Pertanika J. Sci. & Technol. 25 (S): 101 - 110 (2017)

SCIENCE & TECHNOLOGY

Journal homepage: http://www.pertanika.upm.edu.my/

Optimisation of Biomethane Production by Anaerobic Digestion of Food Waste

Nurul Shahida Osman[1], Zainon Mohd Noor[1]*, Zatilfarihiah Rasdi[2], Iwana Izni Zainuddin[3] and Noor Azrimi Umor[3]

[1,2]*School of Biology, Faculty of Applied Sciences, Universiti Teknologi MARA (UiTM), 40450 Shah Alam, Selangor, Malaysia*
[2]*Preclinical Sciences Studies, Faculty of Dentistry Universiti Teknologi MARA (UiTM) Sungai Buloh Campus, 47000 Sungai Buloh, Selangor, Malaysia*
[3]*Faculty of Applied Sciences, Universiti Teknologi MARA (UiTM) Negeri Sembilan, 72000 Kuala Pilah Campus, Negeri Sembilan, Malaysia*

ABSTRACT

Food waste is a mixture of organic residues that affect fermentation process. Thus, appropriate parameters should be optimised to ensure high biomethane production. In this research, response surface methodology (RSM) was utilised for building models, evaluating the significance of several independent factors (pH, temperature, substrate concentration and inocula size) and determining optimum conditions for desirable responses (biomethane yield). The RSM and contour plots set the optimum working factors in order to accomplish the desired biomethane yield. Results suggest that biomethane yield can be increased when pH and temperature are increased. Thus, the main effects of parameters are pH and temperature.

Keywords: Anaerobic digestion, biomethane yield, response surface methodology

INTRODUCTION

Anaerobic digestion (AD) is an organic process that happens when microscopic organisms separate natural matter in the environment with or without oxygen (Zhang et al., 2007). Generally, AD processes are applied to waste water and sewage sludge treatments. However, recently the focus of AD has switched from treatments of waste such as Biology Oxygen Demand (BOD) removal to bioenergy production. An organised anaerobic digestion of biological leftover

ARTICLE INFO

Article history:
Received: 05 January 2017
Accepted: 17 January 2017

E-mail addresses:
nurul_shahida90@yahoo.com (Nurul Shahida Osman),
dzainonmn@salam.uitm.edu.my (Zainon Mohd Noor),
zatilfarihiah@salam.uitm.edu.my (Zatilfarihiah Rasdi),
iwana@ns.uitm.edu.my (Iwana Izni Zainuddin),
noorazrimi@ns.uitm.edu.my (Noor Azrimi Umor)
*Corresponding Author

ISSN: 0128-7680

in a surrounded landfill will create bioenergy in the form of methane. Bioenergy production using AD technique contains around 60% of methane (CH_4) and 40% of carbon dioxide (CO_2) (Molino, Nanna, Ding, Bikson, & Braccio, 2013). Therefore, AD has enormous potential for recuperating bioenergy by using organic waste such as food waste.

Microorganisms are a promising energy source. Good techniques to produce ecologically sound bioenergy synthesis can provide mankind with cheap raw material or substrate, for an environmentally friendly source of energy. It is estimated that approximately 50% of all nourishment produced is gone, changed over or squandered (Joint Declaration against Food Waste, 2010). In Malaysia, 8000 tons of foods are squandered every day (Solid Waste and Public Cleansing Management Corporation, 2008). Thus, food waste is considered as a potential source of bioenergy using the AD technique. Since food waste is a mixture of organic residues that may be affect the fermentation process, suitable parameters need to be optimised to ensure high biomethane production throughout the process.

The RSM is a mathematical and statistical instrument that uses measurable data from different experimental designs to define and concurrently explain multivariate equations (Keshani, Luqman Chuah, Nourouzi, Russly, & Jamilah, 2010). It investigates the connections between several descriptive factors and one or more response factors (Carley, Kamneva, & Reminga, 2004). The aim of this paper is to examine parameters that affect biomethane production using RSM.

MATERIALS AND METHODOLOGY

Inocula and Substrate

An anaerobic sludge was taken from a clarifier tank in Universiti Teknologi MARA (UiTM), Shah Alam, Selangor, and utilised as an inoculum. It (anaerobic sludge was used within a week, and fresh inocula were gathered again from the similar location to ensure consistency in their characteristics.

