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Ultrasonic pretreatment on biogas production from wood-dust mahogany (swietenia mahagoni) with solid-state anaerobic digestion method: effect of time and pretreatment temperature

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Abstract. The availability of fossil energy as the main energy source makes its availability running low. One alternative energy source is biomass and it can be converted into biogas through a solid-state anaerobic digestion process. One of the many biomass types found in Indonesia is the wood-dust mahogany. The content of lignin contained in wood-dust mahogany makes the process of hydrolysis runs slowly. This causes the biogas production rate to be slow. This study examined the effects of temperature and duration of ultrasonic pretreatment on the characteristics of wood-dust mahogany and the rate of biogas formation along with kinetic study. This research was conducted in three stages, beginning with characterization stage to know the character of wood-dust mahogany, followed by ultrasonic pretreatment stage with time variation (10,20,30) minutes without heating and with heating temperature 70℃. The last stage was fermentation by using bioreactor for 60 days. The biogas produced was measured volume in every two days and the data obtained were used to create a biogas production kinetics simulation by using Gompertz and Richards modification models. The results of the evaluation indicated that the ultrasonic pretreatment process leaded to changes in surface morphology and wood-dust mahogany structure has a positive impact on increasing the rate of biogas formation. The longer the time and the higher the temperature of the ultrasonic pretreatment leaded to an increase in the rate of biogas production. The kinetic model of Richards modification equation gave the result of R2 0.999-0.985 value better than Gompertz modification equation model which is only 0.998-0.968.
Keywords: Solid-state anaerobic digestion, mahogany, ultrasonic, Gompertz equation, Richards equation

1. INTRODUCTION

The availability of fossil energy as a primary energy source has dropped dramatically in the last two decades as a result of large-scale exploitation, while new energy reserves are beginning to be rare. On the other hand, the use of fossil energy such as petroleum, natural gas and coal produces CO_2, NO_x, and SO_x exhaust gases that cause global warming, environmental damage, and problems for human health [1]. Therefore, many studies are conducted to find alternative renewable energy sources that are environmentally friendly [2].

Biomass is a valuable alternative energy around the world as a substitute for fossil fuels, as it can be converted into various forms of energy that can be used such as heat, steam, electricity, biogas and transport

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biofuels such as biodiesel, ethanol and methanol [3]. One of the most common examples of biomass in Indonesia is wood-dust waste. Total Indonesian sawn timber production reaches 38.61 million m³ per year. Assuming that the amount of waste formed is 54% of the total production, the resulting sawmill is 20.84 million m³ annually [4].

The development of biomass conversion technology into biogas is beneficial for increasing energy security, reducing greenhouse gas emissions and utilizing renewable resources. One method of converting biomass to biogas is the anaerobic digestion method. Solid-state anaerobic digestion (SS-AD), it is claimed to be better than liquid anaerobic digestion due to smaller size reactors, requires less energy for heating, simpler material preparation, and less energy loss possibilities [5–7].

The lignin content found in the lignocellulose biomass waste causes the hydrolysis process to run slowly and cause the biogas production rate to be slow as well. It is, therefore, necessary to attempt to degrade the lignin content in the wood-dust. This can be done by pretreatment of the saws waste before the process of solid-state anaerobic digestion. The novelty of this research is the use of ultrasonic waves to pretreat mahogany sawdust before the fermentation process in biogas making. The purpose of this paper is to examine the effect of duration and temperature of ultrasonic pretreatment on the wood-dust mahogany waste substrate and the resulting biogas yield along with its kinetic review.

2. RESEARCH METHODS

2.1. Material

Wood-dust Mahogany was obtained from plywood processing company located in Temanggung, Central Java. The inoculum used was rumen cow fluid obtained from the slaughterhouse (RPH) located in Semarang city and as a nutrient used urea production of PT. Petrokimia Gresik. Tabel 2.1 shows the characteristic of wood-dust mahogany, rumen fluid and urea used in this experiment.

