Optimization of biogas production in Indonesian region by liquid anaerobic digestion (L-AD) method from rice husk using response surface methodology (RSM)

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Abstract. Rice husk is one of agricultural waste that is potentially converted to biogas. In this research, rice husk is used as the material to produce biogas by liquid anaerobic digestion (L-AD) with TS concentration below 15/5. The purposes of this research are finding the optimum value of total solid (TS), C/N ratio, and microbial consortium (MC) for biogas production and then analyzing the optimum biogas production rate from rice husk. Because of the high lignin content, rice husk must be through the pretreatment stage using NaOH 3%. The variations of the observed factor are 5%, 10%, 15% for TS concentration; 20, 35, 50 for C/N ratio; and 3%, 6%, 9% for MC concentration. There are 16 reactors in which the variation for each reactor was determined by Central Composite Design. Data obtained from the research will be optimized using Response Surface Methodology (RSM) in Statistica 10. The result shows that the optimum condition of biogas production from rice husk is TS in between 1%-2.5%, C/N ration 33.45, and MC in between 5.5%-7%. Reactor having a combination of value factors that close to the optimum value will be analyzed using Polymath 6.0, which is reactor 10. The kinetics constant of biogas production rate (u) is 3.625 ml/grTS.day, the maximum biogas production (A) is 277.374 ml/gr TS, and the minimum time needed to produce biogas (λ) is 3.878 days.

1. Introduction
Rice husk is one of lignocellulose biomass that can be used as an alternative energy source that is not optimally used. In Central Java, the rice production in the year of 2015 is 11.006.570 tons [1] so that the amount of rice husk produced is around 2.202.314 tons with the assumption of 20% rice production [2]. Biogas, one of a method to transform biomass into energy, is a gas mixture produced from the decomposition of organic compounds by methanogenic bacteria in anaerobic conditions [3]. Before used as the material to produce biogas, lignocellulose material generally will be going through the pretreatment stage to increase the material digestibility [4].

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Various research has been conducted by the former researcher about the effect of TS and C/N ratio on biogas production. Based on the former study, the highest biogas production from rice husk will be obtained if the C/N ratio 35 [5] and a total solid 9% [6] for L-AD method. The microbial consortium consists of Streptomyces sp., Trichoderma sp., and Geobacillus sp. which can help the degradation process of cellulose, hemicellulose, and lignin [7,8,9].

Based on those researches, further research is needed to find the optimum value of the factor to gain optimum biogas production. In this research, the optimization of the biogas production will use response surface methodology (RSM) which the factors are total solid, C/N ratio, and microbial consortium. This research intends to determine the optimum value of total solid, C/N ratio, and microbial consortium for biogas production from rice husk with L-AD method.

2. Materials and method

This research is divided into 2 stages. The first stage is a preliminary test, and it is to know the characteristic of the materials used. For determining total solid of rice husk and cow manure, we used NREL/TP-510-42621 about Determination of Total Solids in Biomass, and Total Dissolved Solids in Liquid Process Samples and the value of C-organic was determined by walkey and Black method, while for the value of N-total, we used spectrophotometry method.

The reactor used in this research is a 1-liter plastic bottle equipped with rubber as the bottle cap. The reactor is connected to a 3/16 inch plastic hose used for gas transferred in the measurement. Materials used in the research are rice husk collected from a granary in Rowosari, Tembalang district, rumen cow fluid as inoculum obtained from Penggaron slaughterhouse in Pedurungan District, Semarang, the microbial consortium is in Decoprima packing powder, and urea obtained from the farm shop in Karangrejo, Banyumanik district. All the materials used are the same as the former researcher [5,6].

There are 3 factors in this research, which are total solid with variations of 5%, 10%, 15%; C/N ratio with variations of 20, 35, 50; and microbial consortium with variations of 3%, 6%, 9%. Optimization of the 3 factors was conducted using response surface methodology (RSM) in central composite design (CCD). Based on data processing by RSM, there are 16 reactors used in the research with a variation of the factors in every reactor will be showed in table 1.

The study was conducted for 60 days with the gas measurement was every two days. It aimed to keep the reactor in the anaerobic condition and there was no significant biogas production every day. Biogas volume was observed by transferring the gas into the measuring glass and the amount of gas produced was calculated from the difference of the water volume in the measuring glass.

After 60 days of biogas observation, data obtained from the observation was analyzed using response surface methodology (RSM) in Statistica 10 to determine the optimum value of each factors. Besides that, the reactor with the highest biogas production was analyzed using Polymath 6.0 to determine the rate of biogas production.

