

MODELING OF ANAEROBIC CO-DIGESTION USING ORGANIC WASTE AND MANURE AS SUBSTRATE

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Abstract

This paper aims to investigate the effect of alkaline pretreatment concentration, the composition of organic waste, and organic loading rate on anaerobic digestion of mixed waste, consisting of pig manure and vegetable waste, on the production of bio-methane. Experiments were conducted in a 60 L semi-continuous polyethylene digester at ambient temperature using water displacement technique to monitor gas production. The optimized pretreatment and methane production conditions were found to be 2.0% (w/w) NaOH concentration, an organic loading rate of 1.2 kg VS m⁻³ d⁻¹ and vegetable residual or 20 % in the organic waste. The amount of methane achieved at this optimized condition is 0.691 m³ kgVS⁻¹. A second order polynomial equation was formulated to solve for the amount of methane produced. Predicted acquired from this formula was found to have an R² of 97.7% which indicate that the predicted results were closed to the actual data obtained from the experiment. It can be concluded from the Box-Behnken design experiment that the pretreatment of vegetable residual with NaOH solution has a positive effect on the production of methane.

Keywords: Anaerobic digestion, vegetable residual, mesophilic, Box-Behnken design

Introduction

Over the past few years, it is obvious that the number of the anaerobic digestion power plant has increased dramatically. There are many possible factors that caused this incline in registered anaerobic digestion system such as the economy, organization incentives (Kyoto

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protocol), government's subsidization policies, difficulty in waste disposal management, exploitation of petroleum oil and most importantly the vision to build a cleaner source of energy platform for the future's electric vehicle (Zglobisz *et al.*, 2010). Establishment of an electricity source from biogas is an interesting opportunity, especially in Thailand because unlike other types of alternative energy, biogas technology does not have to be imported and the establishment of a biogas power plant will job to local people. Therefore, the implementation of biogas systems can solve both the economy and energy. For these reasons the Ministry of Energy (Thailand), planned to increase the production of biogas for electricity generation from 311 MW in 2014 to as high as 3000 MW by 2021 (Sathienyanon, 2015; Worasaen *et al.*, 2017). Additionally, the biogas system also helps eliminate the organic type of waste. Currently, a large amount of vegetable residual from community markets in Thailand are being incinerated in government- owned facilities (Nath *et al.*, 1999). Incineration of these organic waste is both an energy- intensive endeavor and releases a large amount of hazardous carbon monoxide due to the high moisture content in vegetable waste (Siriratpiriya, 2014).

Anaerobic digestion is a process where methane is produced by a specific group of micro-organism, which is grown by consuming organic material in an O₂-free environment (Thamsiriroj and Murphy, 2011). In detailed the organic material is initially exposed to the group of micro-organisms that are responsible for hydrolysis reaction, which is the breakdown of organic material to smaller molecule such as amino acids (from proteins) and sugar (from carbohydrate) (Dai *et al.*, 2017; Lindkvist and Karlsson, 2018). Next, these soluble organic compound will be in contact with another group of micro-organism which decomposes the molecule further to volatile fatty acid and CO₂ in a reaction called acidogenesis. The volatile fatty acid is then converted to H₂ and acetate through the acetogenesis reaction. Ultimately, acetate is converted to CH₄ and CO₂ (Li *et al.*, 2018). Biogas obtained from anaerobic digestion is roughly 50-60% CH₄.

Purified biogas can be compressed and used in both the modified diesel and gasoline engine (Cho and He, 2009; Pannucharoenwong *et al.*, 2017).