Food wastes were obtained from the Cafeteria, UiTM. They consisted in equal parts wastes from the kitchen, namely fruit peel and vegetable parts. As the waste contained impurities, they were sieved to remove the coarse contaminants before the granulating process. A weekly sampling was done.

Analytical Methods and Data Analysis

The parameters analysed for the classification were: Total Solid (TS), Volatile Solid (VS), Total Suspended Solid (TSS), Volatile Suspended Solid (VSS) and Chemical Oxygen Demand (COD) contents referring to the Standard Methods of American Public Health Association (APHA, 1998). Composition of carbon (C), hydrogen (H), nitrogen (N), sulphur (S) and C/N ratio were analysed using Thermo Scientific Flash 2000 CHNS-O. Protein and carbohydrate contents were also analysed using Bradford Assay and High Performance Liquid Chromatography (HPLC) at mobile phase of 100% distilled water, temperature 85°C, flow rate of mL/min with glucose used as standard. The formula in (1) was used to model the kinetics of the biomethane production and to determine specific biomethane production potential (Methane yield). Areas of methane sample and standard methane were measured using gas chromatography for each digester. The methane yield was measured in the unit of ppm.

$$\frac{\text{Area of methane sample}}{\text{Area of standard methane}} \times 57.6 \tag{1}$$

Biomethane Fermentation

The experiment was aimed at estimating the anaerobic digestion of four dissimilar parameters: temperature, pH, substrate concentration (in volume of 68.6 VS/l) and inocula concentration (in volume of COD ranging 75 – 99 mg/l). The tests were carried out in 160 mL serum flasks comprising 100mL media. All serum bottles were loaded with a specific volume of substrate and inoculated with a specific volume of inocula which was then adjusted to a specific pH before incubation for 24 hours in serum bottles to produce methane gas for 15 days of Hydraulic Retention Time (HRT). All bottles were flushed with nitrogen gas to ensure anaerobic conditions throughout the experiments and tightly capped with rubber septum (butyl rubber) before incubation at 35°C. The total gas is measured at 24-hour interval by releasing the pressure in the bottles using 10 mL syringe. The biomethane production was analysed using a gas chromatography equipped with a thermal conductivity detector, and the column was packed with Porapack Q (80/100 mesh). The temperature of injector and column were kept at 0°C and 50°C. Nitrogen was used as the transporter gas at a flow rate of 30 ml/min, and 8% methane gas was used as standard.

Experimental Design

The RSM was used to define the optimum conditions of the biomethane production during anaerobic fermentation of food waste and anaerobic sludge by using Statsoft Statistica 6.0. The optimisation procedure was divided into two designs - Two-level Factorial Design and Central Composite Design (CCD).

In the factorial design, the influence of all experimental variables, factors and interaction effects on the response are studied. Four variables, which are expected to affect biomethane production, were selected based on an earlier study by the present authors. The factors in the two-level factorial design are listed in Table 1.

Table 1
Variables in actual values, for screening by the two-level factorial design

	Variable	Unit	Low Level (-1)	High Level (+1)
A	pH	pH	6.5	8.2
B	Temperature	°C	3.5	37
C	Inoculum Size	%	105	20
D	Substrate Concentration	g/L	40	80

A CCD was established after identifying significant factors by the two-level factorial design. It was utilised to make models between the variables, to optimise the biomethane yield and to decide the main effects of parameters. Therefore, 13 tests were created based on the second-order CCD with two independent variables. The variables in CCD are listed in Table 2. The tests were randomised in order to minimise the effects of unsolved variables in the actual responses due to unimportant factors.

Table 2
Coded and actual values of variables selected for CCD

Variables	Unit	-2	-1	0	+1	+2
pH	pH	6.15	6.5	7.35	8.2	8.55
Temperature	°C	34.59	35.00	36.00	37.00	37.41

RESULTS AND DISCUSSION

Two-Level Factorial Design

In the two-level factorial design of four factors concept, a total matrix would have been based on 24 = 16 runs and 6 were centre point runs for statistical reasons. Thus, a factorial design matrix of 22 runs was used. Each variable was examined at high (+1) and low (-1) levels. The runs of the centre point were included in the matrix, and statistical study was used to recognise the effects of each factor on biomethane production. The runs were randomised for statistical purposes. The significance of factors was identified at confidence level above 95 % (P>0.05). Table 3 shows the maximum and minimum methane yields are 2.97 ppm and

Table 3
Analysis of variance for the regression model and the respective model terms

