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# Parameter Optimisation of Aerobic Granular Sludge at High Temperature Using Response Surface Methodology

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#### **ABSTRACT**

This paper proposes an improved optimisation of sequencing batch reactors (SBR) for aerobic granular sludge (AGS) at high temperature-low humidity for domestic wastewater treatment using response surface methodology (RSM). The main advantages of RSM are less number of experiment required and suitable for complex process. The sludge from a conventional activated sludge wastewater treatment plant and three sequencing batch reactors (SBRs) were fed with synthetic wastewater. The experiment were carried out at different high temperatures (30, 40 and 50°C) and the formation of AGS for simultaneous organics and nutrients removal were examined in 60 days. RSM is used to model and to optimize the biological parameters for chemical oxygen demand (COD) and total phosphorus removal in SBR system. The simulation results showed that at temperature of 45.33°C give the optimum condition for the total removal of COD and phosphorus, which correspond to performance index R<sup>2</sup> of 0.955 and 0.91, respectively.

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#### 1. INTRODUCTION

Sequencing batch reactor (SBR) is a modified activated sludge process that has been applied for numerous wastewater treatment plants such as soy sauce [1], dairy [2], industrial estate [3], textile [4] sewage, synthetic wastewater [5], palm oil mill effluent (POME) [6] and so on. This system is operated in a single reactor and it has five basic operating modes including fill, react, settle, draw and idle [7]. Compared with the costly and large-size of conventional activated sludge process, SBR is a full biological treatment, less power consumption, required no chemical addition and its operates in time rather than in space [8]. Industrial wastewater contains various aromatic hydrocarbons and inorganic substances such as chemical oxygen demand (COD), phosphorus (P), nitrogen (N) and ammonia nitrogen (NH<sub>3</sub>-N) plus it varying in pH, temperature and color. Excessive of these nutrients in aquatic water leads to severe problem of eutrophication on the water surface for lakes and oceans [9].

The development of aerobic granular sludge (AGS) in SBR (AGS-SBR) has been reported since 1990s. AGS is made up of a dense cluster of symbiotic organisms with good biological activity performance and excellent mass transfer efficiency. Usually, AGS can be cultivated using single reactor of SBR systems with airlift or bubble column reactors because nitrification, denitrification and biological phosphorus removal could carried out within the granules [9]. There is a study within lab scale that is demonstrated to identify the most vital feature that influencing the development of AGS such as organic loading rate, hydrodynamic shear

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force, settling time, and substrate composition [10]. AGS formation is a complex ecological process and it may influenced by other parameters that need to be further examined. Ordinarily the studies of aerobic granular sludge using SBR system (AGS-SBR) were performed at 20–25 °C [5] or lower [10]. Therefore, it is not suitable for the country with hot weather or climate likes Arab Saudi. It is a need of further investigation on the effect of aerobic granulation process at different high temperature (30°C, 40°C and 50°C) using SBR system.

Modeling of the nutrient removal in SBR system is vital but to develop the model using classical mathematical approach is complicated. It is because, production of AGS involved a very complex process with many influential factors. Usually, conventional optimization method was carried out by varying a single factor while keeping other factors fixed at a specific value. In this case, response surface method (RSM) is among the best technique to design the model and to find the optimum value of responses. This is due to the capability of RSM in estimating the effects of several factors and examining optimum conditions for desired responses [11]. Several studies also employed RSM in SBR for the textile dye wastewater treatment [4], palm oil mill [6], synthetic wastewater [12] and has shown good results of modeling. From the identified model, it can be seen that there were some limitation in measuring the dynamic of the biological process [13]. Besides, it will help the development of a simple model in order to control the SBR system in an efficient way.

The formation of AGS in SBR process at high temperature has been studied in [5]. Then, [13] has further investigated the AGS process using Neural Network with Gravitational search algorithm (GSA) and Inertia weight particle swarm optimization (IW-PSO) to understand the dynamic behaviour of the AGS. This paper aims to model and to determine the optimum values of the biological parameters for AGS production on SBR system at high temperature using RSM approach.

