Determining optimization of factors affecting biogas production by co-digestion of sweet corncob waste with wastewater using response surface methodology

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Abstract

The objective of this study was to investigate the optimum conditions for biogas production by co-digestion of wastewater with sweet corncob waste (SW) in a 1 L batch reactor using a response surface methodology (RSM) experimental design. With three experimental factors affecting the production of biogas including the ratio of wastewater from sweet corncob milling process (WM) and wastewater from production process (WP) (WM:WP; 0:100, 20:80, 50:50, 80:20 and 100:0), total solid of substrate (TS, 8-16%) and initial pH (6 - 8) were studied. A quadratic model was used for the prediction of biogas production by central composite design (CCD). Probability ($P<0.05$) showed a very significance for the regression model with the $R^2 = 0.987$. The model could be efficiently used for predicting biogas production from co-digestion. The analysis of variance indicated that all of the factors had significant individual effect on biogas production. The optimum conditions were found to be as follows: TS of substrate, 10%; initial pH, 7.0; WM:MP ratio, 50:50. The maximum biogas production was approximately 6,125 mL (7.56 $mLCH_4/gCOD_{removed}$.day of methane yield). The RSM technique was useful to predict the biogas production process in this study successfully.

Keywords:

Co-digestio, biogas production, anaerobic digestion, response surface methodology

1. Introduction

Energy demand is increasing along with the growing world population [1]. The world's energy consumption mainly comes from a fossil fuel formed as a liquid, solid or gas. Fossil fuels are non-renewable of energy resources with a limited supply for future. Therefore, the researchers have sought to find a variety of alternative energy sources for the near future. Alternative energy continues to draw attention in the world that is an alternative to conventional fossil fuels and is generally generated s from a natural resource which is completely sustainable, safe, cost-effective and clean. In addition, alternative energy can improve the environmental quality, and also provide economic opportunities in the rural areas of developing countries. Millions of tons of agricultural wastes are produced each year in the world [2]. Thailand is considered suitable for cultivation of sweet corn in a tropical climate. The amount of corn waste being produced is growing faster such as the husks, shanks, silks, cobs, and wastewater from production process from the corn canning factory. Sweet corn waste is the most abundant biomass agricultural waste with large quantities of lignocelluloses wastes which can be converted to high-value by-products. This study was focused on sweet corn waste and wastewater as the raw materials for the production of biogas. Biogas is a biological process from anaerobic digestion or fermentation of organic waste including manure, sewage sludge, wastewater, municipal solid waste, biodegradable waste or any other biodegradable feedstock [2,3,4,5] in a complex process. The stages of decomposition (Fig. 1) (hydrolysis, acedogenesis, acetogenesis, and methanogenesis) involved microorganisms to transform waste into biogas to produce methane (CH_4) and carbon dioxide (CO_2) as end products [6,7]. Raw biogas contains 50-65% methane (CH_4), 30-45% carbon dioxide (CO_2), with smaller amounts of water vapor and trace amounts of 0.1-1% hydrogen sulfide (H_2S) and other impurities [8]. The first stage in the process (hydrolysis) involves converting the complex organic compounds into smaller units by hydrolytic microbe for use as a source in the next step. The second and third stage (acedogenesis and acetogenesis) involve the intermediate compounds for converting to lower chain products such as acetate, hydrogen and carbon dioxide which can directly be utilized by a group of methanogenic bacteria to produce end products (CH_4 and CO_2) [9,10].

