Title: OPTIMIZATION OF BIOGAS PRODUCTION IN THE SEWAGE TREATMENT PLANT BY USING CENTRAL COMPOSITE DESIGN (CCD).

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Abstract

Most digesters in industrial-scale operate in deficient level and almost nominal due to inefficient process. Optimization of the process may rectify the issue but required a valid method that does not just improve the process yet able to unravel the eventuality of the intricate process if the adjustment needed. A proper tool is required. The central composite design (CCD) was implemented in this study to investigate the suitability of this tool for optimization of anaerobic digestion (AD) process. The main effect of pH and HRT studied in CCD acquired from the screening process show the importance of having neutral pH value and long retention period for a better biogas yield. The process with pH 7.0 and HRT 15.7 days, IP 33%, TS 4% and FR 4% found to be the optimum setting for the process. The new setting successfully improved the production output up to 60% compared with baseline (existing setting), while allowing 50% more sludge to be processed. The X^2 goodness-of-fit test indicates that the mathematical model applied in this study is valid at 95% of confidence level with R^2 of 0.9. The results presented in the paper demonstrate the reliability of CCD as optimization tools for AD process in the industrial scale sewage treatment plant (STP).

Keywords: Anaerobic Digestion, Biogas, Wastewater Treatment, Process Optimization, Process Design

1.0 Introduction

2.0

The demand to have a sustainable wastewater technology has stimulated the anaerobic digestion (AD) system to be implemented and established in the treatment plant [1-4]. Biogas generated from the system is invaluable as fuel for electricity or even heat source for the treatment plant in a colder climate. The challenge to have a better process design for AD become a critical issue among operators, researchers, and engineers. Better process design not just increase the efficiency of sludge treatment, it also establishes a solution for an exorbitant cost of operation, especially for electricity cost. However, the challenge to have a standard design that applicable into all types of a process plant in global scale is impracticable due to the contrast environmental condition and various characteristics of wastewater and sludge [1].

Therefore, a tool for troubleshooting and optimizing the process efficiency was urgently needed. The tool should be applicable in all types of wastewater treatment plants and can be implemented even with a different characteristic of sludge, wastewater and climate. Statistical tools widely implemented in various subjects of science and engineering technology [1, 4-7]. But, it still lacks use in wastewater treatment technology due to insufficient and limitation of knowledge and substantial data samples. The statistical method, however, can now be applied and assisted using various types of software that reduce the data samples size with more reliable results. However, the question arises whether it applies to the study of the AD process. The process involves a delicate balance of settings and the tortuous relationship between microorganism.

The AD process entails four steps of reactions which are hydrolysis, acidogenesis, acetogenesis and methanogenesis. Each step of reaction requires a different type of microorganism to react with the sludge and intermediate products such as sugar, volatile fatty acids, amino acids, and ammonia. But, excesses byproducts would ultimately affect the yield return. The inhibition effect on the process becomes a major concern and widely been discussed and studied in numerous researches [8-13]. An ideal AD process is achievable when the inhibition effect on the process been eradicated [9, 10]. But, it is almost impossible to acquire an ideal condition for the AD process with the involvement of diverse microorganism in the process.

Most of the study performed by various researcher around the globe focused on developing suitable parameters setting to achieve the desired process condition and high return [1, 4-7]. The feasibility of adjusting the universal parameters instead of dealing with a specific setting for each reaction in the process [1] become the main reason for it to happen. The improvement on the yield was notable and intriguing [4-7], but lack studies in the optimization part for AD in STP raised a question. The CCD was implemented in this study to investigate and evaluate the method as a tool for process optimization in industrial scale.

2.0 Materials and Methods

2.1 Inoculum and substrate preparation

The sludge was collected from the anaerobic digester at the wastewater treatment plant in Kuala Lumpur. The inoculum was prepared using the mixture of digestate collected from digester (ID) and both thickened sludge (IL) obtained from the digester inlet line with a ratio of 1: 4 respectively. The sewage sludge was stored at 4°C and seen to be the most practical way to source the sludge while minimising the deterioration effects [14]. Table 1 provides a summary of both characteristic.

