18th International Conference on Sustainable Energy Technologies

20-22 August 2019 - Kuala Lumpur - Malaysia

Sustainable Energy Towards the New Revolution

Conference Proceedings
Volume 2

Proceedings of the

18th International Conference on Sustainable Energy Technologies

SET 2019

20-22 August 2019
Kuala Lumpur, Malaysia

Volume 2

Edited by

Professor Saffa Riffat, Dr Yuehong Su,
Professor Norli Ismail and Dr Mardiana Idayu Ahmad

SET 2019 Admin Support
Department of Architecture and the Built Environment
Faculty of Engineering, University of Nottingham

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Author(s) Surname, Author(s) Initial(s), 2019. 'Title of paper'. In Riffat, Su, Ismail, Ahmad. ed., **Sustainable Energy Towards the New Revolution**: Proceedings of the 18th International Conference on Sustainable Energy Technologies, 20-22 August 2019, Kuala Lumpur, Malaysia. University of Nottingham: Buildings, Energy & Environment Research Group. Volume X, pp XX-XX. Available from: nottingham-repository.worktribe.com/ [Last access date].

ISBN-13: 9780853583318

Version: 10.02.2020

Foreword

The 18th International Conference on Sustainable Energy Technologies was a significant international academic event in the domain of world sustainable energy technologies with a theme of '*Sustainable Energy Towards the New Revolution*'. The conference aimed to provide a forum for the exchange of latest technical information, the dissemination of up-to-date research results, and the presentation of major topics including sustainable energy, low carbon technologies, eco-cities, energy security and environmental policy.

Held from August 20th – 22nd 2019 in Kuala Lumpur, Malaysia, the conference was a collaboration between the World Society of Sustainable Energy Technologies (WSSET), the Universiti Sains Malaysia and University of Nottingham. World-renowned experts and scholars in the area, representatives of prominent enterprises and universities attended to discuss new developments and achievements in the field, as well as promoting academic exchange, application of scientific results, university-industry collaboration and government-industry collaboration.

The papers contained in these proceedings focus on topics such as Energy Storage for the Age of Renewables; Research, Innovation and Commercialisation in Sustainable Energy Technologies; Integrating Planning & Policy, Architecture, Engineering & Economics; Energy and Environment; Engineering Thermo-physics; and Systemic Change for Cities.

About 230 delegates from 30 countries attended SET2019; nearly 400 abstracts were received and 190 papers have been published in the conference proceedings. The proceedings have therefore been divided into three volumes. I hope you enjoy as much as I did the breadth of work you will find in these proceedings.

We would like to thank all participating authors for their contributions to both the conference and to the publishing of this book. We are also indebted to our international scientific committee for their advice and seemingly endless review of papers. We would also like to thank unreservedly Celia Berry, Zeny Amante-Roberts, Dr Mardiana Idayu Ahmad and Professor Dr Norli Ismail for their tireless efforts in making SET2019 one of the most successful conferences we have held. Also a huge thanks to our sponsors First Solar, PCM Products Ltd and Professor Terry Payne.

Professor Saffa Riffat
Chair in Sustainable Energy Technologies
President of the World Society of Sustainable Energy Technologies
Fellow of the European Academy of Sciences
SET 2019 Chairman

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#225: Anaerobic digestion of synthetic wastewater (acetate): effect of COD and C/N ratio in batch mode

Laddawan NOYNOO[1], Hathaikarn THONGPAN[2], Sunwanee JIJAI[3], Norli ISMAIL[4], Chairat SIRIPATANA[5] and Nirattisai RAKMAK[6]

[1] School of Engineering and Resources, Walailak University, Nakhon Si Thammarat, Thailand 80160, ladda1995.ln@gmail.com.
[2] School of Engineering and Resources, Walailak University, Nakhon Si Thammarat, Thailand 80160, hathaikarn_thpn@hotmail.com.
[3] Faculty of Science Technology and Agriculture, Yala Rajabhat University, Yala, Thailand 95000, sunwanee.j@yru.ac.th.
[4] School of Industrial Technology, Universiti Sains Malaysia 11800, Pulau Pinang, Malaysia, norlii@usm.my.
[5] Biomass and Oil-Palm Excellence Center and School of Engineering and Resources, Walailak University, Nakhon Si Thammarat, Thailand 80160, schairat61@gmail.com.
[6] Biomass and Oil-Palm Excellence Center and School of Engineering and Resources, Walailak University, Nakhon Si Thammarat, Thailand 80160, nirattisai.ra@wu.ac.th.

