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## Application of experimental design approach for investigating the short-term effect of operative parameters on performance of SBR

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### ABSTRACT

In this study, the short-time effects of operative parameters including influent COD, COD/N ratio, COD/P ratio, aeration time, settling time, and temperature on performance of a sequencing batch reactor (SBR) were investigated, which was applied for starchy wastewater treatment. For COD removal (%),  $R^2$  is 0.8250, implying that 82.50% of response variability is achieved by a regression model. According to the regression model that obtained for COD removal (%), the maximum COD removal (%) was 78.33% that was obtained at the COD of 400 mg·L<sup>-1</sup>, COD/N ratio of 5, COD/P ratio of 100, settling time of 90 min, and temperature of 22 °C.

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### KEYWORDS

SBR;  
Starch;  
Aerobic;  
Biological;  
Fractional factorial.

### INTRODUCTION

Starch production plants are considered as high polluting industries. The wastewater originated from starch industries are characterized by high chemical oxygen demand (COD) content, ranging from 2000 to 24000 mg·L<sup>-1</sup> as well as high total suspended solid (TSS)<sup>[1-4]</sup>. Washing the feed stocks, starch extraction process, and cleaning the pipelines and instruments are the main resources of wastewater in starch industries<sup>[5]</sup>.

As yet, many methods have been suggested for improving the efficiency of starch wastewater treatment systems. Furthermore, enforcement of stringent discharge standards stimulates the introduction of effective wastewater treatment systems. There are several studies that investigated the ability of anaerobic biological treatment such as upflow anaerobic sludge blanket reactor (UASB)<sup>[6]</sup>, anaerobic baffled reactor (ABR)<sup>[3,7]</sup>, hori-

zontal flow filter (HFF)<sup>[8]</sup>, anaerobic tapered fluidized bed reactor (ATFBR)<sup>[4,9]</sup>, and anaerobic pond<sup>[10]</sup>. Rajasimman et al. studied the treatability of cassava starch wastewater using aerobic fluidized bed bioreactor (FBB)<sup>[11]</sup>. Besides, in a few works, ability of membrane technology in treating starchy wastewater was investigated<sup>[12-14]</sup>.

Activated sludge systems fall within aerobic biological systems. A conventional activated sludge has several disadvantages such as relatively high energy consumption, high biomass production, high operation costs and problems associated to the disposal of large amount of sludge<sup>[15]</sup>. Wastewater treatment with sequencing batch reactor (SBR) is an alternative solution, compared to the activated sludge process. SBRs offer various advantages, including minimal space requirements and ease of management<sup>[16]</sup>. Furthermore, SBRs and modified SBRs have been successfully applied to treat

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complex chemical wastewaters<sup>[17-19]</sup>, and also they are able to remove nitrogen compounds<sup>[20,21]</sup>.

SBR cycle consists of several phases (filling, aeration, settling, decanting and idle); but for nitrogen and phosphorous removal, conducting anaerobic-aerobic condition (with anoxic and aeration phases in each cycle) is necessary.

In this study, the SBR system is applied to treat synthetic starchy wastewater. Thus far, the short-time effects of operative parameters including influent COD, COD/N ratio, COD/P ratio, aeration time, settling time, and temperature on performance of a SBR in treating starchy wastewater have not been reported in literatures. This paper aims to fill this knowledge gap in the SBR process.

### THEORY

The output variables depend not only on input parameters. In many cases, interactions between the input parameters may severely affect the output ones. So these possible interactions must be taken into account. Varying one input parameter and keeping the other ones constant, is the conventional procedure to study the effects of operative parameters. But using this method, the interactions between different factors are overlooked, leading to a misinterpretation of the results<sup>[22,23]</sup>. The performance of a SBR in treating starchy wastewater is a complex and sensitive function of numerous parameters. Applying a systematic approach to study the effects of these parameters on the performance of system and obtaining the optimum condition seems necessary. Design of experiment (DoE) technique can assess the interactions that could not be considered by varying one parameter and keeping others constant. In recent years, DoE has been used in several fields of science and engineering due to its high ability in making design layout of experiments<sup>[24-33]</sup>. Besides, decreasing the number of experiments in studying a phenomenon is of interest. DoE provides a powerful tool that prevents doing unnecessary experiments as well as considering the interactions between factors. DoE consists of several subsets. One of the methods used in this study is fractional factorial design (FFD). FFD is a statistical method for modelling the experimental data as well as screening the insignificant parameters. In this study, Design Expert<sup>1</sup> software was

used to develop the experimental plan layout and analyzing the obtained data.