Table 1 Characterisation for thickened sludge (IL) and digestate (ID)

	IL			ID		
	Min	Max	Mean	Min	Max	Mean
pH	5.6	6.01	5.83	6.44	6.96	6.72
COD	15023	15813	15438	7120	22767	14553
TS (%)	1.43	2.09	1.81	1	4.41	2.1
TSS (%)	1.3	1.58	1.47	0.75	3.02	1.75
VSS (%)	1.1	1.27	1.58	0.63	2.23	1.24
AN (mg/l)	185	342	240.75	250.85	907	539.97
TP (mg/l)	680	976	782.25	340	1250	806
Alkalinity (mg/l)	700	748	724.75	1057	4333	2057.5

The actual plant applied IL as a substrate; however, due to the characteristic variability, the results in laboratory scale would be doubtful. The variability of total solids (TS) was the main culprit of why this phenomenon occurred. A reliable technique for dilution and concentrated the sludge was required to minimized the impact on sludge characteristic [1, 14]. The concentration can be adjusted by adding the discarding liquid phase or adding the solids from centrifuging sludge. The sludge mixture was centrifuged at 2500g for 8 min using benchtop centrifuge (Eppendorf, Germany) to concentrate the sludge mixture, while the resulting supernatant decanted. The sludge supernatant was used to maintain the various physicochemical properties of the sludge, providing a better reflection what would happen in less efficient dewatering process, and the relative soluble COD was deemed to be insignificant when compared to the particulate COD for the volumes added.

2.3 Optimization of factors

The samples prepared by using 40 litre pilot unit with a fixed percentage of IP (33%), TS (4%) and FR (4%) . Only two control parameters chosen to be studied which were pH and HRT. The pH were controlled with the addition of sodium hydroxide (NaOH). The amount of NaOH added was varied to gain the pH value of the sample at 6.0 to 8.0. The experiment was performed for 3 to 24 days based on hydraulic retention time (HRT).

The sample was purged with nitrogen gas to create an anaerobic environment. The water displacement method was used for biogas measurement. The experiment was conducted with ambient temperature and mixing at 55 rpm. All tests were performed in triplicate. The properties of factors are as shown in Table 2.

Table 2 Range and level of factor for optimization process

Factor (Symbol)	-α	-1	0	+1	+ α	Units
pH (A)	6.0	6.3	7.0	7.7	8.0	pH
HRT (B)	3	6.3	13.7	21	24	%

Both of the parameters in Table 2 were represented in alphabetical symbols. As for each level of value studied, the α represented the axial design point, while 1 symbol represented the factorial design points in both positive and negative value. The 0 symbols represented the centre point studied in this optimization process. The experimental data obtained to be fit with the regression to the quadratic model in Equation 1 below:

$$y_i = b_0 \sum_{i=1}^{n} b_i X_i + \sum_{i=1}^{n} b_{ii} X_i^2 + \sum \sum_{i<j} b_{ij} X_i X_j + e \qquad \text{Eq. 1}$$

Where y is the response (measured variable), b_o is the constant coefficient, and b_i, b_{ii}, b_{ij} are coefficient for the linear, quadratic and interaction effect, x_i and x_j are factors (independent/control experimental variables), n the number of variables studied, while e is the error.

Thirteen experiments were performed which consisted of eight factorial points, five replicates for the centre point. The replicates were used to estimate the experimental error. An analysis of variance (ANOVA) and R^2 (coefficient of determination) statistic was used to examine the adequacy of the developed model. The variation of the data around the fitted model (lack of fit) was tested using an F-test. The significance level was stated at 95%, with p-value 0.05. The model validation test was carried out to validate and confirm the equations using the combination of independent variables. Then, the optimal condition obtained from the software was validated by performing some of the suggested optimum points in actual experiments.

2.4 Analytical method

Total solids and volatile solids were measured using APHA method 2540B and 2540E, respectively [17]. The alkalinity, chemical oxygen demand, ammonia were measured using HACH method 8000 and 10031 respectively after the sample had been centrifuged (Eppendorf, Germany) at 2500g for 15min and passed through 0.45 1m membrane filter (Advantec, Japan). pH was determined using a calibrated pH (Mettler Toledo, Switzerland). Biogas composition was measured using Geotech Biogas Analyzer and gas chromatography (GC). The HP Agilent gas chromatography (GC) equipped with a flame ionization detector (FID) was used. The hydrogen used as a carrier gas is injected at 25ml/min. The HP-Plot Q column suggested by the supplier was used and operated at 150ºC. All the measurement were taken in triplicate.