One of the obstacles in creating high biogas productivity is the structure of organic wastes which contain a high degree of lignin (Steffen *et al.*, 2017). The presence of lignin in the structure makes cellulose less exposed to the active micro-organism, hence the decline in methane formation. Onthong and Juntarachat reported a 70% reduction in biogas production rate when straw with high lignin content was employed as a substrate instead of soybean (Onthong and Juntarachat, 2017). The negative impact of lignin content is also reported by anaerobic digestion of *Salvinia molesta* and rice straw (Syaichurrozi, 2018). Therefore, unlike pig mature, organic wastes that are classified as lignocellulosic material, need to be subject to a pretreatment step first before entering the anaerobic digester (Rodriguez *et al.*, 2014; Patinvoh *et al.*, 2017). Chemical pretreatment is among the most effective technique used to reduce lignin content at low expense relative to a physical pretreatment process, such as ultrasonic, microwave and hydrothermal (Deepanraj *et al.*, 2017; Savoo and Mudhoo, 2018; Wang *et al.*, 2018). A 21.4% improvement in biogas yield was observed from alkaline pretreated rice straw (Mancini *et al.*, 2018). Under the thermophilic condition, an increase in methane production of as high as 30% was observed after grass substrate was reacted with Ca(OH)₂ (Khor *et al.*, 2015). Zheng and Pang reported a significant enlargement in surface area of corn stover after pretreatment with NaOH which lead to an enhancement in methane productivity (Pang *et al.*, 2008; Zheng *et al.*, 2009). So far very little work has been done on optimization of anaerobic digestion through statistical analysis (Echaroj *et al.*, 2017; Thirugnanasambandham (2016); Ye *et al.*, 2016). This is due to the fact that biological oriented experiment required a long- duration experiment.

According to a recent survey, the municipality of Ratchaburi province has to collect 470 tons of waste per day, out of which 37% were disposed of inappropriately (open

dumping, open burning and etc). Implementation of the biogas system can help both energy and waste disposal problem. The objective of this research is to investigate the effect of concentration of the alkaline solution (1%, 1.5%, and 2% w/w NaOH) used during the pretreatment step on biogas production. Optimization of the production rate will also be employed through the Box-Behnken design to evaluate the effect of other operating condition such as the organic loading rate (0.5, 1.0, and 1.5 kgVS m⁻³ g⁻¹), and the composition of organic waste (20%, 40%, and 60% vegetable residual). A statistic procedure based on Box-Behnken design and response surface methodology was adopted for the optimization process, which also provides a highly fitted coefficient for a polynomial equation. Results obtained from the optimization was successfully applied by Ratchaburi's municipality which yields an economic profit.

Methods

Preparation and Pretreatment of Vegetable Residual

Vegetable residuals were collected from a local community market in Ratchaburi province. The size of these organic residuals was reduced to approximately 1 cm² by chopping manually and stored in a zip-locked plastic bag at 4°C before being treated. Pretreatment of chopped vegetable residual was performed in a separate 20L polyethylene tank. Pretreatment solution was prepared to have a concentration of 1%, 1.5%, and 2% w/w NaOH. The pretreatment time for each batch is 3 days. After the pretreatment step, the treated vegetable residual is filtered and added to the anaerobic digestion tank. Anaerobic sludge for a microbial inoculum of the anaerobic digestion was obtained from an open lagoon digester of a local pig farm in Ratchaburi Province. The research methodology is shown in Figure 1.

Anaerobic Digestion: Methane Production Process

The first step to a highly effective biogas production is in the design of the digester tank.

A custom made 60L polyethylene tank (diameter of 45 cm and height of 1 meter) sealed with silicon was design to fulfill a semi-continuous digestion process. The inlet for organic loading is 15 cm in diameter and is submerged under anaerobic sludge at all times during the digestion process to ensure no biogas leakage. A valve was added halfway between the top and bottom of the digester tank to control the sludge level (to optimize the anaerobic process, the content inside the tank should not occupy more than half of the total digester's volume) as shown Figure 2. Another valve was added at the bottom to release anaerobic sludge after the end of each experiment. The anaerobic digestion tank is connected to a water displacement tank to monitor the biogas production rate. The digester was designed so that the organic waste can be fed into the reactor without biogas leakage. Approximately 30 L of anaerobic sludge was added to the anaerobic digester tank to initial the microbial inoculum process. The organic loading rate (OLR) during the start-up phase (the first week) was halved of the desired OLR. For this research, the desired OLR was varied