#### 2. RESEARCH METHOD

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### 2.1. Experimental Setup and Analytical Methods

Experiments were carried out in three double-walled cylindrical column bioreactor with working volume of 3L. During the start-up period, 1.5L of activated sludge from a municipal sewage treatment plant was added into the bioreactor as inoculums. Programmable logic controller (PLC) were applied to control the SBR operation (feeding pump, discharge pump and aerator pump with the setting time for each phase). The working temperature for the bioreactor were controlled at three different temperature (30, 40, and  $50 \pm 1^{\circ}\text{C}$ ) using water bath sleeves and the thermostat without controlling the oxygen and pH level. COD and phosphate analysis was performed as described in the Standard Methods for the Examination of Water and Wastewater (APHA, 2012). The data has been collected every three days in 60 days. The appropriate experimental and analytical methods can be refer at this paper [5] The total COD and Phosphorus (P) removal efficiency was calculated using Equation (1):

$$COD removal (\%) = \frac{COD_0 - COD}{COD_0}$$
 (1)

where COD<sub>0</sub> and COD are the initial concentration of municipal sewage wastewater and the concentration of permeate produced.

## 2.2. Response Surface Methodology (RSM)

The temperature and time were selected as independent factors and the total COD removal and total Phosphorus removal was chosen as the response for the optimization model. The two influents are designated at high (+1), middle (0) and low (-1) levels according to Box-Behnken Design (BBD). The ranges and levels used in the experiment were determined by preliminary experiments based on literature (see Table 1), where  $X_1$  and  $X_2$  denoted by temperature and time respectively.

Table 1. Level of variables of Box-Behnken Design design

Variables	Range and levels			
variables	-1	0	+1	
(X₁) Temperature, °C	30	40	50	
(X <sub>2</sub> ) Time, day	1	30	60	

For statistical calculation, the purpose of converted independent variables into dimensionless codified values is to let the comparison of factors of different natures with different units. Besides, it helps to reduce the error in the polynomial fit [14]. The variables  $X_i$  were coded as  $x_i$  according to the following Equation (2):

$$x_{i} = \frac{X_{i} - X_{0}}{\delta X} \tag{2}$$

where  $X_i$  is the uncoded value of the *i*th independent variable,  $X_0$  the value of  $X_i$  at the centre point of the investigated area and  $\delta X$  is the step change. The experiments were performed randomly to avoid systematic errors [15]. A second-order polynomial model by the response surface regression procedure that identified all of the possible interactions between the selected factors was as Equation (3):

$$Y = b_0 + \sum_{i=1}^k b_i X_i + \sum_{i=1}^k b_{ii} X_i^2 + \sum_{i=1}^{i < j} \sum_i b_{ij} X_i X_j \ (i = 1, 2, ..., j)$$
(3)

where Y: predicted response,  $b_0$ : constant coefficient,  $b_i$ : linear effect coefficient,  $b_{ij}$ : interaction effect coefficient,  $b_{ij}$ : quadratic effect coefficient and k: number of factors [11].

Model terms were develop and evaluated by analysis of variance (ANOVA) for each response. The analysis was evaluated using software Design Expert© version 6.0.4 State Ease inc., USA. Several responses were examined in this study and the different degree polynomial models were used for data fitting. In order to quantify the curvature effects, the data from the experimental results were fitted to higher degree polynomial equations such as two-factor interaction (2FI), quadratic model, quartic model etc. [12],[16]. Then, the higher polynomial model terms involved in the equations are those that are selected after the elimination of insignificant variables and their interaction [6],[12],[16].

