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*Original Article*

# Optimization of mixing speed and retention time affecting biogas production from starchy sediment using response surface methodology

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## Abstract

The purpose of this research is to investigate the effects of mixing speed (MS) (5-175 rpm) and retention time (RT) (1-35 days) on biogas production of starchy sediment (SS) that is substantial waste from the modified tapioca starch plants. The AD was performed using biochemical methane potential (BMP) method. The two factor central composite design (CCD) has been employed to design and response surface methodology (RSM) used to evaluate the digestion efficiency and performance. The biogas yield (BY) can be as high as 403 ml/g $TVS_{added}$ at an MS:RT ratio of 112:30, whereas the TVS removal measures equal to 65.07%. It indicated that the SS could be applied as the suitable substrate for AD by operating with this MS:RT ratio. However, the signs of initial system instability were detected. It requires the further study about pH control by adding the chemical to maintain the proper stability.

**Keywords:** biogas, mixing speed, retention time, response surface methodology, biochemical methane potential

## 1. Introduction

Thai government has a policy to stimulate the production of renewable and alternative energy source in order to reduce foreign energy import that accounted for more than 66% of the primary commercial energy demand in 2018 (Department of Alternative Energy Development and Efficiency, 2018). Especially, biogas is produced by anaerobic digestion (AD) of industrial waste such as sludge and wastewater. It was found that the tapioca industry has the second greatest potential for biogas production (Phonphunthin, Uttamaprakrom, & Reubroycharoen, 2014). The modified tapioca starch sector is one of the most vital industries in Thailand, as it is widely used in many manufacturing processes, such as the food, paper, textile, and medical industry. From the high market demand both domestically and internationally were the main driving forces for progressive development in the modified tapioca starch. Thai's modified tapioca starch production has stood at approximately 253,000 tons annually (Asavasanti, Nopharatana, & Ai-Tang, 2006). According to the increasing production, the large amount of starchy waste losses from production process to wastewater. Commonly, starchy waste was separated from wastewater by primary sedimentation in the form of starchy sediment (SS) reaches over 2,530 tons per year (1% of total starch produced) (Thai Tapioca Starch Association, 2018). SS has high contents of biodegradable organic matter consisting of approximately 83% amylopectin and 17% amylose (Pinto, de Araujo, & Peres, 1992).

From its composition and generating volume, it is interesting to study and optimize the biogas production from AD. The use of SS waste in AD is environmental friendly, as it is operated in a closed system to prevent insect nuisance, leachate problems, and unpleasant odors (The Pennsylvania State University, 2012). Moreover, biogas produced can be used to generate heat for starch drying and electricity for wastewater treatment system. Importantly, the use should be concerned about the various factors influencing the performances of the biogas production from the AD. Previous studies have reported that the significant factors that affect biogas production from starch were mixing speed (Sinaga, Hatta, Ahmad, & Mel, 2018; Sindall, Bridgeman, & Carliell-Marque, 2013) and retention time (Ezekoye, & Offor, 2011).

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Since the SS is easy to degrade (McArthur, 2006). Also, the fast mixing speed (MS) can accelerate break down the starch (Farahnaky, Alishahi, Majzoobi, & Al-Hakkak, 2014). It leads to the accumulation of volatile fatty acid (VFA) will cause a sharp drop in pH and consequently resulted in the system instability (Budiyono, Primaloka, Ardannari, Matin, & Sumardiono, 2018). This resulted in a biogas production decrease and finally in a complete stop. Since then, response surface methodology (RSM) as an optimization tool has been used for modeling and analyzing the interaction effects between both factors (Moradi *et al.*, 2016).

The goal of this research is to study the effect of MS and retention time (RT) on the biogas production and digestion performance. Biochemical methane potential (BMP) tests were performed to measure the biogas production from this digestion. The RSM based on central composite design (CCD) is proposed to compute the optimum value. The optimum result can provide not only a practical guide to design but also ensure environmental benefits by recycling SS waste into alternative renewable energy.