![Schematic diagram of series laboratory batch assessment of L-AD.](image)

**Figure 1.** Schematic diagram of series laboratory batch assessment of L-AD.
3. Results and discussion

3.1. Optimization of total solid, C/N ratio, and microbial consortium

The result of 60 days observation can be seen in Table 1. As the data shown in the table above, each reactors has a different amount of biogas produced. The highest biogas production was conducted in reactor 10 with 192.453 ml/grTS. These observation result was analyzed with RSM in Statistica 10 resulting an equation as follows

\[
Y = 57.0878 - 48.1543x_1 + 16.2198x_2 + 7.7355x_1^2 - 12.3509x_2^2 + 0.6298x_3 - 1.5233x_1x_2 - 3.8292x_1x_3 - 3.6375x_2x_3
\]  

(1)
Y = Biogas Production (mL/gr TS)  
X1 = Concentration of Total Solid (%)  
X2 = C/N Ratio  
X3 = Concentration of microbial consortium (%)  

Based on the equation, the value with the positive sign means that the increasing amount of variables both linear (L) and quadratic (Q) will give a positive impact or increase biogas production. Meanwhile, the value with the negative sign both linear (L) and quadratic (Q) will decrease biogas production. Therefore, the coefficient of X1 (L) is in negative sign, it means that the increase of total solid linearly will decrease the biogas production but the increasing of total solid quadratically will increase the biogas production because the coefficient of X1 (Q) has a positive sign. The second variable is X2 which X2 (L) is in a positive sign that means the increasing C/N ratio linearly will increase the biogas production but the increasing C/N ratio quadratically will decrease the biogas production. The last variable is X3 which X3 (L) is in positive sign and X3 (Q) is in negative sign, that the increase of microbial consortium linearly will increase the biogas production.

Figure 2 shows the level of significance for each variable and the interaction between two variables. The significance level for all variables is 98.41%. The red line in the Pareto chart is the value of $P = 0.05$. In Figure 2, the variable significantly affects if the variable chart passes the red line which the value of $P<0.05$. On the contrary, if the chart does not pass the red line, it means the variable does not significantly affect the biogas production with the value of $P>0.05$. The following figure is the Pareto chart.

Based on the Pareto chart, the variables affecting the biogas production are total solid (linearly and quadratically), C/N ratio (linearly and quadratically) and the interaction between the variable of total solid and C/N ratio linearly because the chart passes the red line ($P<0.05$). The other variables like microbial consortium either linearly or quadratically and the interaction between total solid and microbial consortium also between C/N ratio and microbial consortium does not significantly affect the biogas production cause the chart does not pass the red line or the $P>0.05$.

The optimum condition of total solid, C/N ratio, and microbial consortium can be determined by the response surface analysis in the surface plot (3 dimensions) and contour plot chart (2 dimensions). Either the surface plot or the contour plot chart, it consists of 2 independent variables and one dependent variable, cumulative biogas production (ml/grTS), so that the other one variable will be a constant value. The two diagrams show the same analysis, but it is easier to determine the optimum range of variables in the contour plot chart (2 dimensions). In the chart, there is various colors showing the amount of biogas production. The more red the color the greater the amount of biogas production. It can be seen in figure 3 until figure 5.

The following figures 4 and 5 show the surface plot and contour plot between total solid and C/N ratio on 6% MC concentration and between total solid and MC concentration on C/N ratio 35. Based on Figures 3 and 4, it can be seen that the optimum value for the variable total solid is under 10%. This result is the same as the previous research stating that the optimum amount of total solid is 5% [10]. Another reference state that excess substrate value fed into the reactor will increasingly activate and accelerate the growth of acidogenic and acetogenic bacteria leading to an imbalance stage of acidogenesis and acetogenesis [11]. Meanwhile, the organic material that has a low total solid content will be degraded faster leading to un-optimal performances of methanogenic bacteria on producing biogas because of low pH.

Figures 4 and 6 show the surface plot and contour plot between total solid and C/N ratio on 6% MC concentration and between C/N ratio and MC concentration on 10% total solids. Based on the diagram, the optimum value of C/N ratio is in the range of 30 – 40, with the specific value is 33.45. As well as the former research stating that the optimum value of C/N ratio is 35[5]. The other research also stating if value of C/N ratio more than 43, it will inhibit the bacteria performance [12]. If the value of C/N ratio is too high, nitrogen will increase bacterial growth reacting to carbon that gives a negative effect on biogas production [13]. On the contrary, the inadequate value of C/N ratio will lead to nitrogen accumulation in the form of ammonia, affecting pH increasing [13].
Figures 5 and 6 show the interaction between total solid and MC concentration on C/N ratio 35 and the interaction between C/N ratio and the microbial consortium on 10% total solid. It shows that the optimum value of MC concentration is in the range of 5.5% - 7%. Based on the reference, Trichoderma sp. and Geobacillus sp. can produce cellulase enzyme but Geobacillus sp. is thermophilic bacteria [7,9] and Streptomyces sp. can produce ligninase enzyme [8]. Therefore based on data analyzing, it can be concluded that the optimum value for total solid is under 10%, for C/N ratio is 33.45, and the last for MC concentration is in the range of 5.5% - 7%. Reactor 10 shows the highest biogas production and has the closest value with the optimum value with TS 1.54%, C/N ratio 35, and MC 6%.