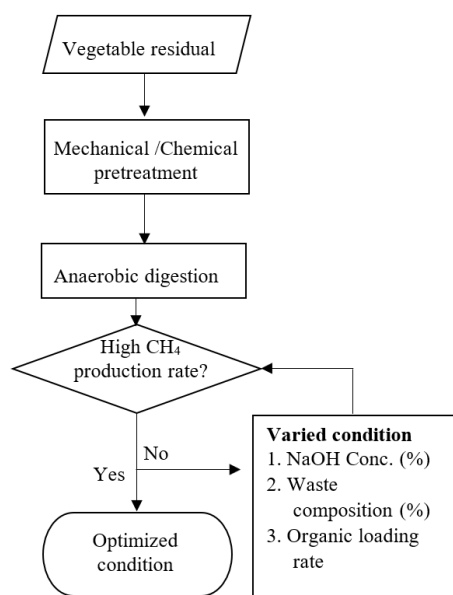


Figure 1. Flow diagram of research concept

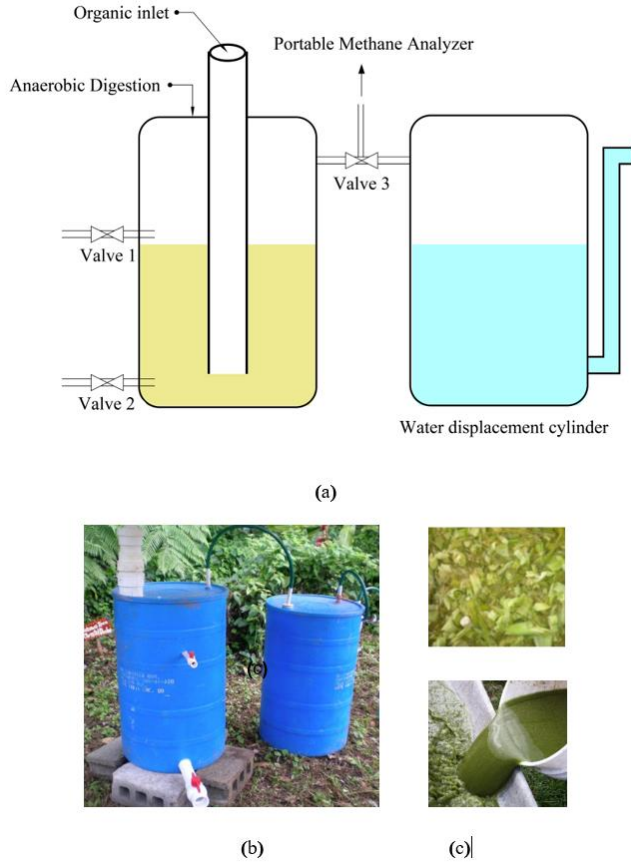


Figure 1. Schematic diagram for the anaerobic digestion system; valve 1 use to control sludge level, valve 2 used to clean digester, valve 3 used as a port to measure biogas production rate (a), constructed anaerobic digester (b), chopped vegetable waste and pig mature (c)

from 0.5 to 1.5 kg VS m⁻³ g⁻¹. The anaerobic digestion was then carried out for another two weeks. Data collected between second and last week of anaerobic digestion were found to be relatively stable. Therefore these values were averaged and used in the optimization model. The amount of methane produced per volatile solid (m³ kg⁻¹ VS) was found by multiplying the methane content with the biogas production rate as shown below.

$$CH_4 \left(\frac{m^3}{kg \text{ VS}} \right) = \frac{CH_4\%}{100} \times \frac{Q_g}{VS_{input}} \quad (1)$$

Where $CH_4\%$ is the percentage of methane inside biogas, Q_g is the flowrate of biogas and VS_{input} is the volatile solid input.