Variables	F-Ratio	P-Value
A	42.01	0.0001
B	10.30	0.0051
C	7.420	0.0144
D	7.09	0.0164
AB	23.79	0.0001
AC	3.29	0.0872
AD	0.72	0.4088
BC	8.40	0.0100
BD	3.70	0.0714
CD	0.045	0.8349
ABC	09.60	0.0065
ABD	13.82	0.0018
ACD	36.54	0.0001
BCD	47.06	0.0001
Lack of Fit		0.6470
R^2		0.9263

1.13 ppm respectively. Here, A, B, C, D, AB, AC, AD, BC, BD, CD, ABC, ABD, ACD, BCD are significant model terms. According to the analysis of variance, the model for methane production was highly significant ($P<0.0001$), while the lack of fit was not significant ($P>0.05$). The coefficient of determination (R^2) was 0.9263.

Central Composite Design

Based on the identification of factors by the two-level factorial design, a central composite design was created for factors that significantly affected methane production. All the non-significant variables were kept at central points ('0' coded level) of the levels used in the two-level factorial design. Table 2 above shows the coded and real values of the levels of factors selected in CCD. The design matrix of the variables together with the experimental results are shown in Table 4.

Table 4
Central composite design of variables for methane yield

Runs	pH	Temperature (°C)	Methane Yield (ppm)
1	7.35	34.59	0.6197
2	8.20	37.00	0.9702
3	8.55	36.00	1.0573
4	6.15	36.00	0.5085
5	7.35	36.00	0.6236
6	6.50	35.00	0.5139
7	8.20	35.00	0.7695
8	6.50	37.00	0.5975
9	7.35	36.00	0.6374
10	7.35	36.00	0.6937
11	7.35	37.41	0.7080
12	7.35	36.00	0.6990
13	7.35	36.00	0.7018

In Tables 5 and 6, the response surface study permitted the development the experimental connection where each response variable (Y_i) was assessed as a function of pH (X_1) and temperature (X_2) and expected as the sum of constant (β_0), two first order effects (linear terms in X_1 and X_2; one interaction term in X_1X_2) and two second order effects (quadratic terms in $X_1{}^2$ and $X_2{}^2$). The obtained results were analysed using ANOVA to get the significant model terms. Only those found significant ($P<0.05$) were included in the reduced model. As shown in Table 6 and Equation 2, the obtained model for expecting the response variables explained the main, quadratic and interaction effects of factors affecting the response variables. The predictable regression coefficients of the polynomial response surface model along with the corresponding R^2 values are shown in Table 5. It was found that the values of "Prob > F" less than 0.05 indicate the model terms are significant.

In this case, X_1, X_2 and $X_1{}^2$ are significant model terms. Values greater than 1.1 indicated the model terms are not significant. Analysis of variance also confirmed that the model is highly significant (P>0.05) for all response variables. The probability (P) values of all regression model were less than 0.05 which had no indication of lack of fit. The R^2 values for these response variables were higher than 0.80 (0.9613), thus ensuring an acceptable qualification of the regression models to experimental data. The following response surface models Equation (2) were plotted to the response variable (Y_1), two independent variables (X_1 and X_2):

$$Y_1 = 0.67 + 0.18X_1 + 0.051X_2 + 0.053X_1^2 - 6.269E - 0.03X_2^2 + 0.29X_1X_2 \tag{2}$$

Table 5

Regression coefficients, R^2, adjusted R^2 probability values and lack of fit for each variable

Regression coefficient	Methane Yield (ppm)
β0	+0.67
β1	+0.18
β2	+0.051
β3	+0.053
β4	− 6.269E-003
β5	+0.29
Regression (p-value)	0.9613
Lack of fit	0.3525

Table 6

ANOVA and regression coefficients of the first and second order polynomial regression models

Variables		Main effects		Quadratic effects	Interaction effects
Y_1	X_1	X_2	$X_1{}^2$	$X_2{}^2$	$X_1 X_2$
p-value	0.0001	0.0096	0.0110	0.6987	0.1966
F-ratio	146.69	12.46	11.74	0.16	2.04

Optimisation of Biomethane Production

The predicted versus actual plots for concentration (Y_1) is shown in Figure 1. The observed points on these plots indicate that the actual values are dispersed relatively close to the straight line and in this case, $R^2 = 0.9613$. The 3D response surface was plotted to well imagine interface effects of independent variables on the biomethane yield. The plots are shown as a function of two factors at one time. These plots are useful in understanding both the main and the interaction effects of these factors.