## 2. Materials and Methods

### 2.1 Feedstock and inoculum

SS was obtained from a primary sedimentation wastewater pond of a modified tapioca starch plant in Rayong province, Thailand. SS was used as a feedstock in this AD (Figure 1a). It was kept in plastic bags and stored in a refrigerator at 4 °C until further use. The inoculum (Figure 1b) used for this BMP study was obtained from an up-flow anaerobic sludge blanket (UASB) of beverage manufacturing plant in Pathumthani province. Both, SS and inoculum were prepared by mixing with distilled water until the concentration was 2% TVS and 2.5% TVS by wet weight basis, respectively, before the experiment.

### 2.2 BMP test

The BMP test was conducted to assess the biogas yield (BY) and digestion performance of this AD adopted from Owen, Stuckey, Healy, Young, and McCarty (1979). The test was carried out in 125 ml serum bottles with a working volume of about of 80 ml consisting of 32 ml of inoculum and 48 ml of feedstock. The ratio of the inoculum to feedstock is equivalent to the ratio 40:60 by volume. After filling both inoculum and feedstock, initial range of pH value in the bottle was adjusted between 6.8 and 7.2 (Fleck, Tavares, Eyng, Andrade, & Frare, 2017) by adding sodium bicarbonate ($NaHCO_3$) and phosphoric acid ($H_3PO_4$). The headspace of bottle was then flushed with nitrogen gas and was sealed air-tight with rubber stoppers. Finally, the BMP bottles were kept on a shaker (Stuart orbital shaker SSL1, UK) and maintained at a room temperature of 25-30 °C.

### 2.3 Experimental setup

The experimental design was set up to test the effects of MS and RT on BY and digestion performance. The MS and RT were varied at different value by design expert software version 11 (Trial version) based on CCD with an alpha value of 1.414 (The National Institute of Standard and

(a) Starchy sediment (b) Inoculum

Figure 1. Starchy sediment and inoculum

Technology, 2013). The MS was set in the range of 5-175 rpm (Sittijunda & Pattra, 2016). The RT was in the range of 1-35 days (Jegede, Zeeman, & Bruning, 2019). The incubator shaker was stopped for 15 minutes every hour. The codes and scales of the variables used in this experimental design were shown in Tables 1 with a total of 14 runs as listed in Table 3. For each run performed in triplicate.

### 2.4 Analysis and interpretation

The characteristics of SS, inoculum and feedstock were analyzed for pH, total solids (TS), total volatile solids (TVS), total organic carbon (TOC), total nitrogen (TN), and carbon to nitrogen (C/N) ratio following APHA standard (American Public Health Association, 2017). In the BMP test, the volume of biogas was measured daily by using displacement of a syringe. At the end, the effluents from BMP bottles were analyzed for pH, volatile fatty acids (VFA), alkalinity (ALK) and TVS for evaluating the digestion performance. The BY was calculated using the following Equation (1).

$$BY\ (\text{ml/g TVS}_{\text{added}}) = \frac{B}{TVS_{\text{added}}} \quad (1)$$

where B is an accumulated biogas volume (ml) and $TVS_{added}$ is a weight of TVS of SS added to the bottle (g).

The mean and standard deviation were used to describe the BY, pH, VFA/ALK ratio and TVS removal. Analysis of variance (ANOVA) was used to evaluate the appropriateness of the predicted BY equation including a probability value (p-value), R squared ($R^2$) value, adjusted R squared (Adj. $R^2$) and the percentage of coefficient of variation (%CV). Moreover, the RSM plot and desirability value were applied to select the optimum MS and RT.

## 3. Results and Discussion

### 3.1 SS and inoculum characteristics

The characteristics of the SS and inoculum are shown in Table 2. SS is mildly acidic with a pH value of 5.31±0.06. Moisture content is only 43±3.21% for SS, while it is 95.32±1.13% for Inoculum. SS consisting of higher organic, TVS was 33.32±1.23% that was suitable for AD. As the SS has very significant C/N ratio of 160 using as the suitable

Table 1. Codes and scales of variables

| Variables | Unit | Coded level | | | | |
|---|---|---|---|---|---|---|
| | | -1.414 | -1 | 0 | +1 | +1.414 |
| MS | rpm | 5 | 30 | 90 | 150 | 175 |
| RT | days | 1 | 6 | 18 | 30 | 35 |