Table 2. 1 Characteristic of wood-dust mahogany, rumen fluid and urea

Parameter	Unit	Wood-dust Mahogany	Rumen Fluid	Urea
Total solid (TS)	%	88,5	1,71	99,5
Volatil solid (VS*)	%	86	1,5	99,5
Total organic carbon (TOC*)	%	31	10	20
Total Kjeldahl nitrogen (TKN*)	%	0,34	0,56	46
Ratio C/N	-	91,1	17,86	0,43

2.2 Equipment

Ultrasonic pretreatment is performed by using SunShine SS-6820C Ultrasonic Cleaner tool at 100 W power and 40 kHz frequency. The fermentation tool circuit Figure 2.1 consists of biodigester targets, rubber valves, valves and HDPE hoses and measuring cups.

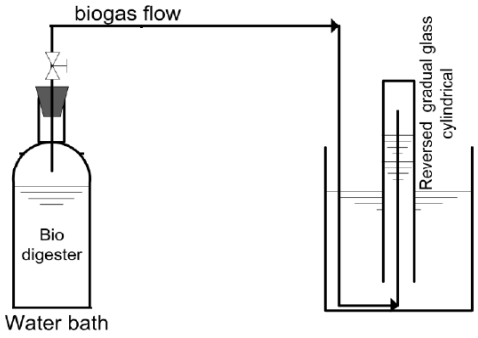


Figure 2. 1 Equipment Setting

2.3 Methods

2.3.1. Stage of Preparation and Ultrasonic Pretreatment. At this stage the sample of wood-dust mahogany was treated in order to obtain uniform size and water content. In the next stage characterization was carried out to determine the initial content of wood-dust. Then wood-dust pretreatment duration of 10 minutes, 20 minutes, and 30 minutes without heating and heating with 70℃

2.3.2. Phase of Operation (making of biogas). Fermentation was carried out for 60 days by using 2 L digester with a working volume of 1 L, with batch operation at neutral pH, ambient temperature (25℃ 35℃) and atmospheric pressure. With a C / N ratio of 30/1, TS content of 20% and 30% inoculum.

2.3.3. Modeling kinetics. This study compared between the Gompertz modification equation with the Richards modification equation, which model gives the best results closest to the experimental data.

Gompertz modification equation:

$$y = A.\exp\{-\exp\left[\frac{U_e}{A}(\lambda - t) + 1\right]\} \qquad (1)$$

Richards's modification equation:

$$y = A\{1 + v\,.\exp(1 + v).exp\left[\frac{U}{A}.(1 + v)^{\left(1+\frac{1}{v}\right)}.(\lambda - t)\right]\}^{-1/v} \qquad (2)$$

Where y is cumulative in biogas production (mL), A is the biogas production potential (mL/gVS), U is the maximum speed of biogas production (ml/gVS.day), λ is the lag phase period (minimum time to produce biogas)), and t is the cumulative time for biogas production (days). The Richards modification equation incorporates the fourth parameter (v) which allows flexibility in the form of curves. The value of each model parameter is calculated using Polymath 6.10 Professional software.

2.3.4. Analytical Procedure. Volume analysis of biogas produced by measuring the volume of biogas every day and starting from day 0 (zero) to the day where biogas is no longer formed. Measurements were made by using the Water Displacement method. Morphology of wood-dust mahogany surface was observed by using SEM (Scanning Electron Microscope) EDX Jeol JSM-6510LA Japan. The structure of wood-dust mahogany was observed with FourPro Transform Infrared Spectroscopy (FTIR) Thermo Scientific Nicolet iS 10.

3. RESULT AND DISCUSSION

3.1 Effect of Ultrasound Pretreatment Against Morphology

Pretreatment using ultrasonic waves caused a morphological change in the surface of wood-dust mahogany. The result of analysis using SEM (Scanning Electron Microscope) can be seen in Figure 3.1 below.