3.2. Biogas production optimum rate from rice husk with L-AD method

Reactor 10 has the closest value to the optimum variable value with 1.54% total solid, C/N ratio 35, and 6% MC concentration. Cumulative biogas production of reactor 10 then calculated its kinetic constant of biogas production rate (U) (ml/gr.TS.day), maximum biogas production (A) (ml/gr.TS), and time needed to produce biogas (λ) (day) using Polymath 6.0. From the calculation, it is obtained the following results.

Table 2. Kinetic constants for optimum biogas production.

| Constants     | Value |
|---------------|-------|
| U (ml/gr.TS.day) | 3.625 |
| A (ml/gr.TS)    | 277.374 |
| λ (day)         | 3.878 |

The constant values then input to the equation 2 to determine the biogas production with t filled with time (day) to be calculated.

\[
P = 277.374 \exp \left[ - \exp \left( \frac{3.625 + 2.178}{277.374} (3.878 - t) + 1 \right) \right]
\]  

(2)

P = cumulative biogas production (ml/grTS)  
t = time cumulative for biogas production (day)

Figure 3 below is the result of Polymath 6.0 analysis until 184 days, which can be seen that the highest biogas production is achieved on 184th days with the result is 277.355 ml/grTS. After 184 days, biogas is still produced in the reactor but the volume produced only ±0.002 ml/grTS per day, so it does not significantly affect the accumulation of yield biogas. It also can be seen in the graph the R² value is 0.9892, which means that the calculation data from Polymath is not significantly different with the data obtained from the research.
Figure 4. Three dimensional / surface plot (left) and two dimensional / contour plot (right) graphs for variable TS and C/N ratio at 6% concentration of microbial consortium from RSM analysis.

Figure 5. Three dimensional / surface plot (left) and two dimensional / contour plot (right) graphs for variable TS and C/N ratio at 6% concentration of microbial consortium from RSM analysis.

Figure 6. Three dimensional / surface plot (left) and two dimensional / contour plot (right) graphs for variable C/N ratio and MC at 10% total solid from RSM analysis.
4. Conclusions
The maximum biogas production can be obtained at the optimum condition of variables: (1) TS concentration is below 10%, (2) C/N ratio of 33.45, and (3) microbial consortium in the range of 5.5% - 7%. The maximum biogas production rate in the reactor that has the closest value to the optimum variable value is reactor 10 with 1.54% TS, C/N ratio 35, and 6% concentration of MC producing biogas yield of 277.355 ml/grTS. The kinetics constant rate of biogas production per day (U) is 3.625ml/grTS, maximum biogas production rate (A) is 277.374 ml/grTS, and minimum time needed to produce biogas is 3.878 days.

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References
[1] Statistik Nasional Indonesia 2017
[2] Waries A 2006 Jakarta Gramedia Pustaka
[3] Mara I 2012 Dinamika Teknik Mesin 1
[4] Al Seadi, Rutz D and Prassl H 2008 Biogas Denmark
[5] Syafrudin, Winardi D N and Hasahi H A M 2017 Adv Sci. 1012204
[6] Syafrudin, Winardi D N and Erica S S 2017 Jurnal Teknik Lingkungan 6 1
[7] Bardant T B, Abimanyu H and Epriyani P L 2013 Jurnal Kimia Terapan Indonesia 15 35
[8] Gawande P V and Kamat M Y 1999 J. Appl Microbiol. 87511
[9] Potrommanee L, Wang X Q and Han Y J 2017 PLoS One 2 1
[10] Sawasdee V 2014 Energy Procedia 61 1129
[11] Soeprjanto, Ismail T and Lastuti M D 2010 Jurnal Purifikasi 11
[12] Dennis A and Burke P E 2001 Olympia: Environmental Energy Company
[13] Budiyono G, Kaerunnisa and Rahmawati I 2013 Jurnal Teknologi Kimia 11 1