Table 2. SS and inoculum characteristics

| Parameters | Substrates | | Analytical method |
|---|---|---|---|
| | SS | Inoculum | |
| pH | 5.31±0.06 | - | pH meter |
| Moisture (%) | 43±3.21 | 95.32±1.13 | Dried at 103–105 ºC |
| TVS (%) | 33.32±1.23 | 5.34±0.021 | Dried at 500–550 ºC |
| C/N ratio | 160 | 8.3 | Walkley-Black and Kjeldahl method |

carbon source. When SS was mixed in the BMP bottle with inoculum, the C/N ratio of feedstock decreased to a lower C/N ratio at 99.3. This may be adequate to produce biogas through AD process since it is quite near the optimal C/N ratio. As regards the C/N ratio, the optimal value equals to 120 for the AD of wastewater (Gil, Siles, Serano, Chica, & Martin, 2019).

## 3.2 CCD design matrices for the variables and the responses

Table 3 presents the characteristics of a total of 14 CCD experimental runs. In each run, BY and TVS removal obtained from BMP tests as the responses for this digestion condition. BY ranged between 18 and 410 ml/g $TVS_{added}$. Note that there was the greater variability in BY among runs depending on the both variables. TVS removals also varied in the range of 19.7 to 66.67%. Comparatively, the study of Fleck *et al.* (2017) found that the AD of tapioca wastewater had TVS removal at 63.12% operating under mesophilic and batch system.

It is noteworthy that the run no. 9 had the highest TVS removal but a low BY. This is because the AD may induce to reduce the TVS contents by converting organic compounds into soluble forms of VFA, which are not the desired end product as methane. It was obviously well supported by its high relative VFA/ALK ratio (more than 0.4) resulted in the dramatic drop in pH (equal to 4.3). Moreover, these were signs of instability in digestion system can also cause a reduction in BY (Drosg, 2013). Thus, several factors (BY, TVS removal, VFA/ALK ratio, and pH) should be considered in selecting the optimum condition for digestion, in order to ensure the complete digestion occurs and intermediate chemicals tend not to over accumulate.

## 3.3 Regression and statistical analyses

Based on the ANOVA for the predicted model, results used BY as a response are given in Table 4. For BY, the suitable model is cubic. The model had a significant fit showing the larger Fisher's value (F-value) equals to 318.27 with smaller p-value less than 0.0001. The results indicate that the BY depends significantly on all terms in the model with p-value less than 0.05 at the 95% confidence interval. Besides, the significant interactions were found between MS and RT affecting the BY.

Moreover, the %CV measures the ratio of the standard deviation to the mean, also known as the relative variability. The smaller %CV is desirable (Canchola, 2017). So, its %CV was equal to 5.40, which indicated low variance having a more consistent. High $R^2$ (0.997) and Adj. $R^2$ (0.9902) near 1 (Rawski, Sanecki, Kijowska, Skitat, & Saletnik, 2016) advocated higher relationship between actual and predicted BY value. From the summarized statistical values, the cubic model has the more suitable for this experimental condition which was found to be well fits with the all criteria (Table 6). The selected cubic model in actual variables was shown in the Equation (2). Clearly, the RT (B) has the highest positive effects on the BY, whereas the MS (B) has a negative effect. The variable squared ($A^2$ and $B^2$) and other interactions (A.B, $A^2B$ and $AB^2$) between variable have the small effects on BY.

Table 3. CCD codes, variables and response results in the experimental runs.