This paper attempts to understand the effect of the main operating variables, find an optimized operating condition, and develop a model for biogas production from Palm Oil Mill Effluent (POME). However, POME is a complex substrate with mixed sources of carbon and nitrogen as well as other essential nutrients that makes it difficult to understand clearly how the C/N ratio and volatile fatty acid (VFA) affect the efficiency of the anaerobic digestion (AD) process. In order to clarify the effects of the main variables and be able to develop a deeper and more realistic mathematical model from focused/basic experimental data, the author chose to optimize the biogas production using a synthetic wastewater (acetate with nutrient supplements instead of POME) digested in batch mode. The effects of C/N ratio (for 10/1 to 50/1) and COD (in range of 1,000-12,000 mg/L) were studied in 1000 mL-reactors containing synthetic wastewater and inoculum (sludge of POME). The results of this study were as follows: the optimal for batch mode and COD (as acetate) was 1,000 mg/l of COD with gave %CH4 greater than 50, CH4 yield was 72.90 mg CH4/g CODremoval and 80.62% of CODremoval. The optimal C/N ratio was 30/1 with %CH4 around 35% and 44.49 of %COD removal. Moreover, all of the experiment data fitted well with both Gompertz and Monod models. Furthermore, it gave a corrected prediction of steady state performance of the biogas both qualitatively and quantitatively. In this work, a model to predict the composition of biogas production from the substrate composition was also preliminarily developed which predicted the biogas composition with a correct trend.

Keywords: C/N ratio; COD; synthetic wastewater; mathematic model; anaerobic digestion

1. INTRODUCTION

The palm oil industry is the biggest agro-industry in Southern Thailand, contributing over 45,000 million Baht each year (2.74 million tons of crude palm oil annually). Palm oil mills are wet factories, producing huge amounts of wastewater which are used for biogas production through anaerobic digestion (AD) processes. A number of published articles on various aspects of AD of palm-oil mill effluents have been reported, increasingly its co-digestion with other nutrient sources has been investigated (Soh Kheang Loh, 2019). However, POME and other agro-industrial wastes are too complex to understand how the main parameters (such as C/N ratio) affect the AD process performance in a clear manner. For example, there are at least two approaches to understand the effect of C/N ratio on methane yield and its evolution rate. The simplest way is to design experiments such that varying C/N substrates (by adding ammonium nitrogen or by co-digesting with high-nitrogen substrates) are tested for biological methane potential (BMP) and then applying statistical methods to find how C/N ratio affect the AD performance. This approach potentially has a large degree of ambiguity because of complex interactions between various factors which obscure the contribution of the C/N ratio. Experimental design would also be more involved because many variables have an influence in the process at the same time while we are focusing on only 1 or 2 factors. Consequently, excessive resources would be needed to clarify the effect of C/N whereas predictive power of the resulting statistical models is limited; they are restricted to specific cases. Since the interaction between multi-substrate and microbial consortium are too complex to be described by moderate kinetic models, kinetic modelling has not been popular or very meaningful.

In this work, a different approach called "the approach of increasing complexity" (called PIC, in short) was used to understand the effect of C/N ratio on AD process in a fundamental way. By looking at main metabolic pathways of the AD process, one would find that acetate is the intermediate to be directly converted to methane and carbon dioxide in the final step of methanogenesis (Enzmann et al, 2018). How much methane produced through this route depends on the substrate compositions and physico-chemical condition surrounding the microbes. However, in most practical AD process, more than 60% of methane produced was derived from acetate. (CU Welte, 2018)

Here we still used the microbial sludge (seed) from an existing POME anaerobic digester in BMP tests. However, instead of using POME and other co-substrates we prepared a synthetic wastewater from sodium acetate (adjusted to different C/N ratios) and essential nutrients for the growth of methanogens. This synthetic wastewater has only acetate as its carbon source. By this way we expected to understand how C/N affects the kinetic of methane production without too many carbon sources getting involved at the same time. We also tried to find out that how far this simple AD system follows Monod and Gompertz kinetics given its inherent simplicity.