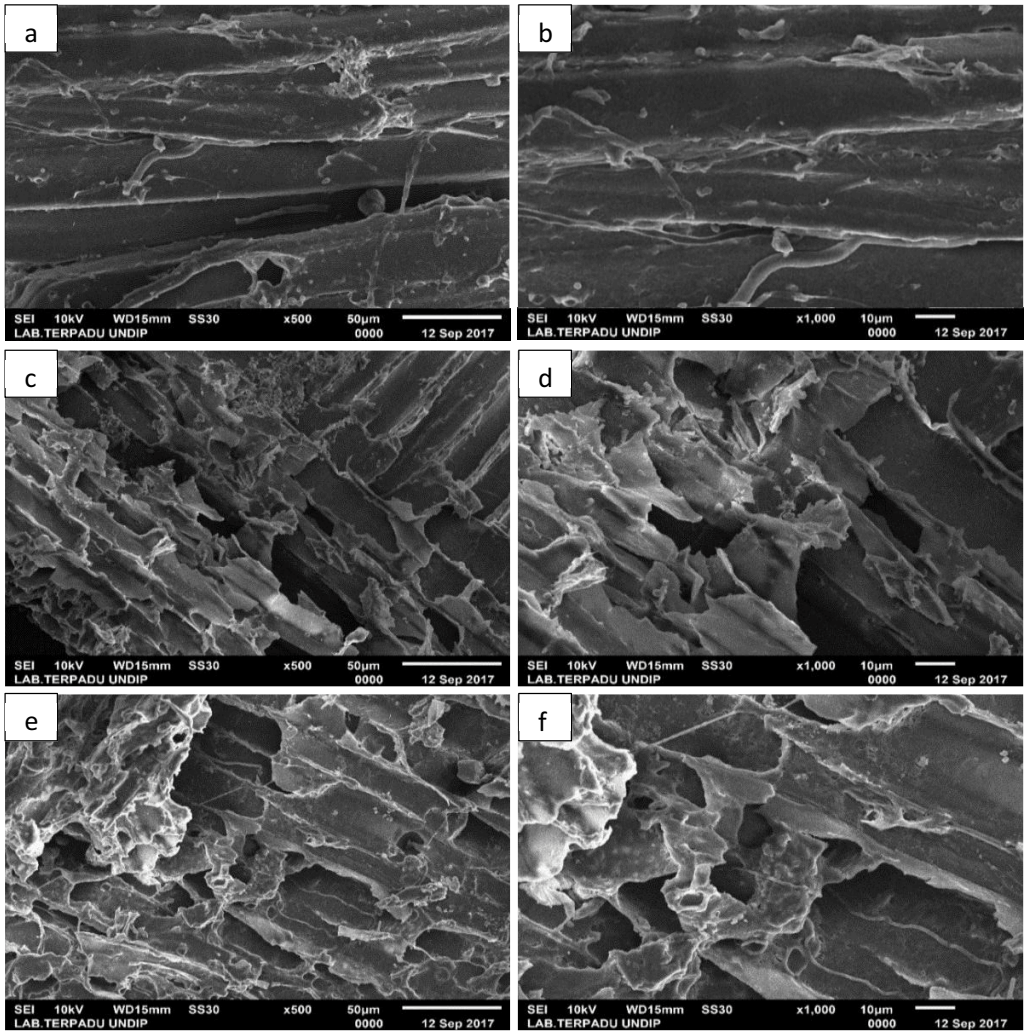


Figure 3.1 SEM analysis of mahogany sawdust without pretreatment (a and b), mahogany sawdust with ultrasonic pretreatment (c and d), mahogany sawdust with ultrasonic pretreatment at 70 ° c (e and f).