| Run | CCD codes | | Variables | | Response | |
|---|---|---|---|---|---|---|
| | MS | RT | MS (rpm) | RT (day) | BY (ml/g $TVS_{added}$) | TVS removal (%) |
| 1 | 0 | 0 | 90 | 18 | 369±0.06 | 51.51±0.03 |
| 2 | 0 | 0 | 90 | 18 | 361±0.12 | 56.06±0.03 |
| 3 | -1 | -1 | 30 | 6 | 18±0.03 | 21.21±0.03 |
| 4 | -1 | 1 | 30 | 30 | 164±0.16 | 33.33±0.03 |
| 5 | -1.414 | 0 | 5 | 18 | 85±0.19 | 25.76±0.03 |
| 6 | -1.414 | 0 | 5 | 18 | 71±0.06 | 19.7±0.03 |
| 7 | -1.414 | 0 | 5 | 18 | 62±0.06 | 25.76±0.05 |
| 8 | 1.414 | 0 | 175 | 18 | 201±0.17 | 53.03±0.03 |
| 9 | 1.414 | 0 | 175 | 18 | 178±0.06 | 54.55±0.05 |
| 10 | 1.414 | 0 | 175 | 18 | 184±0.11 | 57.57±0.03 |
| 11 | 0 | -1.414 | 90 | 1 | 49±0.06 | 19.7±0.03 |
| 12 | 0 | 1.414 | 90 | 35 | 410±0.33 | 66.67±0.03 |
| 13 | 1 | -1 | 150 | 6 | 250±0.08 | 50±0.05 |
| 14 | 1 | 1 | 150 | 30 | 269±0.10 | 75.76±0.03 |

Table 4. ANOVA analyses of the predicted BY model

| Source | Term df | Error df | F-value | p-value | |
|---|---|---|---|---|---|
| Whole-plot | 4 | 6.00 | 318.27 | < 0.0001 | significant |
| A-Mixing speed | 1 | 6.00 | 187.22 | < 0.0001 | |
| $A^2$ | 1 | 6.00 | 806.97 | < 0.0001 | |
| $B^2$ | 1 | 6.00 | 212.22 | < 0.0001 | |
| $AB^2$ | 1 | 6.00 | 53.80 | 0.0003 | |
| Subplot | 3 | 6.00 | 239.08 | < 0.0001 | significant |
| B-Retention time | 1 | 6.00 | 614.96 | < 0.0001 | |
| AB | 1 | 6.00 | 38.06 | 0.0008 | |
| $A^2B$ | 1 | 6.00 | 140.85 | < 0.0001 | |

$$BY = 235.2 - \ + (32B) + (0.13A.B) + (0.003A^2) - (0.9B^2) - (0.002A^2B) + (0.005AB^2) \quad (2)$$

where, BY is biogas yield (ml/g $TVS_{added}$), A is MS (rpm), and B is RT (day).

Table 5 presents the ANOVA results using TVS removals as response. The two factor interaction (2FI) model was selected. Its ANOVA result was significant by showing a F-value of 21.15 and p-value of 0.0115. Both variables, MS and RT had relative effects on the TVS removals. An interaction between both variables had no correlation to TVS removals. The F-value of 1.90 and p-value of 0.2164 which were more than 0.05 and thus it was insignificant. For the $R^2$ and the Adj. $R^2$ was 0.9305 and 0.88771 (Table 6), which closes to value 1 indicating the high predicted accuracy.

The fitted 2FI for TVS removals as response in actual variable was shown in Equation (3). It shows that both variables and the interaction between MS and RT had positive effects on TVS. In this Equation, the RT had more influence over the others.

$$\text{TVS removal} = 12.6 + (0.13A) + (0.66B) + (0.005A.B) \quad (3)$$

where TVS removal is considered as the percentage of TVS removed from an initial TVS (%), A is MS (rpm), and B is RT (day).

Table 5. ANOVA analyses of the predicted TVS removal equation

| Source | Term df | Error df | F-value | p-value | |
|---|---|---|---|---|---|
| Whole-plot | 1 | 3.78 | 21.15 | 0.0115 | significant |
| A-Mixing speed | 1 | 3.78 | 21.15 | 0.0115 | |
| Subplot | 2 | 6.06 | 28.78 | 0.0008 | significant |
| B-Retention time | 1 | 6.06 | 55.66 | 0.0003 | |
| AB | 1 | 6.06 | 1.90 | 0.2164 | |

Table 6. Statistical analyses of the predicted equation

| Response | Std. Dev | Mean | %CV | $R^2$ | Adj. $R^2$ |
|---|---|---|---|---|---|
| BY | 10.29 | 190.79 | 5.40 | 0.997 | 0.9902 |
| TVS removal | 8.02 | 43.62 | 18.38 | 0.9305 | 0.8871 |