2. KINETIC OF BIOGAS

Monod and Gompertz kinetics were proposed to study the effect of biogas production, concentration of substrate and microbial biomass (Tongpan et al., 2016). It was based on one-limiting-substrate assumption and sigmoidal growth-rate as a function of the substrate concentration (Monod, 1949). If no microbial death occurs during the fermentation/AD process and all yield coefficients is constant, we can write the following system of differential equations.

Equation 1:

$$\frac{dX}{dt} = \mu X = \frac{\mu_m S}{K_S + S} X$$

Equation 2:

$$\frac{dS}{dt} = \left(-\frac{1}{Y_{X'/S}} \right) \mu X$$

Equation 3:

$$\frac{dS}{dt} = \left(-\frac{Y_{P/S}}{Y_{X/S}} \right) \mu X$$

Substituting the equation below into Equations (1) – (3),

$$Y_{PS} = \Delta P / \Delta S, Y_{X'S} = \Delta X' / \Delta S, Y_{PX'} = \Delta P / \Delta X' = Y_{PS} / Y_{X'S} \text{ and } P_0' / Y_{PS} = X_0' / Y_{X'S}$$

we obtain the following:

Equation 4:

$$\frac{dS}{dt} = \mu \left(S - S_0 - \frac{X_0}{Y_{X/S}} \right)$$

Equation 5:

$$\frac{dP}{dt} = Y_{P/S} \mu \left(\frac{P}{Y_{P/S}} - \frac{P_0}{Y_{P/S}} - \frac{X_0}{Y_{X/S}} \right) = \mu P$$

Solving Equations (1)-(5), one obtains the following analytical solution

Equation 6:

$$t = \left[\left(K_S / C \right) ln\left(S_0 (C - S) / S(C - S_0) \right) + ln\left(\left(C - S \right) / \left(C - S_0 \right) \right) \right] / \mu_m$$

Where $C = \left(X_0 / Y_{X'S}\right) + S_0 = \left(X_0 / Y_{X'S}\right) + \left(P_\infty / Y_{PS}\right) = \left(P_0' + P_\infty\right) / Y_{PS} = \left(P_\infty' / Y_{YS}\right)$

The prime notation in X′ referred to the total accumulative microbes due to growth if there were no deaths.

Where:

- X = concentration of active microbes (cell biomass) at a specific time
- μm = maximum growth rate at a particular substrate concentration
- μ = specific growth rate at a particular substrate concentration
- k_d = specific death rate
- K_s = Monod saturation constant

The equivalent biogas generated by X is defined as P′=P+ P'_0 However, although very useful for interpretation, this relation is only hypothetical because we did not observe how microbial consortia had evolved before we start the batch AcoD experiments. Thus, P'_0 or the estimated X_0 should be interpreted as initial microbial activity rather than the actual microbial density at the initial time of AcoD process. In general, high P'_0 (or high X_0) indicates high initial microbial activity which can be translated as relatively high microbial density and vice versa.

3. MATERIALS AND METHODS

3.1. Experimental set-up

AD was carried out using the synthetic wastewater that consisted of mixture of 5 chemicals and acetate at different ratio as in Table 1 to give the COD values of 1000-12,000 mg/L.

Table 1: Composition of the synthetic wastewater

Chemicals (g/l)	Concentration of COD (mg/l)				
	1,000	3,000	6,000	9,000	12,000
Sodium Acetate)g/l)	2.09	6.27	12.54	18.81	25.08
Sodium dihydrogen phosphate)g/l)	0.2083	0.625	1.25	1.875	2.5
Ammonium Chloride)g/l)	0.083	0.25	0.5	0.75	1
Hydrochloric acid)ml/l)	0.083	0.25	0.5	0.75	1
Nickel (II) chloride)mg/l)	0.083	0.25	0.5	0.75	1
Ferric chloride)mg/l)	0.083	0.25	0.5	0.75	1

The inoculum was collected from an anaerobic sludge at the bottom of the active anaerobic pond in POME waste treatment plants in Nakhon Si Thammarat province. The inoculum was kept at 40ºC for 3 days to completely consume the residue nutrients. The AD was performed under room temperature using 1 L volume of serum bottles. A working volume of 850 ml was used in all experiments. The serum bottles were covered with the rubber stoppers and sealed with aluminium caps. The volume of biogas was measured daily by using water displacement method. The experiment setup is shown in Figure 1.