Figure 3.1 shows the morphology of sawdust mahogany surface before pretreatment with pretreatment. Figures (a) and (b) are the result of SEM sawdust analysis of mahogany wood without pretreatment. In the picture, the structure of sawdust mahogany is still neat, orderly and strong. The sawdust mahogany is a lignocellulose waste composed of cellulose, hemicellulose and lignin binding to cellulose and hemicellulose bound to one cross-linking so that it becomes strong [8].

Figs. (c) and (d) are the result of SEM sawdust analyzed by ultrasonic pretreatment. From the picture, it is seen that there has been a change in the morphology of the wood-dust mahogany surface, the lignin structure that binds cellulose and hemicellulose looks damaged and broken, the bonds begin to slip off and there are holes that make the surface no longer tight. This occurs because ultrasonic pretreatment produces

sonochemical and mechanoaqoustic effects that affect the chemical and physical structure of lignocellulose through the cavitation process [9].

Fig. (e) and (f) are the result of SEM wood-dust mahogany analysis which has been done ultrasonic pretreatment at 70 ° C. from the picture, it is seen morphological structure of wood-dust surface of mahogany damaged and broken as in picture c and d result of pretreatment ultrasonic at room temperature. However, the damage caused was more evenly and pore becomes bigger. This occurs because when the water was heated at 70°C the viscosity was reduced so that cavitation is more likely to occur and gives a greater penetration effect on the cross-linking of lignocellulosic polymers, resulting in greater damage than the ultrasonic pretreatment at low temperatures [10].

3.2. Effect of Ultrasonic Pretreatment on Wood-dust Mahogany Structure

FTIR analysis was performed on wood-dust mahogany without pretreatment, mahogany sawdust with ultrasonic pretreatment and mahogany sawdust with ultrasonic pretreatment at 70 ° C.

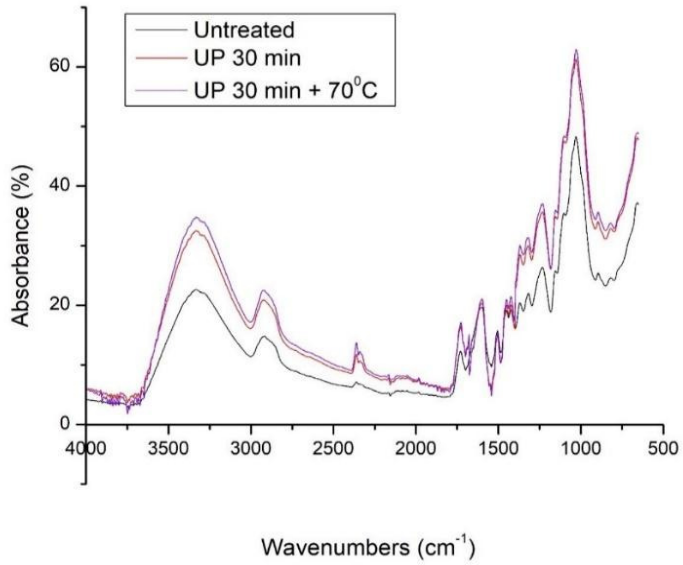


Figure 3.2 Results of FT-IR analysis of wood-dust mahogany before and after pretreatment.

Based on the results of FTIR analysis in Figure 4.2 it is known that the wavelength of C = C lignin group ranges between 1465 - 1610 cm-1. At the bend of Wavelength 1465 - 1610 cm-1 there is a decrease of C = C group this occurs because pretreatment using ultrasonic can damage carbon-carbon bond in lignin molecule due to cavitation effect arising from ultrasonic wave causing water molecule to hit lignin molecule and cut carbon-carbon bonds [16].

From Table 3.1, it can be seen that crystallinity index is considered as A1430/A898, where Wavelength 1430 and 898 are cellulose I and cellulose II [16]. Crystallinity index of wood-dust mahogany without pretreatment was 0.815. After pretreatment by using ultrasonic crystallinity index was decreased to 0.698 and 0.678 after ultrasonic pretreatment at 70 °C [11].