Results and Discussion

Table 3 show the results from optimization test. The highest yield obtained is at 5.89 l biogas/g COD and the lowest at 1.36l biogas g/COD with residuals difference of 0.1038 and 0.813 respectively.

Table 3 Experimental data for optimization of Biogas production

Standard Order	Actual Value (l biogas/ g COD)	Predicted Value (l biogas/ g COD)	Residuals
1	1.373	2.2190	-0.8460
2	1.36	2.1730	-0.8130
3	4.947	5.4313	-0.4843
4	4.72	5.1713	-0.4513
5	5.093	4.4211	0.6719
6	4.83	4.2047	0.6253
7	1.893	0.9886	0.9044
8	5.773	5.3801	0.3929
9	5.893	5.7892	0.1038
10	5.733	5.7892	-0.0562
11	5.893	5.7892	0.1038
12	5.627	5.7892	-0.1622
13	5.8	5.7892	0.0108

Analysis of Variance (ANOVA)

The analysis of variance (ANOVA) was performed to estimate the coefficients of the model, check the significance of each parameter, and indicate the interaction strength of each parameter. The confidence level from the ANOVA analysis in Table 6 was higher than 95%, while the *p-value* of the model was less than 0.0021. The model with *p-value* <0.05 was statistically significant. The second-order quadratic model F-value of 12.7154 also implies that the model is significant. The *p-value* or "*Prob> F*" shows that there is only 0.21% chance that "Model F-value" this large could occur due to noise which implied that the model was suitable for this study. In this study, B, A^2, B^2 are significant model terms.

Table 4 ANOVA for optimization of biogas production from STP; response biogas yield (l biogas/ g COD)

Source	Sum of Squares	df	Mean Square	F- Value	***p-value*** **Prob > F**	
Model	33.4295	5	6.6859	12.7154	0.0021	significant
A-A	0.04681	1	0.0468	0.0890	0.7741	
B-B	19.2856	1	19.2856	36.6777	0.0005	
AB	0.0114	1	0.0114	0.0218	0.8869	
A^2	3.7905	1	3.7905	7.2088	0.0313	
B^2	11.8002	1	11.8002	22.4418	0.0021	

Residual	3.6807	7	0.5258			
Lack of Fit	3.6296	3	1.2099	94.6441	0.0004	significant
Pure Error	0.0511	4	0.0128			
Cor Total	37.1102	12				
R-Squared(R^2)	0.9008					

Values of "*Prob> F*" <0.0500 indicate model terms are significant

Adj- R^2 = 0.8300, Pred-R^2= 0.3023
Adeq precision = 9.7448

R^2 value obtained in this study was 0.9008. The previous study stated that R^2 value at least 0.6 to be acceptable or more than 0.70 is considerably accurate and satisfactory [18-20]. The R^2 for these response variables was higher than 0.7, indicating that the regression models explained the mechanism well. The value of adj-R^2 was 0.8300. Adj-R^2 was acceptably close with R^2 shows that the model could be used as a predictor to determine optimum parameter setting precisely. "Adeq Precision" measures the signal to noise ratio, where a ratio > 4.0 is desirable. The ratio obtained in this study 9.7488 implies an acceptable signal. Thus, this model can be used to navigate the design space.

From the table, the ANOVA also showed a significant lack of fit. The lack of fit is the opposite of the whole-model test, where the tests signify whether all terms included in the model are significant. Lack of fit tests shows whether anything left out of the model is significant. Significant lack of fit indicates that the variation of the replicates about the mean values is less than the variation of the design points about the predicted values. In a simple word, the runs replicate well, and the variance is small [15,21]. The quadratic equation in Eq.2 describes the correlation between the variables and the yield of biogas produced in coded terms.

$$Y = 5.79 - 0.076A + 1.55B - 0.054AB - 0.74A^2 - 1.30B^2 \quad \text{Eq. 2}$$

In Eq.2 *Y* represent biogas yield, *A* is pH, *B* is HRT and *AB* is the interactions involved in the process.

Actual versus Predicted data

Figure 1a shows the experimental versus predicted biogas volume obtained from equation 2. Linear distribution observed indicate a well-fitting model. The values predicted from Eq. 2 were close to the observed values of biogas production. The normal probability plot presented in Figure 1b indicates that the residuals (the difference between actual and predicted value) follow a normal distribution and form an approximately straight line.