## 3.4 RSM plots

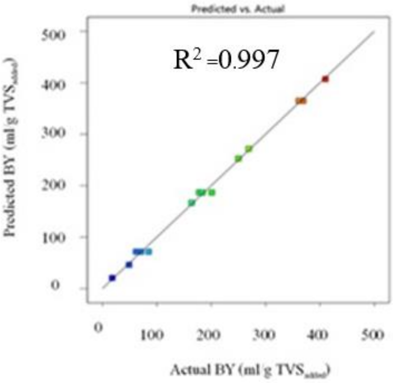
Figure 2a shows the plot between predicted and actual value of BY. Obviously, the most correlation between these two values stayed in the straight line. This plot indicated that the data followed a good correlation and also referred to satisfy. The effects of MS and RT interactions on the BY presented by three-dimensional RSM plot in Figure 2b. At the shorter RT (6 days), the BY increased constantly from 18 to 250 as the MS increased. While at the longer RT (30 days), the BY increased from 164 to 410 when the MS increased until to reach approximately 120 rpm. After that, the shift in MS has resulted in a gradual reduction of BY. Due to the effect of high MS showed a rapid increase in the accumulation of VFA. This resulted in a pH drop as indicating instability. As shown in Figure 3, there was a strong decrease in pH values from 6.4 to 4.6 with increasing MS from 90 to 150 rpm. Typically, the digestive instability have a negative effect on biogas production (Meisam & Hossein, 2018).

For TVS removals as depicted in Figure 2c all data points are closer to the regression line. Accordingly, the higher $R^2$ obtained at 0.9305 representing the smaller differences between the predicted and actual TVS removals. In this case, the predicted TVS removals range between 21.21 and 75.76% as shown in Figure 2d. Its RMS plot illustrated that the raise of both MS and RT had the same effect tends to increase the BY.

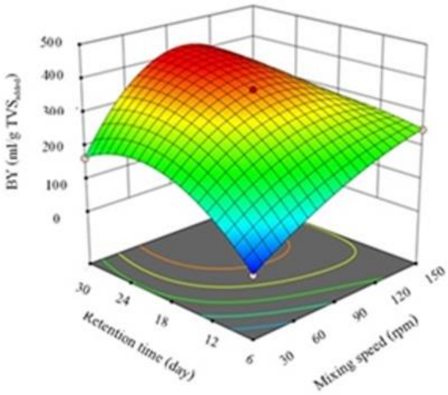
## 3.5 Digestion performance

Figure 3 presents the evaluation of digestion stability by both effluent VFA/ALK ratio and pH. Markedly, all the runs had the ratio of VFA/ALK that were too high than the recommended ratio for AD (It should be less than 0.4 (Filer, Ding, & Chang, 2019). Except the runs, were conducted with both lower MS and shorter RT at the MS:RT ratio of 5:18 and 90:1 by VFA/ALK ratio equal to 0.3. Moreover, there was a significant increase of the VFA/ALK ratio, when the MS was over than 150 rpm. For instance, at the MS: RT ratio of 150:6, 150:30, and 175:18 were 0.7, 0.8, and 1.3, respectively. Thus, the MS should be used less than 150 rpm.

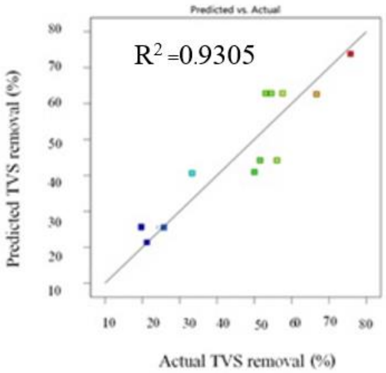
When considering the effluent pH, we found that at constant MS at 90 rpm, the effluent pH had relatively decreased, when the RT increased from 1 to 35 days. Also, the result showed very clearly that the expanded RT affected a major reduction in the effluent pH. Including, while fixing MS at 150 rpm, it was found that the effluent pH values were very



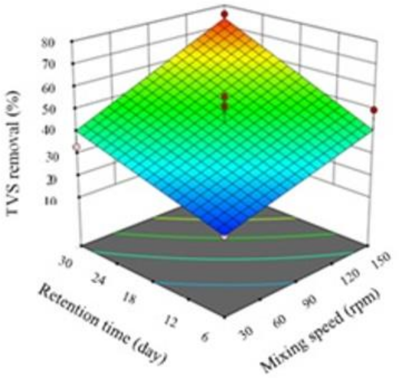

(a) Relationship between predicted




(b) RSM plot of BY. and actual value of BY.