Total crystallinity index considered as A1375/A2900 on wood-dust mahogany without pretreatment was 1.54. The total crystallinity index decreased after ultrasound pretreatment to 1.32 and 1.308 after ultrasonic pretreatment at 70 °C. This is because the amount of lignin from wood-dust mahogany was reduced due to the pretreatment [12].

3.3. Effect of Ultrasonic Pretreatment on Biogas Production

The effect of ultrasonic pretreatment on the production of wood-dust mahogany biogas with ultrasonic pretreatment without heating and by heating 70℃ can be seen in Figure 3.3. Total production of wood-dust mahogany biogas with ultrasonic pretreatment with heating 70 ℃ was higher than without heating, it can

be seen variable UP 10 minutes total biogas production of 94 mL/g.TS, whereas in variable UP 10 minutes with heating 70 ℃ reached 146 , 5 mL/g.TS greater than 55%. In variable UP 20 minute total of biogas production equal to 131,5 mL/g.TS, whereas in variable UP 20 minute with heating 70 ℃ reach 161,5 mL / g.TS bigger 22%. In variable UP 30-minute total of biogas production equal to 183,5 mL / g.TS, while at variable UP 30 minute with heating 70 ℃ reaching 199,5 mL / g.TS bigger 8%.

Table 3.1 Characteristics and wavelength variations of FTIR spectra of sawdust of mahogany wood

Wave Length (cm-1)	Functional cluster	Assignment	Control	UP 30 min	UP 30 min + 70°C
3175	- OH stretching intramolecular hydrogen bonds	Cellulose II	0.176	0.207	0.224
2900	C - H stretching	Cellulose	0.137	0.175	0.185
1740	C = O stretching of acetyl or carboxylic acid	Hemicellulose and lignin	0.113	0.119	0.124
1610	C = C stretching of the aromatic ring	Lignin	0.194	0.162	0.166
1598	C = C	Lignin	0.196	0.165	0.171
1510	C = C stretching of the aromatic ring	Lignin	0.152	0.113	0.111
1465	asymmetric bending in C - H3	Lignin	0.172	0.138	0.146
1430	C - H2 bending	Cellulose	0.198	0.217	0.227
1420	C - H2 symmetric bending	Cellulose	0.202	0.221	0.232
1375	C - H bending	Cellulose	0.211	0.231	0.242
1335	- OH (in plane bending)	Cellulose	0.223	0.236	0.249
1315	C - H2 wagging	Cellulose	0.239	0.248	0.26
1158	C - O - C astmmetric stretching	Cellulose	0.255	0.29	0.298
898	asym., out of phase ring stretching (cellulose)	Cellulose	0.243	0.311	0.335

Pretreatment condition	Crystallinity index (A1430/A898)	Total crystallinity index (A1375/A2900)
Without pretreatment	0.815	1.540
Pretreatment ultrasonic for 30 min	0.698	1.320
Pretreatment ultrasonic for 30 min at 70°C	0.678	1.308

From Figure 3.3 it is known that ultrasonic pretreatment without heating or heating does not affect the rate of biogas formation in the early phase of fermentation (lag phase), but it has a significant effect on increasing the rate of biogas formation in the exponential phase and stationary phase when compared with control variables, and this result consistent with previous studies [10,13,14]. The duration of ultrasonic pretreatment time has a positive effect on increasing the rate of biogas formation. The longer the ultrasonic pretreatment performed on the substrate, the more the rate of biogas formation produced. The temperature of the ultrasonic pretreatment also affects the production of biogas, the higher the temperature when pretreatment is done the resulting biogas is also greater [15].

Increased biogas production in wood-dust mahogany has undergone ultrasonic pretreatment with heating or non-heating compared without pretreatment occurs because the structure of wood-dust

mahogany becomes rougher and its lignin content decreases after the ultrasonic pretreatment so the relative area increases. Another cause is soluble substances dissolved on wood-dust mahogany substrate which result in rapid oxygen consumption in the reactor by aerobic and facultative aerobic bacteria causing increased methanogenic bacteria [16].