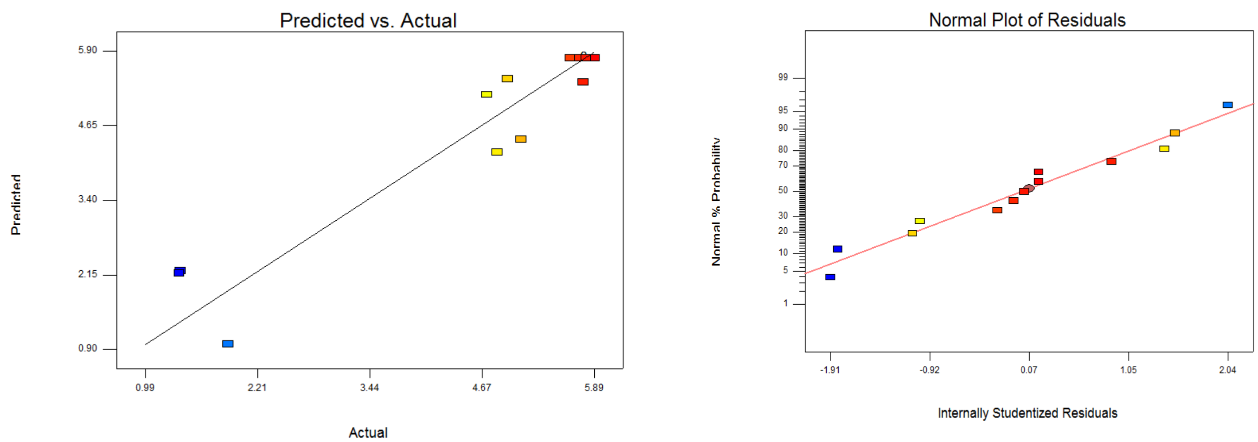


Figure 1. Correlation of actual conversion and values predicted by the model (a), and normal probability of residuals (b).

Main and Interaction Effects

Figure 2 shows the effect of two independent variables on the biogas production from sewage sludge. The HRT plays a vital role in biogas production from sludge. The production seems to affect by HRT profoundly. The biogas production improved drastically with increasing HRT; the production was low when HRT at 6.3 days and increasing until 13.65 days. However, upon increasing the HRT from 13.65

to 21.0 days, the production seemed to level off. The biogas production involved consortia of microbes that highly depending on substrates amount and process environment. Limited amount of substrate available in the process at a more extended period could halt the microbes to work efficiently. These results indicate that excessive period of HRT did not necessarily have a positive influence on biogas production.