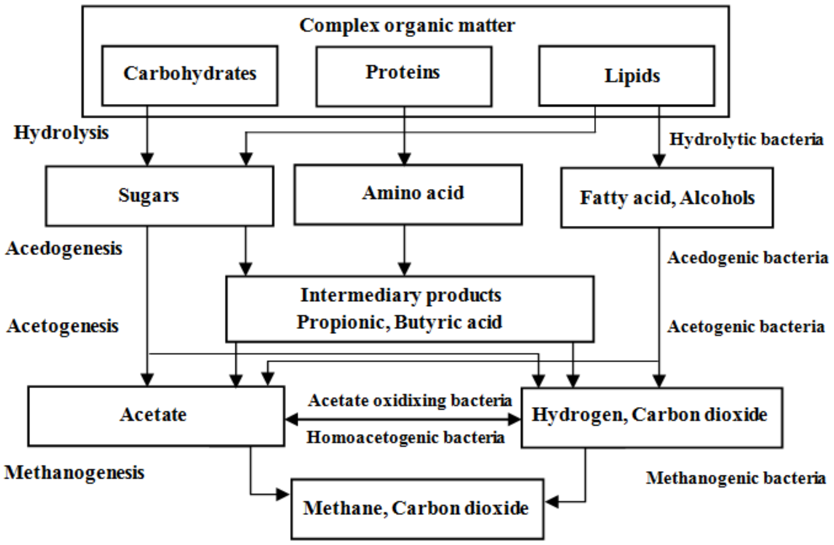


Fig. 1 The stages of decomposition in anaerobic digestion process [4, 7, 10].

Response surface methodology (RSM) is a statistical technique for designing experiments in order to optimize the multivariable system. This method can reveal the alternative effects between the components and is also less time consuming to determine optimum levels, which are reliable. In the conventional approach, optimization is usually carried out by varying one variable a time while all other variables are fixed in the experiments. RSM explores the relationships between a dependent variable or more response variables and several independent variables. The interactions of possible influencing factors on response can be evaluated with a limited number of planned experiments [11]. The main objective of this study was to investigate the effects of the total solid of substrate (TS), initial pH and the ratio of WM:WP on biogas production by DOE using of RSM in anaerobic co-digestion of sweet corncob waste (SW), wastewater from the sweet corncob milling process (WM) and wastewater from production process (WP) in a 1 L batch reactor.

2. Materials and methods

2.1 Raw material

In this research, sweet corncob waste (SW), wastewater from production process (WP) and wastewater from sweet corncob milling process (WM) as the feedstock were kindly provided from a company located in the north of Thailand (Sun Sweet Company Limited, Chiang Mai, Thailand) as shown in Fig. 2A-C. Inoculum was obtained from anaerobic reactor located in Thailand. This reactor is being operated for the treatment of corn canning-processing wastewater. The SW was ground to small pieces of an average 1-2 cm length by grinder the raw material.

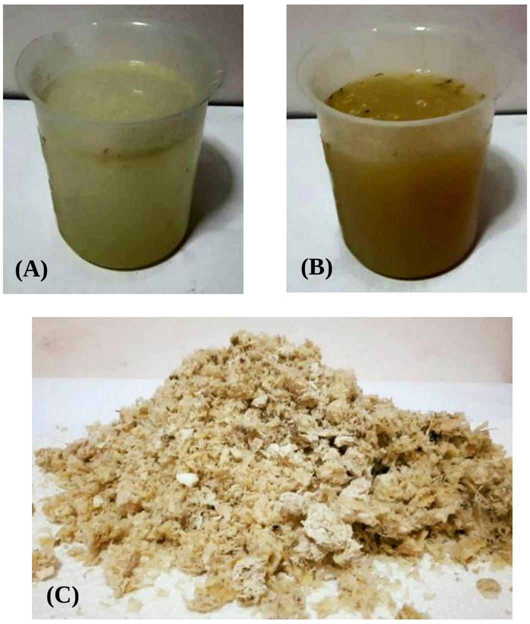


Fig. 2 Illustration of wastewater from production process; WP (A), wastewater from sweet corncob milling process; WM (B) and sweet corncob waste; SW (C) as the feedstock.

2.2 Analytical methods

The collected WM, WP and inoculum were analyzed for the characteristics pH, chemical oxygen demand (COD), total solid (TS), volatile solid (VS), alkalinity (ALK) and volatile fatty acid (VFA) according to the APHA standard methods [12] and analyzed for the ratio of carbon and nitrogen (C/N ratio) and moisture content for SW in accordance with the standard methods [13]. The moisture content and C / N ratio of SW in these experiments were 6.4% and 12, respectively. The pH value was directly measured from liquid samples with a digital pH meter. Measurements of every item for each sample were repeated for three times and the average value was selected for determination. Characteristics of the feedstock are presented in Table 1.

Table 1 Characteristics of the feedstock.