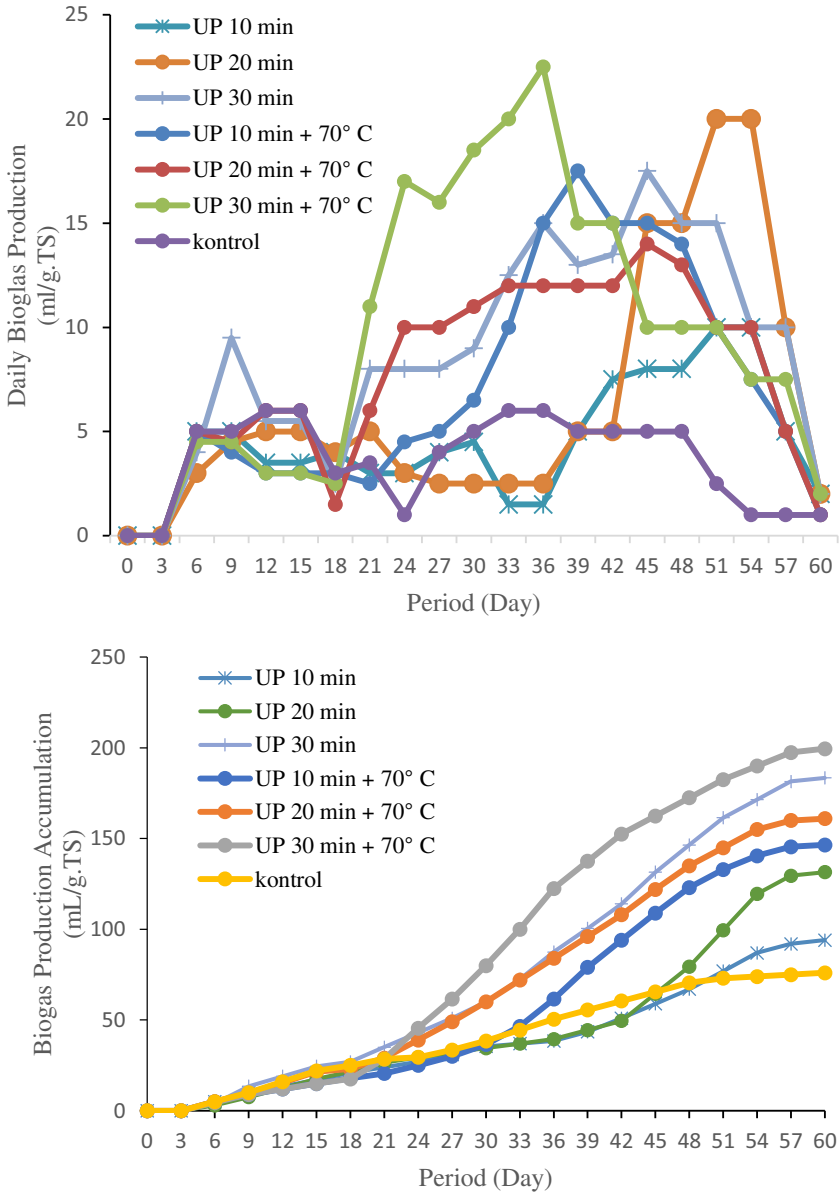


Figure 3.3 Accumulation and production of biogas of wood-dust Mahogany with ultrasonic pretreatment (10, 20, 30) minutes, and ultrasonic pretreatment (10, 20, 30) minutes on 70° C

3.4 Biogas Production Kinetic Model

Assuming the speed of biogas production in batch conditions related to the specific growth rate of methanogenic bacteria in biodigester, the biogas production rate is predicted to follow the Gompertz modification equation and Richards modification equation [17]. The kompathic parameters of the Gompertz modification equation and the Richards modification present at Table 3.2.

