affects the CH₄ content in biogas. In contrast to the analysis of lignocellulose in lignin, hemicellulose, and cellulose in variable A (without pretreatment), which revealed a high lignin concentration, the lignin content decreased in variables B1 – B10 with pretreatment. This result indicates that the pretreatment process affects lignin yields, affecting the amount of cellulose and hemicellulose digested by microorganisms. The CH₄ gas content in hot water pretreated bath (B1 – B10) resulted in a higher % CH₄ than those without pretreatment (A). The optimum results of % CH₄ reached were 67.27%, which was achieved in pretreated hot water bath of pineapple peel waste at a temperature of 100° in 25 minutes. Future works may consider comparing the process and method of thermal pretreatment with other pretreatments such as mechanical and chemical pretreatment. The results can determine which pretreatment method is the best pretreatment to increase the yield of production and obtain the purest CH₄ content without impurities in the produced biogas.

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