Items	Unit	Value		
		WM	WP	Inoculum
COD	mg/L	24,000	12,000	8,000
TS	mg/L	68,075	11,200	4,920
VS	mg/L	51,730	6,885	2,670
ALK	mg/L $CaCO_3$	1,202	681	1,423
VFA	mg/L	3,950	732	1,423
pH	-	3.46	5.40	7.30

2.3 Operating procedure

The batch digestion of biogas production test was performed in a reactor with a total capacity of 1 L glass bottle and a working volume of 800 mL for containing the SW, WP, WM, and 10% (v/v) of inoculum. The WP and WM were placed in the reactor at different ratios of WM:WP; 0:100, 20:80, 50:50, and 100:0 with SW at different TS (6, 8, 10, 12 and 14 %) according to the designation. The pH values (6, 6.5, 7, 7.5 and 8) were adjusted using 5M NaOH and 5M HCL [14]. The gas production was measured on a daily basis. The batch fermentation in the reactor was carried out for 20 days. The entire study was carried out at ambient temperature (28 ± 3 ºC) and then slowly shaken manually for about 1 min once daily for mixing the organic content. At the beginning of the experiment, the headspaces of the reactor were flushed with nitrogen gas in order to remove the oxygen content to ensure anaerobic condition. A glass bottle was tightly sealed with silicone sealant and aluminum caped after it was filled with inoculum and substrate. The volume of biogas was monitored daily through the water displacement method from the collecting plastic cylinder directly as shown in Fig. 3 and the corresponding cumulative biogas volume calculate. The compositions of the gas were determined with a gas detector to detect methane, carbon dioxide and hydrogen sulfide from the gas-tight aluminum bags. All the experiments were carried out in triplicate and the results were expressed as means.

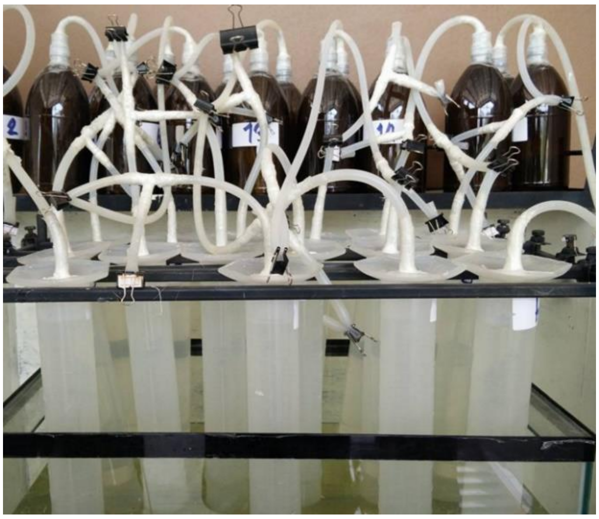


Fig. 3 Illustration of experiment apparatus in anaerobic digestion.

2.4 Experimental design and optimization

In this study, the statistical graphics Design-Expert software was analyzed for the optimum conditions of factors equivalent to the highest biogas produced for regression and graphical analyses of the obtained data optimization. DOE is a tool to develop experimentation strategy using minimum resources by investigation all factors and their interactions [15]. The optimum conditions are used to find by the response surface methodology (RSM) technique when a number of factors are involved in process and the response that fit an empirical polynomial model based on three groups of design, fractional factorial points, axial points and center points. The response can be simply related to the independent factors a quadratic model consisting of linear, two factorial and quadratic terms as shown in Eq. (1). Central composite design (CCD) was used in the optimization of biogas production from co-digestion of SW, WP and WM. The TS of substrate (X_1), initial pH (X_2) and WM:WP ratio (X_3) were chosen as shown in Table 2.

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^{\ 2} + (\sum_{i=1}^{k-1} \sum_{j=i+1}^{k} \beta_{ij} x_i x_j)_{i<j} \quad (1)$$

In this polynomial equation, y is the predicted response evaluated; x_i x_j are independent variables; β_0 is the offset term; β_i, β_{ii}, and β_{ij} are the linear coefficients, the quadratic coefficient and interaction coefficient, respectively; and k is the number of studied factors. Each independent variable in the CCD was carried to determine at five different levels assigned as, -α, -1, 0, +1, and +α, respectively to cover the experiment conditions. Biogas production (mL) was used as the response of experiment.