(c) Relationship between predicted




(d) RSM plot of TVS removal and actual value of TVS removal

Figure 2. Scattered and RSM plots

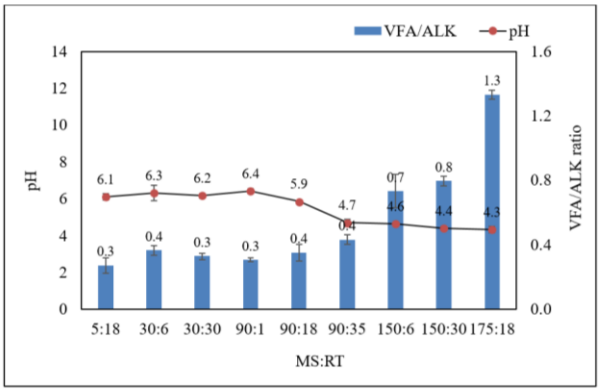



Figure 3. Effluent VFA/ALK ratios and pH in the BMP tests at different MS and RT

lower than many recommended (5.5-7.5) (Mahmood & Ali Shah, 2014) in every RT (6, 18 and 30 days). These are reasonable support as the MS must be less than 150 rpm. A higher MS induced a faster digestion of SS. Over accumulations of VFA occurred that cannot be converted to methane immediately. Then, this leads to the drop of pH in the AD system. At the MS: RT ratio of 90:18 is possible and suitable for AD due to the optimum VFA:ALK ratio of 0.4 and pH obtained of 5.9.

However, following the results of desirability analysis (Table 7) indicated that at the MS: RT of 118:30 achieved the maximum BY of 403 ml/g $TVS_{added}$ and the TVS removal of 65.07% with the proper desirability value of 0.89. The value was close to 1 which indicates better (Amdoun *et al.*, 2018). However, this ratio was not being included in the total 14 runs. It was conducted to test the stability. The observed effluent VFA/ALK ratio and pH were 0.57 and 4.9±0.3 respectively, that is inappropriate and the sign of system failures. It is a substantial limitation for the application which needs to be adjusted the pH value during the digestion. In case the AD system without the ability to optimize the pH, at the MS:RT ratio 0of 90:18 is the appropriate choice. Although the obtained BY was only 365 ml/g $TVS_{added}$ that was down by 9.43% as compared to the maximum BY (403 ml/g $TVS_{added}$), it provided the good system stability. These results were compared with other AD studied biogas production from different biomass wastes (Roati *et al.*, 2012).

Table 7. Desirability analysis

| The ratio of MS:RT | BY (ml/g $TVS_{added}$) | TVS removal (%) | Desirability value |
|---|---|---|---|
| 118:30 | 403 | 65.07 | 0.89 |
| 90:18 | 365 | 44.18 | 0.79 |

It was found that the BY quite close to the BY of manure (200-500 ml/g $TVS_{added}$), crop (350-400 ml/g $TVS_{added}$) and sewage sludge (250-350 ml/g $TVS_{added}$). It indicates that SS is a potential substrate and source of renewable energy to be performed under these conditions.

## 4. Conclusions

This study demonstrated that the effect of MS and RT on BY from SS in the BMP experiments were investigated using the RSM for analysis. The results showed that the MS and RT recommended of 112 rpm and 30 days are optimum for maximum BY achieved approximately 403 ml/g $TVS_{added}$ with the TVS removal 65.07%. However, the instability is very aware of this AD. Thus, a pilot study in the model should be further performed and developed to optimize the whole pH during digestion. Furthermore, to improve this AD system stability, a co-digestion of SS with other organic materials is possible and practical to compromise this solution.

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