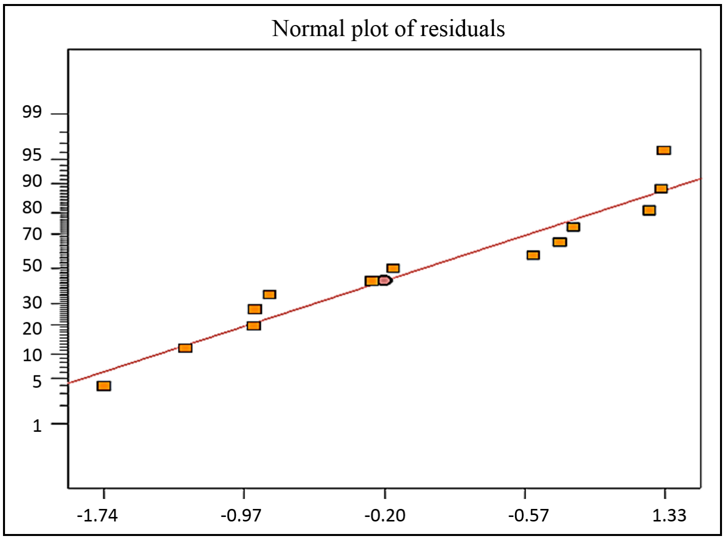

Figure 1. Predicted versus actual data for biomethane yield

As shown in Figures 2 and 3, the existence of curvatures in the biomethane yield curve set that the variation of methane yield (Y_1) was clarified as a nonlinear function. It is clear from the figures that the biomethane yield increased corresponding with the pH and temperature. Referring to Table 6, the main parameter effects are in the following order: Main effect pH > temperature. P values of parameters are 0.0001 and 0.0096 respectively. It can be seen from Figures 2 and 3, when pH was 7.35, the methane yield is 0.6711 ppm at temperature 36°C. This was supported by Foster, Perez & Romero (2008) where a higher temperature implies greater biomethane production in a shorter time.

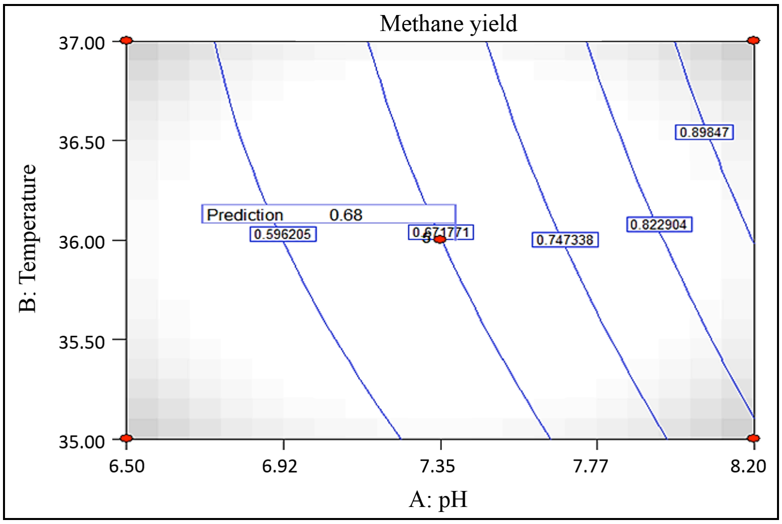

Figure 2. Contour plots graph for biomethane yield as a function of pH (A) and temperature (B)

A sharp increase in temperature should be avoided because they can bring a reduction in biomethane production due to the death of particular bacterial strains that are sensitive to temperature changes. Moreover, each group of microorganisms has a dissimilar optimum pH range. Methanogenic bacteria are extremely sensitive. The optimum pH is between 6.5 and 7.2 (Turovskiy & Mathai, 2006). Lower pH condition was due to accumulation of volatile fatty acids (VFAs) and increment of alkalinity (Appels, Baeyens, Degrève, & Dewil, 2008). The pH values beneath the optimum temperature can restrain methane bacteria activity (Appels et al., 2008; Nurul Shahida, Zainon, & Zatilfarihiah, 2015).