Analytical Methods and Devices

Volatile solids (VS) contents were measured by placing the sample in a calcination oven at 550°C for 3 h. Methane content during anaerobic digestion was measured using a portable biogas analyzer with an accuracy of ±10% (Vapor Analytical Bio Genius - 02). The amount of biogas produced per day is found by measure the volume of water displaced by biogas in the second tank. The pH meter

Table 1. Box-behnken design tabulated with experimental and predicted results based on the second-order polynomial equation

Run	NaOH conc. (%)	OLR (kg VS m ³ d ⁻¹)	Comp. org. waste ¹ (%)	Methane production (m ³ /kg VS)	
				Experimental	Prediction
1	1.5	0.5	60	0.178 ± 0.012	0.204
2	2.0	1.0	60	0.411 ± 0.015	0.390
3	1.5	1.5	60	0.232 ± 0.005	0.209
4	1.5	0.5	20	0.562 ± 0.009	0.584
5	2.0	1.5	40	0.522 ± 0.011	0.565
6	1.5	1.0	40	0.376 ± 0.014	0.370
7	1.0	1.0	20	0.543 ± 0.004	0.564
8	1.5	1.0	40	0.385 ± 0.007	0.370
9	1.0	0.5	40	0.421 ± 0.017	0.377
10	1.5	1.5	20	0.592 ± 0.011	0.566
11	2.0	0.5	40	0.565 ± 0.014	0.560
12	1.5	1.0	40	0.348 ± 0.010	0.370
13	1.0	1.0	60	0.101 ± 0.003	0.119
14	2.0	1.0	20	0.699 ± 0.013	0.681
15	1.0	1.5	40	0.354 ± 0.014	0.359

¹ Composition of vegetable residual in organic waste

(Hanna instruments) used to measure pH of the anaerobic sludge with an accuracy of ± 0.1 pH.

Experimental Design of Response Surface Methodology

One of the most difficult aspects of a biological experiment is the time required to collect data. For this experiment data were collected every day for at least a month in order to evaluate the activity of the micro-organism responsible for methane production. In order to save time and cost of conducting the experiment, a Box-Behnken Design (BBD) method was employed to represent the optimize methane production model factor were chosen including the concentration of alkaline solution during the pretreatment stage, organic loading rate (OLR) and composition of organic waste. 17 experimental runs were designed in Minitab software consisting of 3 axial points and 12 factorial points were utilized to obtain the optimum methane production. The variables for optimization included X_1 (concentration of alkaline solution for pretreatment step), X_2 (organic loading rate) and X_3 (composition of organic waste). For box-behnken design, the three levels of each variable were represented by -1 (lowest), 0 (intermediate) and +1 (high) code. A numerical

method was employed to determine the second-order polynomial as shown in the equation:

$$\text{Methane production} = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{i=1}^3 \sum_{j=1+1}^3 \beta_{ij} X_i X_j \quad (2)$$

Results and Discussion

Biogas production from vegetable residual and pig mature was relatively stable after the second week of anaerobic digestion. During the first week, the methane content is relatively low due to competitive production of H_2 which correlative with low pH (pH = 5.5) atmosphere inside the digester. The methane content measured by using the portable biogas analyzer after the second week is very stable at 55.5±3.1%.

Validation of Regression Model

Statistical analysis through Box-Behnken design is shown in Table 2. This table illustrated the significance of NaOH concentration (X_1) and composition of organic waste (X_3), which produced high F-value and P-value of lower than 0.05. For this experiment, it should also be pointed out that the OLR does not have a

Table 2. ANOVA for response surface model of the biogas production

Source	df	Sum of squares	Mean square	F-value	P-value
Regression	9	0.347330	0.007408	24.6	0.001*
NaOH conc., X_1 (%)	1	0.075661	0.014979	8.88	0.031*
OLR, X_2 (kg VS m ⁻³ d ⁻¹)	1	0.271585	0.011708	1.30	0.306
Comp. Org. Waste, X_3 (%)	1	0.271585	0.011708	6.94	0.046*
X_1X_2	1	0.000144	0.000144	0.09	0.780
X_1X_3	1	0.005929	0.005929	3.51	0.120
X_2X_3	1	0.000144	0.000144	0.09	0.782
X_1^2	1	0.018368	0.018964	11.2	0.082
X_2^2	1	0.002209	0.002156	1.28	0.310
X_3^2	1	0.000030	0.000030	0.02	0.900
Residual error	5	0.008438	0.001688	-	-
Lack-of-fit	3	0.007694	0.002565	6.89	0.129
Pure error	2	0.000745	0.000745	-	-
$R^2 = 97.7\%$					