## 3. RESULTS AND ANALYSIS

#### 3.1. ANOVA Analysis

Experiments were carried out using BBD method to generate a correlation between two different process variables (temperature  $(X_1)$  and time  $(X_2)$ ) and two responses (% of COD removal and % of phosphorus removal). There are 13 runs of experiments were conducted and the responses obtained were specified in Table 2. The response of experimental results shows that the percentage of COD removal was in the range of 41% to 98.17%, while the percentage of Phosphorus removal was in between 15% and 72%. From Table 2, the predicted responses followed the experimental responses well.

Table 2. Experimental and predicted values of total COD and phosphorus in SBR syste	Table 2. Experimental and	predicted values	of total COD and	phosphorus in	SBR system
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	Variables			Responses (%)			
Run	Temperature	Time	Total COI	O removal	Total Phosph	orus removal	
	$X_1$ (°C)	$X_2$ (day)	Expt.	Pred.	Expt.	Pred.	
1	50	1	41.000	37.08	15.000	19.94	
2	50	15	56.000	57.34	41.610	33.45	
3	50	45	91.000	89.23	65.000	62.411	
4	50	60	98.171	99.28	72.460	76.88	
5	40	3	47.500	53.65	30.000	36.04	
6	40	18	71.795	70.01	38.762	45.87	
7	40	39	89.744	86.32	63.790	59.64	
8	40	57	95.122	94.18	66.250	71.44	
9	30	1	62.500	60.51	53.500	49.52	
10	30	21	76.154	75.91	55.714	56.44	
11	30	45	83.415	85.17	70.000	64.74	
12	30	60	85.366	85.85	68.500	69.93	
13	50	30	72.000	75.25	53.640	47.93	

The results were analyzed using Analysis of Variance (ANOVA) and summarized in Table 3. The analysis shows the quadratic regression model for total removal of COD, while total phosphorus removal was two-factor interaction regression model (Equations 4 and 5). This two regression models indicated the model are highly significant due to the P value (Prob>F) was very low (<0.0001). It is because, in order for the model to be significant at 95% confidence level, the "Prob>F" must less than 0.05 [6]. The smaller the magnitude of the P value, the more significant was the corresponding coefficient. Therefore, it can be seen that the significant terms for COD removal are  $X_2$ ,  $X_2^2$  and  $X_1X_2$ , whereas  $X_1$  and  $X_1^2$  are trivial for COD removal. While, all terms used for the total phosphorus ( $X_1$ ,  $X_2$  and  $X_1X_2$ ) are significant. This P values were used as a tool to confirm that the quadratic or two-factor interaction model was statistically significant and sufficient to explain the actual relationship between the response and the significant variables [17]. However, model F-value was calculated as a ratio of mean square regression and mean square residual. The F-value and the corresponding P values were given in Table 3.

The square of correlation coefficient for each response was compute as a coefficient of determination,  $R^2$  and the value is always between 0 and 1 [12], The value of  $R^2 > 0.90$  indicates that the model is excellent. According to S. Sathian et al.[4], a good statistical model should have  $R^2$  value closes to 1.0. In this case, the predicted  $R^2$  value for total removal of COD and phosphorus responses was in reasonable agreement since the fit of the model was found to be 0.977 and 0.91 respectively. It concludes that the prediction model obtained using BBD technique of RSM approach can relatively reflect the effects of temperature and time on the performance of SBR system on the examination of synthetic wastewater treatment based on nutrient removal.

Table 3. ANOVA analysis for total COD and phosphorus removal

			1 1	
Source	Total COD removal		Total I	P removal.
	F Value	Prob > F	F Value	Prob > F
Model	59.47	< 0.0001	30.52	< 0.0001
X <sub>1</sub> -temperature	4.15	0.0811	7.84	0.0207
X <sub>2</sub> -time	244.46	< 0.0001	69.85	< 0.0001
$X_1^2$	1.16	0.3168		
$X_{2}^{^{1}2}$	8.97	0.0201		
$X_1X_2$	31.04	0.0008	11.14	0.0087
Sum of squares	3948.46		3328.14	
Mean squares	789.69	789.69 1109.38		
Std. Dev.	3.64		6.03	
Mean	74.60		53.40	
$\mathbb{R}^2$	0.9770		0.9105	