According to the design, 17 runs of experiments were performed with three replications of the center points. For optimal point prediction, a second order polynomial model function was fitted to the experimental results. The regression model was calculated by analyzing the analysis of variance (ANOVA). The quality of the model was expressed by the value of correlation coefficient, R^2. Model term was evaluated by P-value (probability) with 95% confidence level [16]. The model described the interaction among the factors influencing the output variable by varying them concurrently.

Table 2 Experimental levels of the independent variables used for optimization of biogas production.

Variable	Label	Code variable level				
		-α	-1	0	+1	+α
X_1	TS of substrate (%)	6	8	10	12	14
X_2	Initial pH	6	6.5	7	7.5	8
X_3	WM:WP ratio	0:100	20:80	50:50	80:20	100:0

3. Results and Discussion

3.1 Model fitting and statistical analysis

The design experiment of the variables in the code and actual values is depicted in Table 3 with the experimental values of biogas production as response. The predicted values of response were obtained from quadratic model and evaluating between TS of substrate (X_1), initial pH (X_2) and WM: WP ratio (X_3). The central composite design (CCD) was used for fitting a second-order model from experimental runs. These methods involved in the mathematical model for designing chemical processes and analyzing the process results. Among them, response surface methodology (RSM) was employed for mathematical model formation and optimization performing for biogas production in an anaerobic reactor. The three independent variables were converted to dimensionless ones (X_1, X_2, X_3), with the coded values at levels: -α, -1, 0, +1, +α. A three-factorial, CCD model suggested conducting a total of 17 experiments, considering five levels and three center points. The model predicted factors and associated response based on experiments are shown in Table 3. The biogas production data obtained from the experiments was fitted to develop the model. The significance of each factor was evaluated from the P-value by ANOYA. RSM suggested quadratic model with P-value and F-value of 101.83 and 0.0001, respectively as shown in Table 4. This can be seen in Fig. 4 by comparing the actual values against the predicted responses by the model for biogas production. Accordingly, the ANOVA of the suggested model analyzed P-value to be less than 0.0001 indicated that the model was reliable, the smaller the P-value for the independent factor, the more significance [1, 17, 18, 19,]. The P-value for all the factors was found to be less than 0.0001 indicates that the model was statistically significant and interaction terms affected the response. Statistically estimated R^2 value for the fitted model determines the quality of the model. The obtained value (R^2 = 0.987) indicated that 98.7% of the experimental results confirmed consistency with the data predicted using the design of experiment by code suggested a model by the following quadratic Eq. (2). The response surface model developed in this study for predicting the response was perfectly adequate (R_2 adj. = 0.977). Design Expert program could be removed and improved the model; the stepwise elimination procedure was selected to automatically reduce the insignificant terms and the resulting ANOVA data for the reduced quadratic model of total flux are given in Table 4.

$$Y = 6093 - 167.38X_1 - 279.55X_2 + 388.42X_3 + 275.59X_1X_3 - 2080.37X_1^2 - 893.90X_2^2 + 792X_3^2 \quad (2)$$

Where *Y*: is the response, X_1 (TS of substrate (%)), X_2 (initial pH), and X_3 (WP: WP ratio) are factors in DOE code. The regression analysis of the experiment design showed that the linear model terms and quadratic terms were significant (P<0.05) (Table 4).

Table 3 The central composite design (CCD) of experimental factors in coded and actual values with responses from experimental results.

Standard Run no.	**Run**	**Code values**			**Actual values**			**Response values**
		X_1	X_2	X_3	X_1	X_2	X_3	
10	1	+1	0	0	14	7.0	50	180
9	2	-1	0	0	6	7.0	50	540
15	3	0	0	0	10	7.0	50	6,116
13	4	0	0	-1	10	7.0	0	3,280
3	5	-α	+α	-α	8	7.5	20	2,420
6	6	+α	-α	+α	12	6.5	80	3,620
4	7	+α	+α	-α	12	7.5	20	1,850
7	8	-α	+α	+α	8	7.5	80	2,410
2	9	+α	-α	-α	12	6.5	20	2,470
12	10	0	+1	0	10	8.0	50	3,400
16	11	0	0	0	10	7.0	50	6,120
1	12	-α	-α	-α	8	6.5	20	2,470
17	13	0	0	0	10	7.0	50	6,140
8	14	+α	+α	+α	12	7.5	80	3,000
5	15	-α	-α	+α	8	6.5	80	2,756
11	16	0	-1	0	10	6.0	50	4,020
14	17	0	0	+1	10	7.0	100	4,810

X_1, X_2, and X_3 were the code values of TS of substrate, initial pH and WM:WP ratio, respectively.