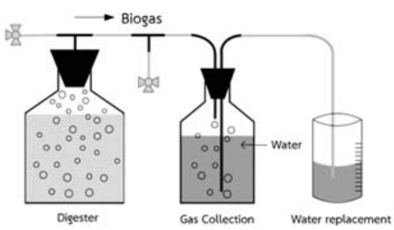

Figure 1: The experiment set-up for biogas production in batch mode

3.2. Analytical methods

Digestate was sampled from reactors and was performed every two days. In all experiments, pH, Chemical oxygen demand (COD), Total solid (TS), Total volatile solid (TVS), Suspended Solid (SS), Volatile Suspended Solid (VSS), Mixed Liquor Suspended Solid (MLSS), Mixed Liquor Volatile Suspended Solids (MLVSS), Alkalinity and Volatile Fatty Acid (VFA) were evaluated according to standard methods for the examination of water and wastewater (APHA, 2017). Daily biogas production was settled by a water displacement method. The composition of biogas was analysed using Gas Chromatography.

3.3. Experiment design

Activity 1: Study the effect of COD on AD system

The inoculum for this study was treated before using as seed in batch experiments by daily adding 10% (by volume of inoculum) of synthetic wastewater into digester until 100% of volume. AD batch system was set up in 5 different conditions including mixing of synthetic wastewater at 1,000, 3,000, 6,000, 9,000 and 12,000 mg/l of COD and inoculum at fixed 28/1 of C/N ratio. The experiments were conducted at 35ºC until batch completion. The experiments were duplicated in all digesters. A flowchart of this experiment designed for study the effect of COD on the maximum biogas production in batch mode was shown in Figure 2.

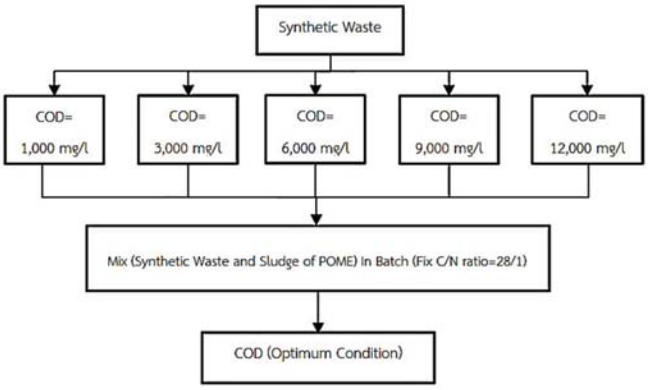

Figure 2: Experiment designed for study the effect of COD on AD system.

Activity 2: Study the effect of C/N ratio on AD system

The optimum COD value from Activity 1 that gave highest biogas yield was used to study the effect of C/N ratio on AD system. The synthetic wastewater and inoculum were prepared at difference C/N ratio including 10/1, 20/1, 30/1, 40/1 and 50/1. The experiments were conducted at 35ºC until batch completion and were duplicated in all digesters. A flowchart of the experiment designed for study the effect of C/N on the maximum biogas production in batch mode is shown in Figure 3.

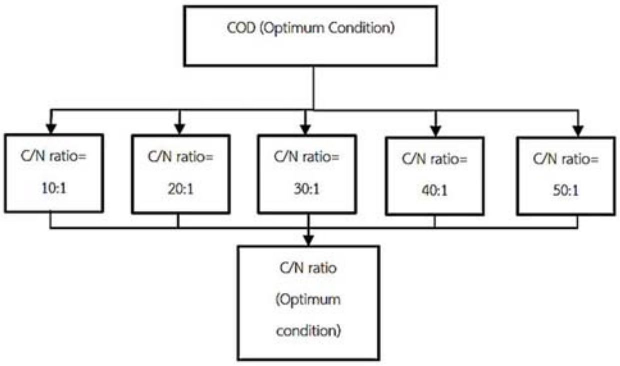

Figure 3: Experiments designed for investigating the effect of C/N on the performance batch AD system.