In order to investigate the effect of high temperature on the formation of AGS in SBR system and overall performance of biological treatment, total COD and phosphorus removal efficiency was calculated as a response. Figure 1 (a) and (b) demonstrates a very good conversion between the experimental and predicted values of two responses (COD and phosphorus), with removal efficiency 0.977 and 0.91 respectively. Actual values are the measured response data for a particular run and the predicted values were evaluated from the model and generated by using the approximating function. It shows that the predicted values of the response from the model agreed with the observed values and could be properly applied to navigate the design space [18]. By applying multiple regression analysis on the experimental data, both responses were fitted and produce quadratic and two-factor interaction models. The empirical models in terms of coded factors for the quadratic regression equation of COD removal were stated as Equation (4), while phosphorus removal response was present as two-factor interaction model (Equation (5)):

Total COD removal (%) = 
$$80.65 - 2.50X_1 + 21.88X_2 - 2.37X_1^2 - 7.59X_2^2 + 9.22X_1X_2$$
 (4)

Total phosphorus removal (%) = 
$$54.07 - 5.65X_1 + 19.34X_2 + 9.13X_1X_2$$
 (5)

where  $X_1$  and  $X_2$  were the coded values of the test variables temperature (°C) and time (day), respectively. The positive sign and negative sign represents the synergistic effect and antagonistic effects between mutual interaction and individual parameters respectively [17].

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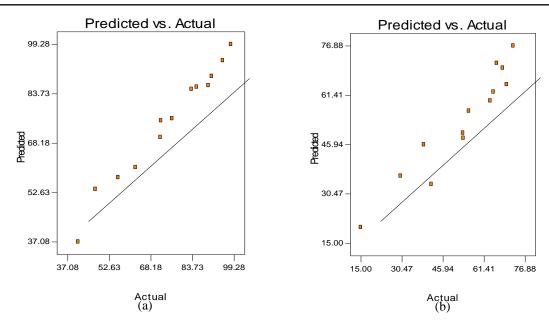


Figure 1. Predicted vs. actual values plot for COD removal (a) and phosphorus removal (b).

## 3.2. Effect of variables on treatment of synthetic wastewater in SBR system

The response surface and contour plots were generated for different interactions of any two independent variables, while holding the value of the other variables as a constant [4]. The optimization of process variables was aimed at finding the levels of independent variables, which would give maximum percentage of total removal for COD and phosphorus removal. The response surface plots and the corresponding contour curves described by the regression equation are shown in Figure 2 and 3, (a) and (b). Figure 2 (a) shows the interaction between time and temperature on percentage of total COD. The observation reveals that the percentage of total COD removal increased when the time was increase at temperature increase. The maximum total COD removal (86.33%) was observed at temperature (40°C) and operational time of 39 days. After that, under constant operational time, a slight increase in total COD removal was observed for higher temperature (>40°C). This has been supported by M.H. Ab Halim et al.[5], where high temperature was lead for the high ash content in biogranules and accordingly increase the size. The results can be more clearly revealed by referring to Table 3, where the F-value of time (244.46) was higher than the temperature (4.15), suggesting that the operational time had more impact on removing COD. Besides that, the significant as P-value less than 0.05 were found in linear term of time (X<sub>2</sub>), square term  $(X_2^2)$  of time and quadratic term  $(X_1X_2)$  of temperature and time. These results demonstrate that temperature and time were required by COD removal to maximize AGS formation in SBR system.