Table 4 Analysis of variance for quadratic polynomial model.

Source	Sum of Squares	df	Mean Square	F-Value	p-value Prob > F
Model	4.901E+007	7	7.002E+006	101.83	< 0.0001
X_1	3.465E+005	1	3.465E+005	5.04	0.0484
X_2	9.667E+005	1	9.667E+005	14.06	0.0046
X_3	2.072E+006	1	2.072E+006	30.14	0.0004
X_1X_3	5.311E+005	1	5.311E+005	7.73	0.0214
X_1^2	4.570E+007	1	4.570E+007	664.73	< 0.0001
X_2^2	8.438E+006	1	8.438E+006	122.72	< 0.0001
X_3^2	6.864E+006	1	6.864E+006	99.84	< 0.0001

R^2 = 0.9875
Adjusted R^2 = 0.9778
Predicted R^2 = 0.9325

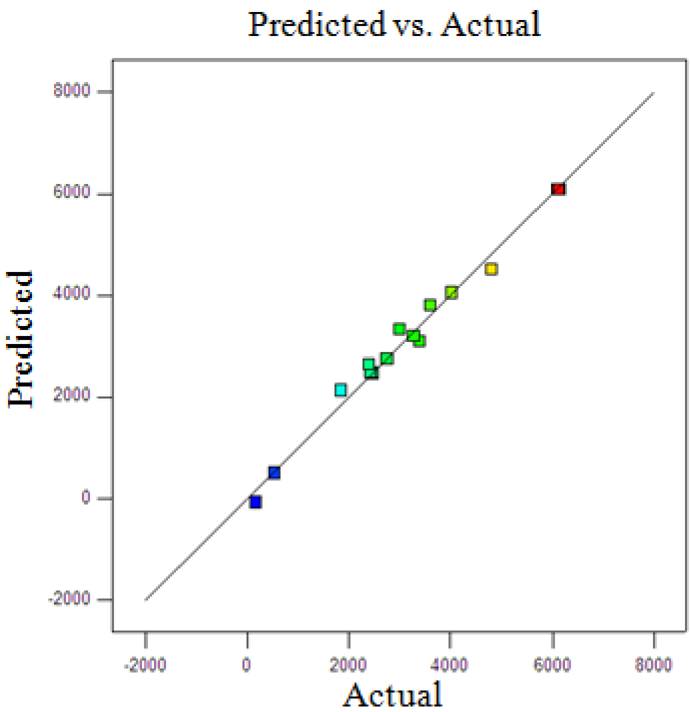


Fig. 4 Plot of predicted response vs. actual value from response surface reduced quadratic model.