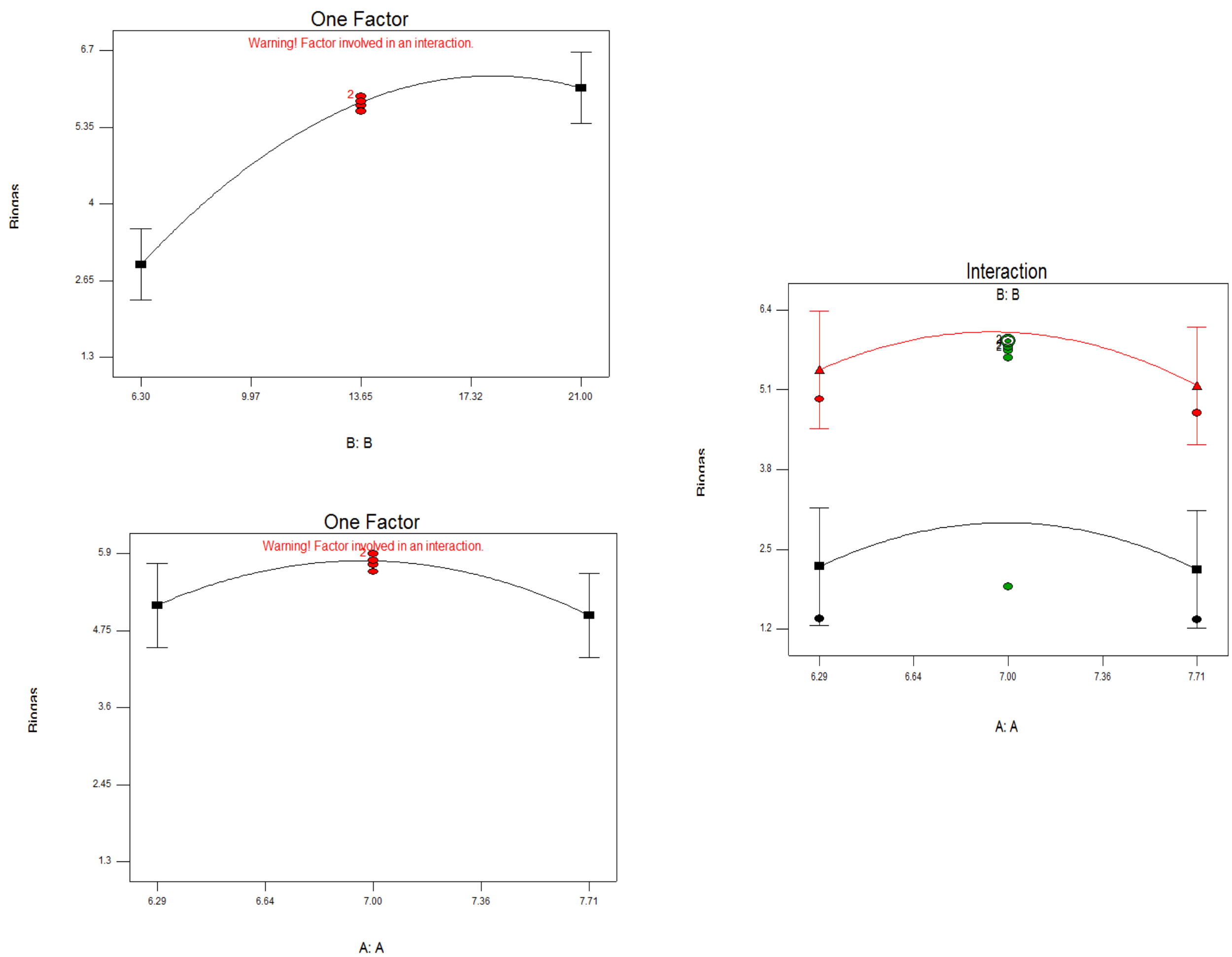


Figure 2. Effect of two individual parameters: HRT (a), pH (b). One parameter is varied while the others. are kept constant at their centre point. Interaction effect between AB (pH and HRT) on biogas production (c)

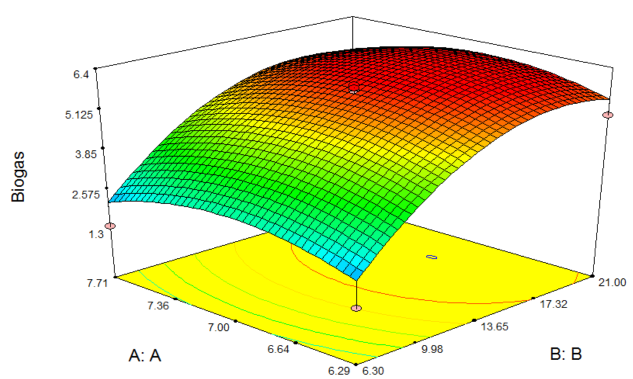
According to Alepu et al., (2016), the effect of HRT and highly depending on organic loading rate (OLR), where high OLR required longer HRT for higher yield and vice versa. Meanwhile, shorter HRT reported being a leading cause of methanogens washout and pH decline [22]. Methanogens required longer regeneration time compared to hydrolytic, acidogenic, and acetogenic bacteria. Therefore, HRT must be long enough to retain methanogens and to prevent methanogens washout from happening.

The biogas production increased when neutral pH 7.0 was applied, as shown in Figure 2b; the biogas production decrease when pH was lower or higher than 7.0. Since the main component of biogas consists of methane (50-70%) and produced by methanogens (methane producer bacteria), the neutral pH of 7.0 could be excellent for methanogens to work efficiently. According to various report, methanogens have optimum pH value around 6.8-7.2 [20, 23]. Besides, the results obtained in this study, for acidic pH was higher compared to alkaline pH. The pH lower or higher than 7.0 might decrease the methanogens efficiency in biogas production. The growth rate of methanogen hindered and greatly reduced when pH is acidic (below than 6.6) and extreme alkaline pH may lead to the disintegration of microbial granules and subsequent failure of the digestion process [20].