4. RESULTS AND DISCUSSION

4.1. The effect of COD on AD system

Physico-chemical characterisation of substrate (mixing of synthetic wastewater and inoculum) at the beginning of the anaerobic digestion process was performed. Chemical characterisation of substrate used in this study is showed in Table 2.

Table 2: Characteristics of substrate (mixture of synthetic wastewater and inoculum) used in the digestion experiments.

Parameter	Synthetic wastewater at difference of COD)mg/l)					Inoculum
	1000	3000	6000	9000	12000	
pH	5.82	5.84	5.85	5.90	5.85	6.60
TS, mg/l	1,555	3,890	7,195	11,405	13,641	-
TVS, mg/l	1,050	1,695	2,580	3,790	5,674	-
SS, mg/l	110	130	160	150	260	-
VSS, mg/l	580	845	840	680	565	-
MLSS, mg/l	-	-	-	-	-	28,530
MLVSS, mg/l	-	-	-	-	-	25,730
C/N ratio	25.58	25.58	25.58	25.58	25.58	-

Table 3: Characteristics of substrate (mixing of synthetic wastewater and inoculum) used in the digestion experiments.

COD		ALK (mg/l)	VFA (mg/L)	VFA/ALK	COD (mg/L)	MLVSS (mg/l)	Methane yield (ml CH_4/g COD removal)
1,000	run system	4,875	4,225	0.87	9,677	20,755	72.90
	close system	4,375	3,000	0.39	1,875	39,669	
	%COD removal				80.63		
3,000	run system	5,338	4,263	0.8	10,101	21,259	70.06
	close system	4,925	2,313	0.47	3,279	39,669	
	%COD removal				67.54		
6,000	run system	7,475	6,850	0.92	19,355	18,004	65.05
	close system	6,575	2,313	0.42	3,279	21,980	
	%COD removal				49.18		
9,000	run system	8,175	7,538	0.92	25,806	14,630	53.34
	close system	8,500	3,138	0.37	13,125	15,561	
	%COD removal				49.14		
12,000	run system	9,313	8,500	0.91	19,355	21,224	47.33
	close system	7,475	2,625	0.35	10,625	22,631	
	%COD removal				45.10		

Initial and final characteristics of the digestates from synthetic wastewater and inoculum with different COD levels are shown in Table 3. The cumulative biogas production and %CH_4 of difference COD initial levels in batch digestion of mixed synthetic wastewater and inoculum are shown in Figure 4 and Figure 5. For the COD levels in the range of 1,000-12,000 mg/L, the cumulative biogas production fell in the range of 761-1,711 mL and %CH_4 was more than 50% in all experiments. During the first 5 days, yield of cumulative biogas production in all experiments was low because the pH of synthesis wastewater at the beginning of anaerobic digestion was lower than 6 and methanogens need to adjust to this slightly acidic environment. The higher the COD input value, the longer time for the microbes to produce biogas because microorganisms in inoculum needed to adapt before becoming stronger to produce biogas after 5 days.

It was found that 1,000 mg/L of COD input gave the highest methane yield of 72.90 ml CH_4/g COD removed because the VFA/ALK at this condition was in the suitable range of 0.1-0.4 which was non-inhibitive (Nabarlatz, 2013). From results tabulated in Table 3, 1,000 mg/L of COD input value gave maximum %COD removal of 80.62% following by the input COD of 3,000-12,000 mg/L (67.54-45.10). Moreover at the input COD of 1,000 mg/L the AD process gave the maximum MLVSS too. From the results, the optimal input COD was 1,000 mg/L was the optimum condition on for anaerobic digestion of synthetic wastewater and the inoculum based on the maximum cumulative biogas production at day 10 of batch AD, %COD removal and amount of microbial sludge after batch completion.