3.2 Effect of TS of substrate and initial pH on biogas production

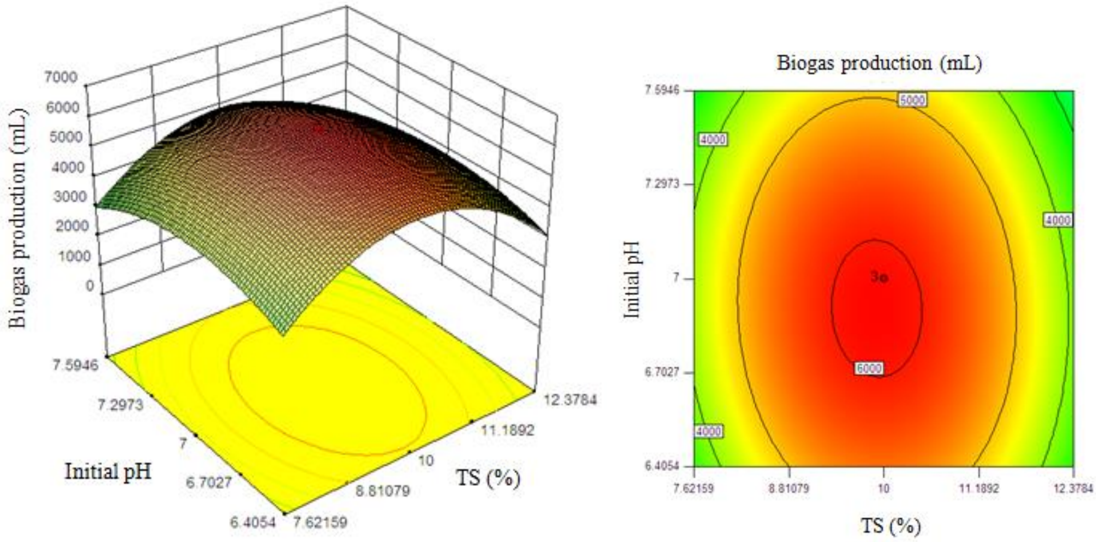
The three dimensional (3D) response surface and two dimensional (2D) contour plots of TS of substrate and initial pH on biogas production are shown in Fig. 5 A-B. The predicted maximum value of TS of substrate and initial pH on biogas production was indicated at the top of surface. The results indicated that the TS of substrate on biogas production were significant (P > 0.05). Increase in TS of substrate resulted in an increase of biogas production when initial pH and WM:WP ratio were kept at a central value. However, the biogas production was produced low level at TS of substrate of 6 and 14% when initial pH and WM:WP ratio were kept at the central value. The results were in accordance with previous reports [20, 21, 22] reported that overloading of substrate resulted in microbial inhibition and reduction of the biogas production process. The highest biogas production, approximately 6,125 mL (7.56 $mLCH_4/gCOD_{removed}$.day of methane yield) was obtained at 10% of TS of substrate when initial pH and the ratio of WM:WP were kept at 7.0 and 50:50.

3.3 Effect of WM:WP ratio and TS of substrate on biogas production

The relationship between WM:WP ratio and TS of substrate on biogas production is shown in Fig. 6 A-B. The results show that the factors of WM:WP ratio on biogas production showed the most significant effect ($P < 0.05$) and TS also significantly affected the biogas production ($P < 0.05$). In addition, the interactive effect of WM:WP ratio and TS of substrate was significant ($P < 0.05$) as shown in Table. 4. The observation in this study indicated that increasing WM:WP ratio resulted in an increase in biogas production but the biogas production decreased when the ratio of WM:WP exceed 50 percent since a higher ratio may lead to an acidic condition in the acedogenesis process which decrease biogas production. The optimum WM:WP ratio for biogas production was 50:50 which obtained the highest biogas production when TS of the substrate and initial pH were found to be 10% and 7.0, respectively.

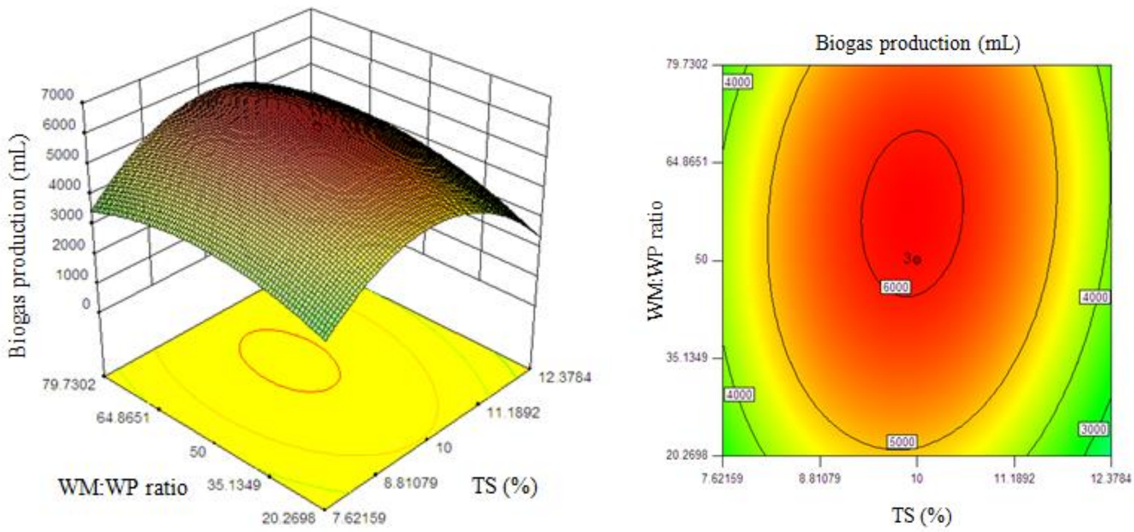
3.4 Effect of initial pH and WM:WP ratio on biogas production