There is a similar trend of 3D graph for the total COD and phosphorus removal (Figure 2 (a) and Figure 3 (a)), where the percentage of total phosphorus removal increased when the time was increase at temperature increase. Then, the operational time was highly significant on the phosphorus removal compare to time. However, the phosphorus removal was increases gradually with time when the temperature was increase. The same trend were archived from the previous study reported by M.H. Ab Halim et al.[5], when the average removal rate for 50°C is the highest, followed by 30°C and 40°C. In addition, the percentage of COD removal was seen to be higher than phosphorus removal. These was due to acetate (COD) was utilized to 34.5% at 30°C, 51.22% at 40°C and 38.79% at 50°C, after that, the rest of acetate was utilized in the period of aerobic cycle while phosphate was removed. As illustrated in ANOVA analysis, the mutual interaction between both parameter was highly significant, it was observed by a very small "Prob>F" value (0.0087). Thus, it may conclude that the interaction of temperature and time were required in removing the total COD and phosphorus in SBR system. Besides that, it shows that temperature is not a critical factor in the formation of AGS in SBR, however it was able to induce the morphology and settling ability of granules [19]. Although, this experimental results was differ with the statement made by Song Zhiwei et al. [19], which declared that the bioactivity of microbes will become worse and could also influence the biological reaction efficiently when the temperature exceed 30°C.

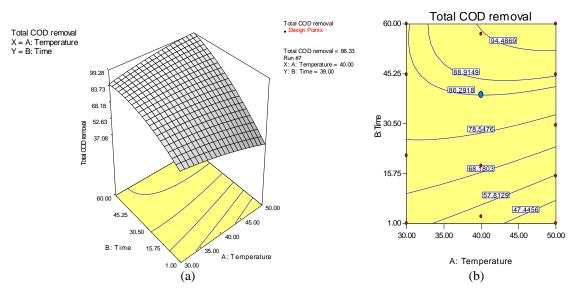


Figure 2. Graph depicting the response surface plot (a) and contour plot (b) showing the effect of temperature  $(X_1)$  and time  $(X_2)$  and their mutual interaction of total COD removal in SBR system

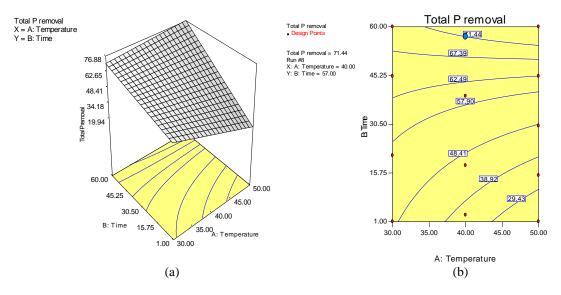


Figure 3. Graph depicting the response surface plot (a) and contour plot (b) showing the effect of temperature  $(X_1)$  and time  $(X_2)$  and their mutual interaction of total phosphorus removal in SBR system

The experimental condition were proposed to find the optimum point in order to obtaining the highest conversion on maximize the total removal condition. The result of the proposed condition is shown at Table 4. The optimize condition values suggested by RSM approach were 45.33°C at 60 days, which corresponded to the actual experimental values of 97.84% and 75.26% for total removal of COD and phosphorus respectively. The experimental values were relatively close to the predicted values by the model and were therefore confirming the adequacy and validity of the predicted model.

Table 4. Optimized value of the process

		• [- 1		F	
Solution Temperature (°C)		Time	Total COD	Total P removal	Desirability
polation reinperature (C)	(day)	removal (%)	(%)	Desiraomity	
1	45.33	60.00	97.8422	75.2578	0.998

#### 4. CONCLUSION

The response surface methodology for parameter optimisation of aerobic granular sludge SBR has been presented. The simulation results showed that RSM can predict the aerobic granular sludge parameters well at different temperature condition (30, 40 and 50  $^{0}$ C). The result also showed that the high temperature influenced the morphology and settling ability of granules, although less significant in biological removal. Besides that, with only a few number of experiments, RSM can developed an excellent COD removal efficiency. This study demonstrates that RSM approach is suitable for predicting the high condition of AGS in SBR system based on the percentage of total removal of COD and phosphorus. Therefore, the model developed in this work can be useful for predicting the formation of AGS in SBR system at hot climate.

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