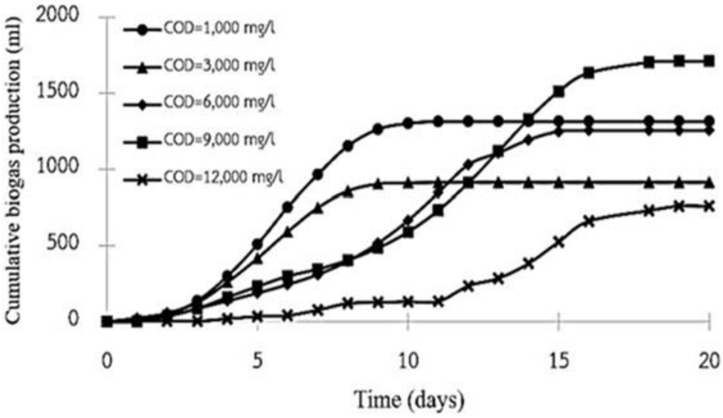

Figure 4: Cumulative biogas production at difference input COD. (at C/N ratio of 25.58)

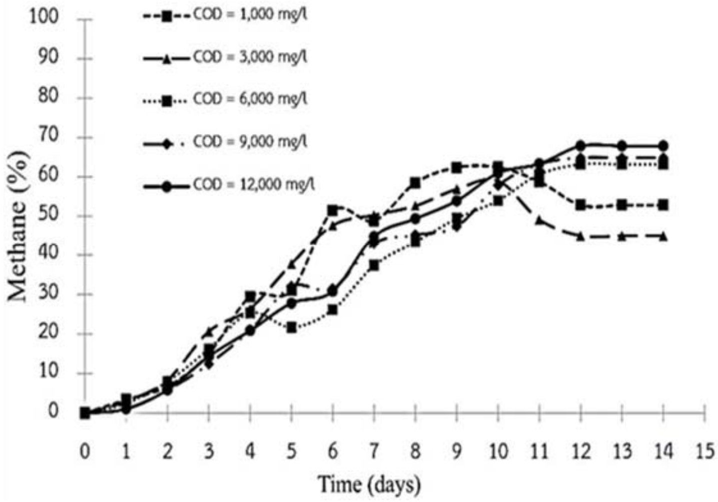

Figure 5: %CH4 of the biogas versus time for different input COD.

4.2. The effect of C/N ratio on the performance of AD system

The results of anaerobic digestion of acetate synthetic wastewater having 1,000 mg/L input COD and inoculum from POME sludge at different C/N ratios (10/1-50/1) are shown in Table 4. Figure 6 and Figure 7 show cumulative biogas production and %CH4 produced at difference C/N ratio of digestates after 30 days of batch AD experiments. From the result, during the first 5 days, yields of cumulative biogas production in all experiments were low because the microorganisms needed some time to adjust in the early stages of digestion. In all batch tests, yield of biogas production were very low in the first 10 days of the batch AD because VFA/ALKs of all experiments were out of the suitable range (0.1-0.4). At C/N ratio of 40/1 and 50/1 VFA/ALK were 0.94 and 0.95 respectively, which is too high for biogas production thus giving low %COD removal in the range of 26.38-28.22%. This was because a high proportion of acetate appeared in prepared synthetic wastewater to adjust C/N ratio. Interestingly, C/N ratio 10/1 gave minimum %COD removal (while producing highest accumulative biogas) as compared to 20/1 and 30/1 C/N ratios. This could be due to a high proportion of nitrogen in these substrates which was converted to toxic ammonia and inhibited the microbial growth. In addition, the anaerobic digestion requires a suitable carbon to nitrogen (C/N) ratio (about 15.6) for the substrate. The substrate with low or high C/N would inhibit the hydrogen producing bacteria to efficiently produce fermentative hydrogen (Xia et al., 2014). Whereas, the C/N ratio of microalgae biomass was too low due to the high protein content and relatively low carbohydrate content. Hence, flocculants with high carbon content could be used to improve the C/N ratio and the mixture could be used as the substrate for later energy conversion. From the result, C/N at 30/1 was the optimum condition to produce biogas at maximum accumulated biogas production and %CH_4. (Xia et al., 2014).