The interactive effects of initial pH and WM:WP ratio on biogas production were insignificant on biogas production ($P > 0.05$). However, the analysis of variance indicated that initial pH and WM:WP ratio was significant on biogas production ($P < 0.05$). The relationship between the initial pH and WM:WP ratio on biogas production is shown in Fig. 7A-B The optimum initial pH for biogas production was 7.0 when TS of the substrate and WM:WP ratio were kept at the central value. The biogas production was at a low level when the initial pH was 8.0. The results agree with [20, 23, 24] which the excessive alkaline pH could inhibit the activity in the methanogenesis process resulting in the decrease in biogas production.



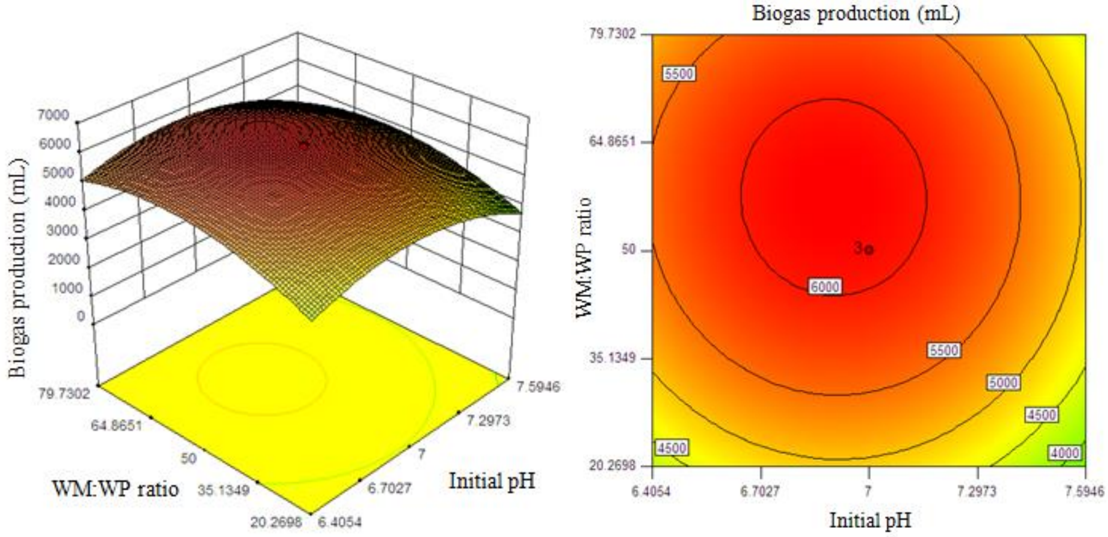
(A) (B)

Fig. 5 The effects of TS of substrate and initial pH on the biogas production: (A) response surface and (B) contour plots.



(A) (B)

Fig. 6 The effects of TS of substrate and WM:WP ratio on the biogas production: (A) response surface and (B) contour plots.



(A) (B)

Fig. 7 The effects of initial pH and WM:WP ratio on the biogas production: (A) response surface and (B) contour plots.

4. Conclusions

This work studied the effects of TS of substrate, initial pH and WM:WP ratio on biogas production in anaerobic digestion for finding the optimum condition using the statistical methodology. The results found significant effect on the model ($p < 0.05$). All of the factors had significant individual effect on biogas production. However, only the interaction of TS of the substrate and WM:WP ratio demonstrated significant effect on biogas production. The optimum conditions were 10% TS of substrate, initial pH 7.0 and WM:MP ratio of 50:50. The maximum response value for biogas production was estimated as 6,125 mL. RSM was useful to predict the biogas production process in this study successfully. The value of R^2 for RSM was 0.987. This high value of R^2

indicated that the model could be efficiently used for prediction of biogas production from co-digestion of SW in wastewater.

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