* Significant (P-value, 0.05)

significant effect on methane production. However, an increase in OLR beyond the interval observed in this experiment may have a more dramatic effect on the outcome of the anaerobic digestion. According to Table 2, the interactive and squared parameters were not insignificant. The regression model was found to be significant, which indicate that the second-order polynomial fit well with the experimental data. This is confirmed by the R^2 of 97.7% which is very close to 100. The coefficient of each parameter, interaction and squared are also found and plugged into the second-order polynomial equation.

$$\text{Methane production} = 1.474 - 0.8434X_1 - 0.260X_2 - 0.015X_3 + 0.02400X_1X_2 + 0.00385X_1X_3 + 0.0006X_2X_3 + 0.287X_1^2 + 0.097X_2^2 - 0.00001X_3^2 \quad (3)$$

Effect of Different Operating Parameters on Methane Production

According to Figure 3(a) and d, an increase in NaOH concentration has a positive effect on the ability of the organic waste to release methane. This correlated well with other researchers conducted on mesophilic anaerobic co-digestion (Kondusamy and Kalamdhad, 2014). However, at NaOH concentration higher than 1.85% an increase in organic loading rate was found to slightly improve the methane production. On the other hand, if the NaOH concentration used to treat biomass is lower

than 1.65% then an increase in OLR up to 1 kg VS m⁻³ d⁻¹ initially reduced methane production. An increase of OLR further would eventually increase methane production. However, the influence of OLR is not significant in this case.

The influence of NaOH concentration and composition of organic waste on methane production is shown in Figures 3(b) and 3(f). It is obvious that an increase in vegetable content has a negative impact on methane production. This is anticipated because vegetable residual contained lignin which hinders bacterial contact with the cellulose. Additionally, pig manure demonstrated relatively higher biological oxygen demand (BOD) compared with vegetable residual. The optimum condition for methane production can be accomplished by pretreatment at a high percentage of NaOH concentration and using organic loading with a low composition of vegetable residual. Similarly, Figures 3(c) and 3(e) suggested that organic loading rate has an insignificant effect on methane production. It is found by plugging the optimized conditions' value (composition of vegetable residual of 20%, NaOH concentration of 2 (w/w)% and organic loading rate of 1.2 kg VS m⁻³ d⁻¹) in the second order polynomial equation that optimum methane production was 0.691 m³ kgVS⁻¹. This value of methane production is relatively high compared with anaerobic digestion conducted in other researches as shown in Table 3.