Table 4: Characteristics of substrate (mixture of synthetic wastewater and inoculum) used in the digestion experiments.

COD		ALK (mg/l)	VFA (mg/L)	VFA⁄ALK	COD (mg/L)	MLVSS (mg/l)	Methane yield (ml CH_4/g COD removal)
1,000	run system	4,875	4,225	0.87	9,677	20,755	72.90
	close system	4,375	3,000	0.39	1,875	39,669	
	%COD removal				80.63		
3,000	run system	5,338	4,263	0.8	10,101	21,259	70.06
	close system	4,925	2,313	0.47	3,279	39,669	
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6,000	run system	7,475	6,850	0.92	19,355	18,004	65.05
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12,000	run system	9,313	8,500	0.91	19,355	21,224	47.33
	close system	7,475	2,625	0.35	10,625	22,631	
	%COD removal				45.10		

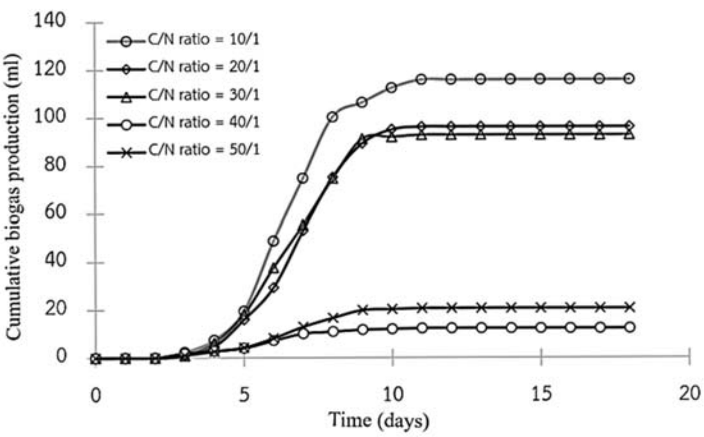

Figure 6: Cumulative biogas production at difference C/N ratio.

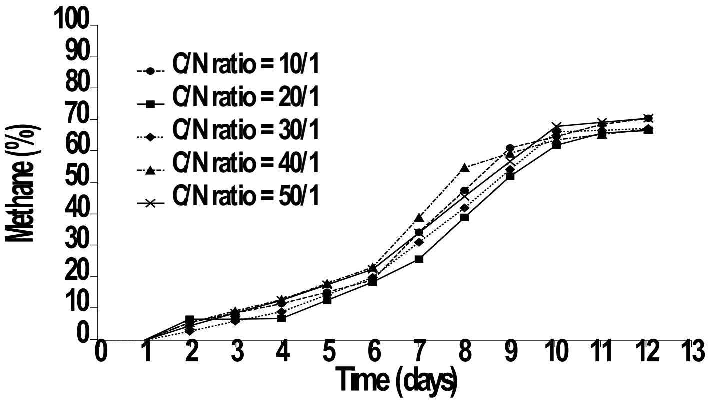

Figure 7: %CH_4 production at difference C/N ratio.

4.3. Fitting the data set to Gompertz models

The results of curve-fitting for Gompertz and Monod models are presented in Figure 8 and Figure 9. The best estimated parameters including those of the corresponding original models are summarised in Table 5.

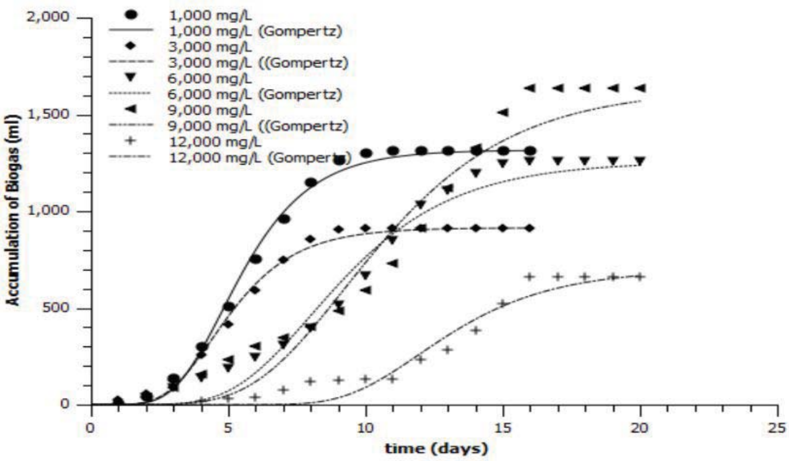

Figure 8: Biogas accumulation for Gompertz model.