**TABLE 1 : Operative parameters and levels for foldover fractional factorial design**

Operative parameters	levels		
	-1	0	1
(A) COD (mg L <sup>-1</sup> )	200	300	400
(B) COD/N ratio	5	12.5	20
(C) COD/P ratio	20	60	100
(D) Aeration time (min)	90	180	270
(E) Settling time (min)	10	15	20
(F) Temperature (°C)	22	27	32

**TABLE 2 : Design layout of 2<sub>III</sub><sup>6-3</sup> fractional factorial design**

Run	Block	Operative parameters					
		A	B	C	D	E	F
1	1	200.00	5.00	100.00	270.00	10.00	22.00
2	1	200.00	20.00	100.00	90.00	10.00	32.00
3	1	300.00	12.50	60.00	180.00	15.00	27.00
4	1	400.00	5.00	100.00	90.00	20.00	22.00
5	1	400.00	20.00	20.00	270.00	10.00	22.00
6	1	300.00	12.50	60.00	180.00	15.00	27.00
7	1	200.00	5.00	20.00	270.00	20.00	32.00
8	1	400.00	20.00	100.00	270.00	20.00	32.00
9	1	200.00	20.00	20.00	90.00	20.00	22.00
10	1	400.00	5.00	20.00	90.00	10.00	32.00

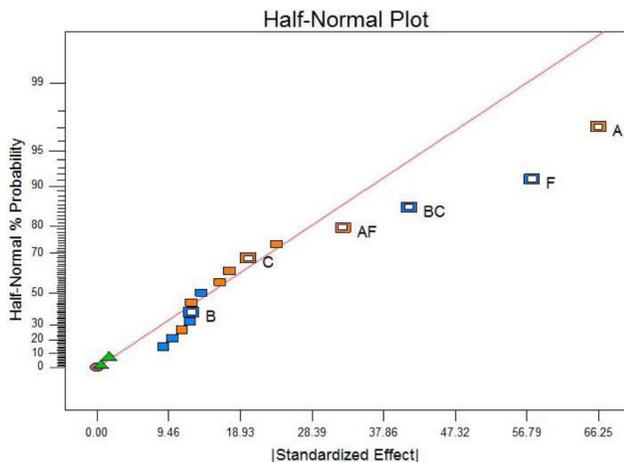
**TABLE 3 : Design layout of 2<sub>III</sub><sup>6-3</sup> foldover fractional factorial design.**

Run	Block	Operative parameters					
		A	B	C	D	E	F
11	2	200.00	20.00	100.00	270.00	20.00	22.00
12	2	200.00	5.00	20.00	90.00	10.00	22.00
13	2	300.00	12.50	60.00	180.00	15.00	27.00
14	2	200.00	20.00	20.00	270.00	10.00	32.00
15	2	400.00	20.00	100.00	90.00	10.00	22.00
16	2	400.00	5.00	20.00	270.00	20.00	22.00
17	2	300.00	12.50	60.00	180.00	15.00	27.00
18	2	400.00	20.00	20.00	90.00	20.00	32.00
19	2	400.00	5.00	100.00	270.00	10.00	32.00
20	2	200.00	5.00	100.00	90.00	20.00	32.00