The parameter value A of the control variable of the modified equation model Gompertz 95,35 (mL/gVS) and the Richards modification equation model 126.19 (mL/gVS) was smallest compared to other variables, indicating that ultrasonic pretreatment can increase biogas production potential from substrate of wood-dust mahogany. In the equation model modification Gompertz and Richards modification equation model shows the value of U on the smallest control variables are 1.69 (mL/gVS.d) and 1.59 (mL/gVS.d) while the highest U value is in the variable UP 30 min + 70 ℃ ie 6.23 (mL/gVS.d) and 6,523 (mL/gVS.d). These results prove that ultrasonic pretreatment can increase the maximum speed of biogas production. In the variable performed by ultrasonic pretreatment, the ultrasonic pretreatment progressively decreases the λ value, which means that by doing ultrasonic pretreatment can accelerate the initial time of biogas formation [18].

The Richards modified equation kinetics model gives better results than the Gompertz modification equation model. It can be seen from the value of R2 of the two equation models, the Richards equation model has a R2 value of 0.999-0.985 better than the R2 equation of the Gompertz modified equation of 0.998 -0.968 this is due to the Richards modification equation model combining the fourth parameter (v) which allows flexibility in the form of curves when faced with possible biogas production curves that vary from the shape of the prolonged sigmoidal S-shape or the inverted L-shape [19].

Table 3.2 The kompathic parameters of the Gompertz modification equation and the Richards modification

Parameter	Kontrol	UP 10 min	UP 20 min	UP 30 min	UP 10 min + 70℃	UP 20 min + 70℃	UP 30 min + 70℃
Modification Model Gompertz							
A (mL/gVS)	95,35	436,19	506,00	326,75	229,18	235,59	223,65
U (mL/gVS.d)	1,69	2,93	4,39	4,47	4,10	3,98	6,23
λ (days)	5,37	27,35	30,14	16,09	19,67	14,54	16,73
R^2	0,991	0,987	0,968	0,996	0,989	0,997	0,998
Yield biogas measured (mL/gVS)-60 days	76	94	131,5	183,5	146,5	161	199,5
Yield biogas prediction (mL/gVS)-60 days	78,50	97,55	131,93	191,93	156,28	168,08	201,86
Modification Model Richards							
A (mL/gVS)	126,19	1635,32	131,28	221,67	148,75	190,04	208,95
U (mL/gVS.d)	1,59	4,26	6,31	4,94	5,55	4,35	6,52
λ (days)	3,52	49,28	35,94	18,29	25,28	16,52	17,69
v	-0,60	-0,33	33,96	0,98	2,98	0,69	0,53
R^2	0,992	0,988	0,985	0,997	0,998	0,998	0,999
Yield biogas measured (mL/gVS)-60 days	76	94	131,5	183,5	146,5	161	199,5
Yield biogas prediction (mL/gVS)-60 days	79,71	97,22	131,28	187,81	146,63	165,04	198,70

4. CONCLUSION

Non-heating or ultrasonic pretreatment causes morphological changes and wood-dust mahogany structure. After pretreatment using ultrasonic crystallinity index was decreased from 0.815 to 0.698 and 0.678 after ultrasonic pretreatment at 70 °C. The total crystallinity index also decreased from 1.54 to 1.32 and 1.308 after ultrasonic pretreatment at 70 °C. Non-heating or ultrasonic pretreatment did not affect the rate of biogas formation in the early phase of fermentation (lag phase), but has a significant effect on increasing the rate of biogas formation in the exponential phase and stationary phase when compared with control variables. The Richards modified equation kinetics model gave better results than the Gompertz modification equation model. It can be seen from the value of R2 of the two equation models, the Richards equation model has a R2 value of 0.999-0.985 better than the R2 equation of the Gompertz modified equation of 0.998 -0.968.

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