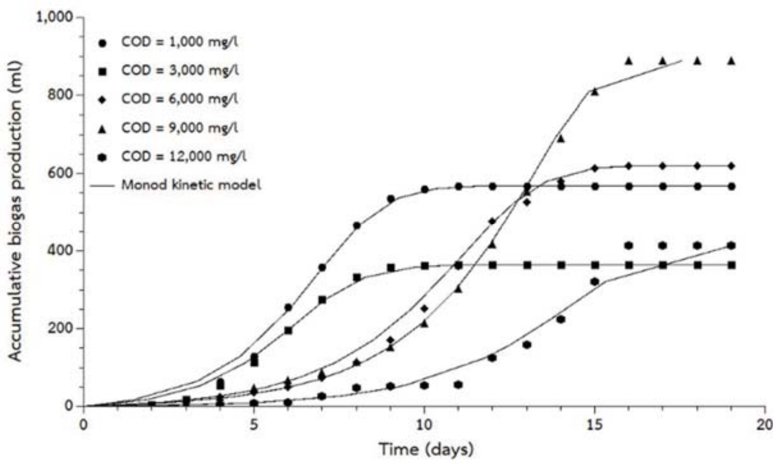

Figure 9: Biogas accumulation for Monod kinetic model.

Table 5: Parameters and the best-fit parameter (R^2) of accumulative biogas production for Gompertz and Monod model.

Model	Parameter	COD concentration (mg/l)				
		1,000	3,000	6,000	9,000	12,000
Gompertz equation $P' = (P_\infty + P_0)\left[\exp\left(-\frac{\gamma_0}{\alpha}\exp(-\alpha t)\right) - \exp\left(-\frac{\gamma_0}{\alpha}\right)\right]$	$\gamma_0\ (d^{-1})$	8.7541	8.5404	5.8172	4.6106	9.0661
	$\alpha\ (d^{-1})$	0.5708	0.6026	0.3503	0.2965	0.2834
	Fitted $P_\infty\ (ml)$	1,315	913	1,255	1,634	785
	R^2	0.99	0.99	0.98	0.96	0.96
Monod kinetic of producing biogas $t = \left(\frac{1}{\mu_m}\right)\left(\left(\frac{K_S Y_{PS}}{P_\infty + P_0'}\right)\ln\left(\left(\frac{P_\infty}{P_\infty - P}\right)\left(\frac{P_0'+P}{P_0'}\right)\right) + \ln\left(\frac{P_0'+P}{P_0'}\right)\right)$	$K_S\ (mg/l)$	5,000	5,000	5,000	5,000	5,000
	$Y_{PS} (ml/mgCOD)$	0.0729	0.0701	0.0651	0.0533	0.0473
	$P_\infty\ (ml)$	570	365	625	890	415
	$P_0'\ (ml)$	15	9	7	6	1.6
	$\mu_m\ (d^{-1})$	0.85	1.16	0.6	0.48	0.6
	R^2	0.99	0.99	0.99	0.98	0.97

The model fitting clearly shows that corrected Gompertz model represented all data sets very well. These results confirm that the Gompertz model is highly suitable for describing batch AD data for simple-(single-) substrate surrounded by a high density of active microbial biomass which is true in these cases. Monod kinetics on the other hand is more suitable for the cases of relatively low biomass concentration.

5. CONCLUSION

In summary, this work, to some extent, has clarified the effect of COD levels of purely volatile fatty acid (acetate) and the C/N ratio on the performance of batch AD systems for this simplest system. This work almost confirm the usability of the Gompertz model for single-substrate surrounded by excessive microbial mass.

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