### MATERIALS AND METHODS

The wastewater used throughout the study was a

synthetic starch effluent. The wheat starch powder was dissolved in tap water to attain the required COD of feed, COD concentration was in the range 200–400 mg·L<sup>-1</sup> and was analyzed following the standard methods<sup>[34]</sup>. The air was introduced by an aerator in the bottom of the reactor. To prepare the seed sludge a cow-dung has been used as seed culture and sludge was developed within 50 days in SBR by a synthetic starchy wastewater. The SVI was determined by reading the volume of the settled sludge in a 1 liter container after 30 min settling (after the aeration phase and before settling and effluent withdrawal phase) and calculated from the settled sludge volume and the total suspended solids. A cubic reactors with dimensions of 30×30×60 cm and working volume of 45 l, was operated in sequencing batch mode. Effluent was drawn at 20, 30, and 40 cm from the bottom, so 16 l was left in the reactor after effluent withdrawal. Influent adding was done from the top of the reactors. Aeration is off during feeding, effluent withdrawal, and settling phases.



**Figure 1 : A half-normal probability plot for COD removal (%)**

The operative parameters and their values at the two levels (high, +1 and low, -1 levels) set in the design have been shown in TABLE 1. Also, the center points (level 0) were set along the design layout to check the curvature and experimental error. These points are located just in the midpoint of high and low levels.

The tests configurations and experimental results of two-level FFD, consisting of 8 experiments as well as two center points, are summarized in TABLE 2.

The number of experiments for factorial design with 7 operative parameters is 2<sup>6</sup>. So, the current design

layout is actually a 1/8th fraction of factorial design. It is obvious that every effect is aliased with 7 other ones, which may result in misinterpreting the effects of operating parameters and their relationship. The aliases structures for 2<sup>6</sup><sub>III</sub>-<sup>3</sup> design are as below<sup>[35]</sup>:

$$[A] = A + BD + CE$$

$$[B] = B + AD + CF$$

$$[C] = C + AE + BF$$

$$[D] = D + AB + EF$$

$$[E] = E + AC + DF$$

$$[F] = F + BC + DE$$

$$[AF] = AF + BE + CD$$

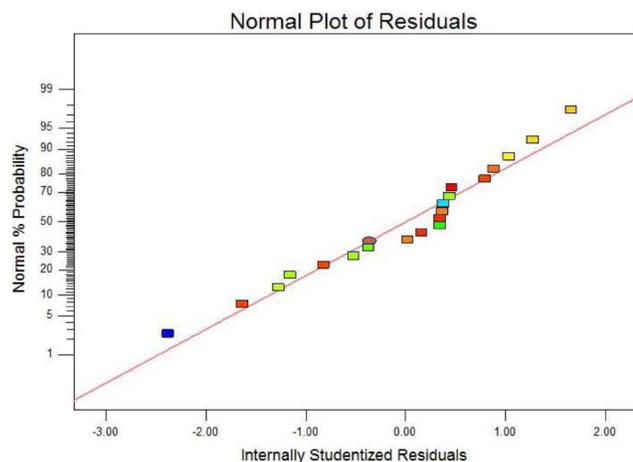
It is important to note that three and higher factor interactions were ignored to avoid unnecessary screen clutter. It is found that main effects are confounded with plausible two factor interactions. For the situation that every two factor interaction has unimportant effect on the output parameters, this aliasing is negligible. But if it does not, de-aliasing main effect and two factor interactions seems necessary. In other words, running several more experiments via foldover design would clear up the confounding. The foldover design layout was presented in TABLE 3.

## RESULT AND DISCUSSION

### Half-normal probability plot

Figure 1 shows Half-normal probability plot of the effects, a diagram in which the absolute value of every effect was plotted along the X-axis while the probability percent was plotted along the Y-axis. Half-normal probability plot is a useful tool to determine the significance of the main and the interaction effects. The largest effect is located at the right side of diagram. Every effect that lies along the line is insignificant, but significant ones are pretty far from the line. Hence, the main effects including COD (A), temperature (F), AF and BC as the interaction effect significantly influenced the COD removal percentage. As is shown in Figure 3 (a), the main effects of COD/N (B) and COD/P (C) are not significant, but to present a hierarchic model; these parameters are included in the model. Non-hierarchically models are incorrect<sup>[36]</sup>. Figure 2 shows Pareto chart of the selected effects. Pareto chart is a useful graphical tool for showing the relative size of effects. In analyzing

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**Figure 3 : Normal probability plot of residual for COD removal (%)**

this chart, the effects would be divided into three categories: (I) significant effects (above Bonferroni limit), (II) possibly significant effects (above t-value limit and below Bonferroni limit), and (III) insignificant effects (below t-value limit). The results of Pareto chart are similar to half-normal probability plot ones.