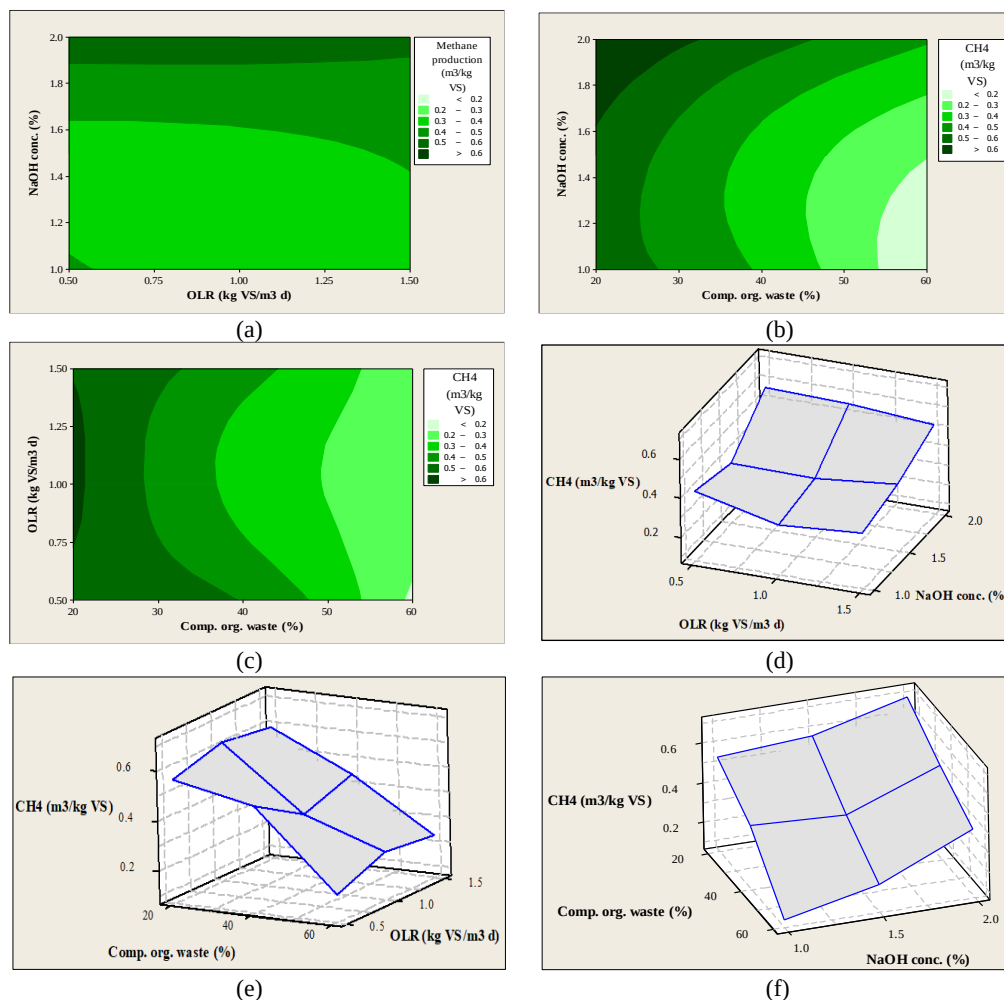


Figure 3. Contour plots (a, b, and c) and response surface plots (d, e, and f) showing the impact of parameters including X1 (NaOH concentration solution for pretreatment step), X2 (organic loading rate), and X3 (composition of organic waste) on methane production (CH₄)

Conclusions

The designed anaerobic digester successfully demonstrated the production of methane from pig manure and vegetable residual. Additionally, the pretreatment and operating conditions were optimized by utilizing the Box- Behnken design. The experimentally measured methane production responses of the anaerobic digestion were close to the data

predicted by the second order polynomial equation. This experiment illustrates the enhancement in biogas production through alkaline pretreatment step. It was also found that the composition of vegetable residual has a dramatic effect on methane production. However, the range of organic loading rate does not have a significant effect on the methane produced. Compared with other researched on anaerobic digestion of vegetable and food waste, the amount of methane

Table 3. Comparison of semi-continuous anaerobic digestion of organic waste

No.	Pretreatment method	Composition of organic waste	OLR (kg VS m ⁻³ d ⁻¹)	Operating day	Methane production (m ³ kgVS ⁻¹)	References
1	-	Food waste (53% vegetable)	3.7	40	0.417	(Nagao <i>et al.</i> , 2012)
2	-	Vegetable (32%) + manure (68%)	5	20	0.205	(El-Mashad and Zhang, 2010)
3	-	Food waste (50%) + Green waste (50%)	6.5	25	0.716*	(Liu <i>et al.</i> , 2009)
	Hydrothermal alkaline (70°C and 10 pH)	Household waste	3	10	0.500	(Chamchoi <i>et al.</i> , 2011)
4	Alkaline Ca(OH) ₂	Municipal solid waste	5.1	19	0.150	(López Torres and Espinosa Lloréns, 2008)
5	NaOH (2%)	Vegetable (20%) + manure (80%)	1.2	21	0.691	This work
6	NaOH (2%)	Vegetable (40%) + Manure (60%)	1.2	21	0.431	This work
7	NaOH (2%)	Vegetable (60%) + Manure (40%)	1.2	21	0.288	This work

* Batch operation under thermophilic condition (50°C)

production from this work is relatively higher. It should be noted that a more effective method of pretreatment can be developed so that the composition of organic waste fed can be completely composed of vegetable residual.

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