Figure 2 (c) shows the effect of interaction between pH and HRT depicts the effect of pH and HRT on biogas production. The effect of HRT is significant at long HRT. For example at HRT 6.3 and 21 days, the production increase from 1.3 l biogas/g COD to 1.9 l biogas/g COD and 4.7 l biogas/g COD to 5.9 l biogas/g COD, respectively. This effect has also been discovered by Shi e al., (2017) where they reported that HRT and pH play an important role in operation stability since lower pH cannot be used in biogas production when the HRT is lower than 20 days. It should be avoided at all cost since the buffer capacity available during that period is not supported for production of methane.

Response Surface Plot

The results from the comparative study showed that the biogas production was affected by HRT parameter. The effect between HRT and pH in surface and contour plot shown in Figure 3 (a) and (b). An increase in biogas production discovered when HRT increased from 6.3 to 14.0 days. However, the production tends to level off at longer HRT from 13.65 to 21.0 days. The effect is identical to all levels of pH applied in the study. For example, when pH set at 6.29, the production increased from 1.37 to 4.8 l biogas/g COD upon increasing HRT period from 6.3 to 13.65 days and, a slight improvement on yield (4.95 l biogas/g COD) when the HRT period raised to 21.0 days. A similar trend observed for biogas production with pH 7.71, where the production increased from 1.36 to 4.6 l biogas/g COD when HRT applied from 6.3 to 13.65 days. The yield 4.72l biogas/g COD tends to level off towards 21.0 days of HRT.



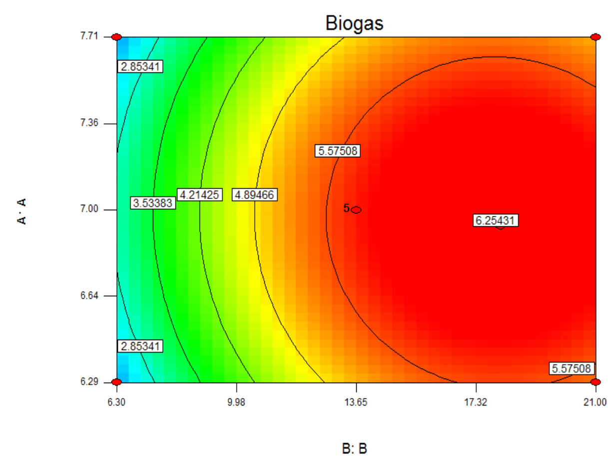


Figure 3. Response surface (a) and contour plot (b) of biogas production as a function of HRT and pH.

The AD process was greatly influenced by HRT due to the ammonia content. Mata-Alvarez et al., (2000) reported that a high concentration of ammonia more than 1000 -1500 mg/l is toxic for the microbial activity in AD. Alepu et al., (2016) added that the ammonia concentration in an efficient process could increase up to almost 700mg/L at extended HRT, with organic loading rate (OLR) set at 0.6 gCOD/(L.d).

Model Validation

The model equations adequacy to predict optimum response values was verified using the condition in Table 5 below. The parameter setting for maximum recovery applied to validate and predict the values of the responses based on the mathematical model. A close agreement exists between values calculated using the model equation and the experimental values of the response variables at the point of interest. The X^2 goodness-of-fit test applied to examine the validity of the model (Table 5). The test shows that there is no a significant difference between the predicted and actual values since the X^2 value (0.05) is much smaller than the cut-off value of X^2 for 95% confidence level for 3 degrees of freedom (7.81). It indicates that the generated model is valid at 95% confidence level.

Table 5 Validation of model equation

Run	**Variables**		**Biogas Production (l biogas/ g COD)**		**Percentage Error (%)**
	A (pH)	B (HRT (d))	Actual	Predicted	
1	7.0	10.7	5.01	4.96	1.1
2	7.0	15.7	5.85	6.12	4.7
3	6.9	15.9	5.67	6.14	8.3
4	7.0	13.7	5.7	5.91	7.8

Table 5 shows the percentage error between actual and predicted of all experiments. The errors in the table were all in acceptable range (rule of thumb of an adequate error percentage is <30%). The actual yield produced at the suggested optimum condition was 5.01 l biogas/g COD. It was not the highest yield among all four validation test, but it was the most feasible option with only 1.1% error. The production by using sludge as substrate for biogas production was in range with other reported researches [26-27]. Colon et al., (2015) also reported around 1.44 l biogas/ g COD to 6.16 l biogas/g COD produced during their study on biogas production while controlling total ammonia nitrogen content in the process. The process could generate a high yield when ammonia nitrogen content reduced at the lowest amount. The biogas production of this study was also in range with other studies performed by using different types of substrates such as food waste, agricultural waste and manure [29-32].