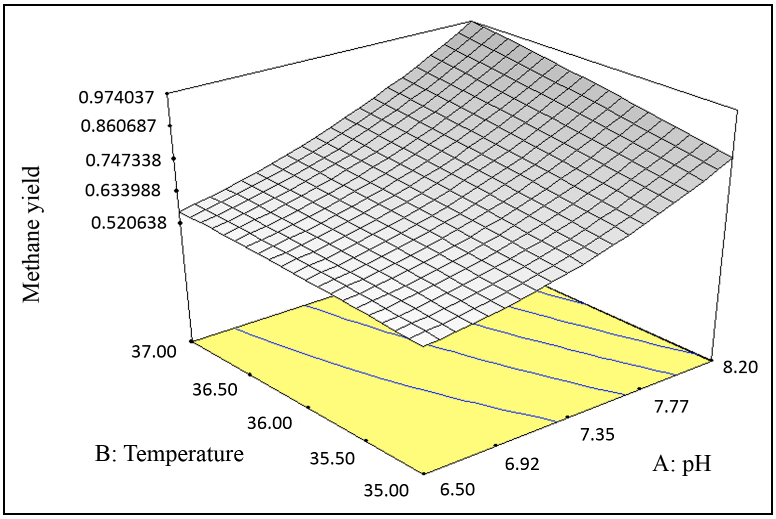

Figure 3. Three-Dimensional plots graph for biomethane yield as a function of pH (A) and temperature (B)

CONCLUSION

The RSM is a convenient technique for building models, evaluating the significance of several independent variables (pH, temperature, substrate concentration and Inocula size) and determining optimum conditions for desirable responses (biomethane yield). The RSM and contour plots set the optimum functioning factors that can be obtained graphically in order to achieve the desired biomethane yield. Therefore, it is suggested that the biomethane yield can be increased corresponding with the pH and temperature. The main effects of parameters are in the following order: Main effect of pH > Temperature.

ACKNOWLEDGEMENTS

The authors acknowledge with gratitude the editorial support from their main supervisor Assoc. Prof. Dr. Zainon Mohd Noor and co-supervisor Zatilfarihiah Rasdi, Iwana Izni Zainuddin and Noor Azrimi Umor. The authors also gratefully acknowledge Universiti Teknologi MARA (UiTM) Malaysia in providing the Exploratory Research Grant Scheme (ERGS) Fund: 600-RMI/ERGS 5/3 (11/2013).

REFERENCES

Appels, L., Baeyens, J., Degrève, J., & Dewil, R. (2008). Principles and potential of the anaerobic digestion of waste-activated sludge. *Bioresour Technol, 34*, 755- 781.

American Public Health Association (APHA). (1998). Standard methods for the examination of water and wastewater. 18th ed. American Public Health Association, Washington, DC, USA.

Carley, K. M., Kamneva, N. Y., & Reminga, J. (2004). Technical report of response surfaces methodology CASOS. USA: School of Computer Science, Carnegie Mellon University, ISRI-CASOS.CMU-ISRI-04136.

Foster, T., Perez, M., & Romero, L. I. (2008). Thermophilic anaerobic digestion of source- sorted organic. Fraction of municipal waste. *Bioresour Technol, 99*, 6763-6770.

Joint Declaration against Food Waste. (2010). EU- and UN- Declaration. Retrieved from www.greencook.n/images/stories/joint_declaration_against_food_waste.pdf.

Keshani, S., Luqman Chuah, A., Nourouzi, M. M., Russly A. R., & Jamilah, B. (2010). Optimization of concentration process on pomelo fruit juice using Response Surface Methodology (RSM). *International Food Research Journal, 17*, 733- 742.

Molino, A., Nanna, F. Ding, Y, Bikson, B., & Braccio, G. (2013). Biomethane production by anaerobic digestion of organic waste. *Fuel, 103*, 1003-1009.

Nurul Shahida, O., Zainon, M. N., & Zatilfarihiah, R. (2015). The preliminary anaerobic fermentation of food wastes using anaerobic sludge. *Advances in Environmental Biology, 9*, 11-15.

Solid Waste and Public Cleansing Management Corporation, (2008). Retrieved from http://www.ppsppa.gov.my.

Turovskiy, I. S., & Mathai, P. K. (2006). *Wastewater Sludge Processing*. Wiley, New York, USA.

Zhang, R., El-Mashad, H. M., Hartman, K., Wang, F., Liu, G., Choate, C., & Gamble, P. (2007). Characterization of food waste as feedstock for anaerobic digestion. *Bioresour Technol, 98*, 929-935.