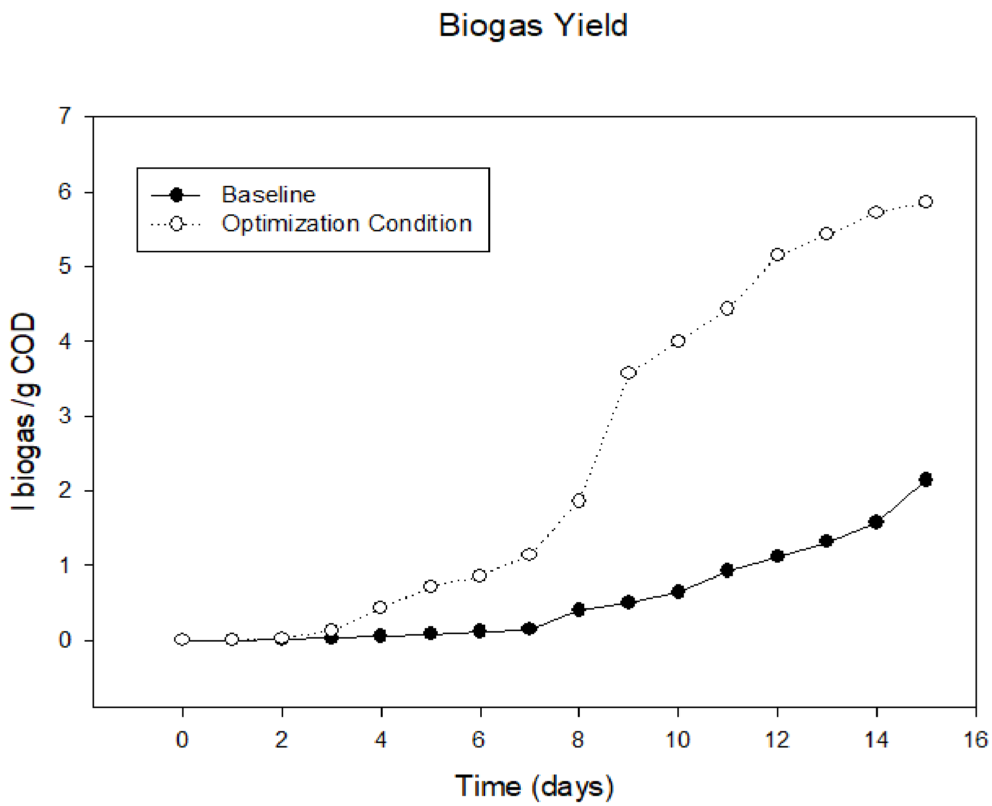


Figure 4. Biogas yield for both Baseline and Optimization Condition

The process evaluation between baseline and the optimum setting was shown in Figure 4. The result justifies that AD is highly dependent on bacteria. The high inoculum volume and feedstock recirculation would increase the bacteria amount and contact time between them and substrate [33]. Even though pH plays an essential role in the improvisation, the most significant main effect found to be HRT. The HRT provide contact time between bacteria and substrate, and the HRT length would affect microbe adaptation on the process. Extended HRT would decrease the chance of methanogen washout from the reactor, which will improve the methane yield in the process [34]. A high inoculum volume could improve the hydrolysis and acidogenesis rate [35-37], thus reducing the rate limiting step in the most biogas production. The results also proving that CCD is a useful tool for optimization and suitable for the improvement of the AD process.

Conclusion

The results corroborate the improvement and optimization of the process can be completed by the CCD method with 60% increment on biogas output. The analysis also unveils the significant impact of the interaction factor between inoculum and substrates amount in the process. Besides, the pH and HRT effect on the process depended on the substrate and inoculum amount applied. Although the optimum point was successfully acquired in this study, the feasible option can be applied in the system with lower retention time which allowing more sludge to be treated on a larger scale. The performance of the pilot-scale study substantiates the tools to be applied on the industrial scale.

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