### ANOVA analysis

Design Expert software makes data analysis in coded form. Operating parameters are coded by the following equation<sup>[36]</sup>:

$$C_i = \frac{x_i - \frac{x_{i,high} + x_{i,low}}{2}}{\frac{x_{i,high} - x_{i,low}}{2}} \quad (1)$$

where  $C_i$  is the coded value of operative parameter  $x_i$ ,  $x_{i,high}$  and  $x_{i,low}$  are the values of this parameter at high and low levels, respectively.

The accuracy of model was measured using correlation coefficient index ( $R^2$ ), which is defined as follows:

$$R^2 = 1 - \frac{\sum (y_{exp.} - y_{pred.})^2}{\sum (y_{exp.} - \bar{y})^2} \quad (2)$$

$$\bar{y} = \frac{\sum y_{exp.}}{N} \quad (3)$$

Where  $y_{exp.}$  and  $y_{pred.}$  are experimental and predicted values, respectively, and  $N$  is the number of data. The normal probability plot of the studentized residuals is shown in Figure 3. The normal probability plot indicates whether the residuals follow a normal distribution or not. The points that follow a straight line confirm that

errors were normally distributed with a mean of zero. Definite patterns like an “S-shaped” curve indicate that a transformation of response may be needed.

The ANOVA table for assessing the COD removal (%) is shown in TABLE 4.

**TABLE 4 : ANOVA table for COD removal (%)**

Source	Sum of squares	df	Mean square	F value	Prob>F	
Block	8820.00	1	8820.00			
Model	44037.50	6	7339.58	9.43	0.0006	Significant
Curvature	3.47	1	3.47	4.091E-003	0.9501	Not significant
Residual	9339.72	12	778.31			
Lack of Fit	7739.72	10	773.97	0.97	0.6092	Not significant
Pure error	1600.00	2	800.00			
Cor. total	62197.22	19		R-squared	0.8250	
				Adj. R-squared	0.7375	
				Adeq. precision	11.448	

Values of ( $Prob > F$ ) less than 0.05 indicate the model terms are significant and values greater than 0.10 indicate they are insignificant. So, according to the ANOVA table for COD removal (%), the model is significant. Adequate precision of 11.448 indicates an adequate signal. The Model F-value of 9.43 implies that the model is significant. There is only a 0.06% chance that a Model F-Value this large could occur as a result of noise. The “lack of fit F-value” of 0.97 implies that the lack of fit is insignificant relative to pure error. Pure error is a measure of error related to repeatability. This term is the sum of squares of the repeated observations divided by the degree of freedom. There is a 60.92% chance that a “Lack of Fit F-value” this large could occur due to noise. Non-significant lack of fit is good. The integrity of a model can be checked by the determination of  $R^2$  coefficient and adjusted  $R^2$ <sup>[22]</sup>. In this study, for COD removal (%),  $R^2$  is 0.8250, implying that 82.50% of response variability is achieved by a regression model.  $R^2$  increases as the number of terms increases in the model, while no improvement in model is observed<sup>[37]</sup>. So to face this problem, adjusted  $R^2$  was introduced, which would increase only and only due to the model improvement. In this case adjusted  $R^2$  is 0.7375 reasonably in accords with  $R^2$  that means that this value of  $R^2$  is not unreal. The final equation for permeate flux in terms of coded factors obtained from regression of values is as below:

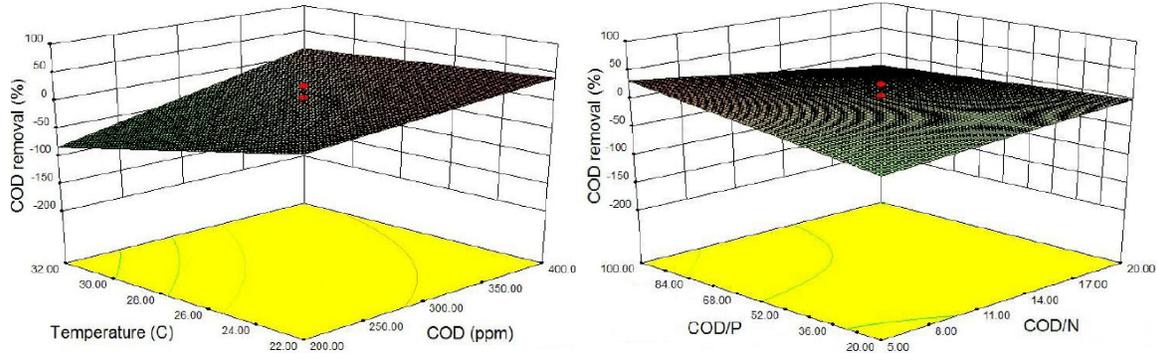


Figure 4 : 3-D diagram of COD removal (%) vs. significant operative parameters

TABLE 5 : The optimum condition

COD (mgL <sup>-1</sup> )	COD/N ratio	COD/P ratio	Aeration time (min)	Settling time (min)	Temperature (°C)	COD removal (%)	Desirability
400.00	5.00	100.00	90.00	20.00	22.00	78.33	1.000

$$\text{COD removal\%} = -4.17 + 33.13 \times A - 6.25 \times B + 10.00 \times C - 28.75 \times F + 16.25 \times A \times F - 20.62 \times B \times C \quad (4)$$

The final equation for COD removal (%) in terms of coded parameters obtained from regression of values. But, expression of COD removal (%) in actual values is preferred. So, by converting the coded values to actual ones using equation 1, the following equation in terms of actual value achieved:

$$\text{COD removal\%} = 258.81250 - 0.54625 \times \text{COD} + 3.29167 \times \text{COD/N} + 1.10937 \times \text{COD/p} - 15.50000 \times \text{Temperature} + 0.032500 \times \text{COD} \times \text{Temperature} - 0.068750 \times \text{COD/N} \times \text{COD/P} \quad (5)$$

Subjected to: 200 < COD < 400 (mgL<sup>-1</sup>), 5 < COD/N < 20, 20 < COD/P < 100 and 90 < Aeration time < 270 (min), 10 < settling time < 20 (min), 22 < Temperature < 32 (°C). This model can be used to predict the COD Removal (%) within the limits of operative parameters. As mentioned earlier, finding the optimum condition is so important because by running the reactor on the basis of optimum condition the performance of treatment process will be at the maximum value.

The Design Expert software finds optimum condition. The optimum condition for this process is presented in TABLE 5.

### Data analysis

The related diagram obtained from equation 5 is illustrated in Figure 4. In Figure 5, a comparison between filtrate and non-filtrate COD has been made. Figure 6 shows a comparison between experimental data and the data predicted by regression model for COD removal (%). It is found from the ANOVA table

that the aeration time and settling time have no considerable effect on reactor performance in treating the starchy wastewater. According to Figure 5, non-filtrate COD removal (%) showed large variations during the experimentation and on several runs reached negative values. The negative COD removal efficiency means that the effluent COD concentration is higher than that of the influent one. The negative COD removal (%) may be due to improper setting properties or a sort of “memory”

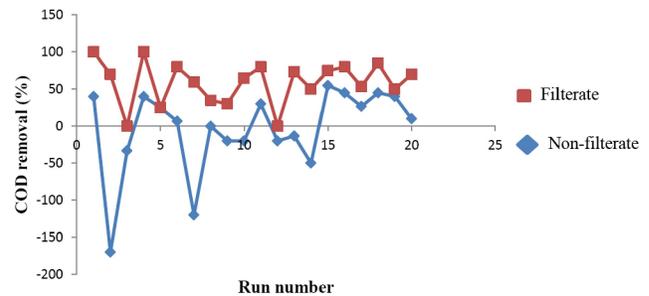


Figure 5 : Comparison between filtrate and non-filtrate COD removal (%)

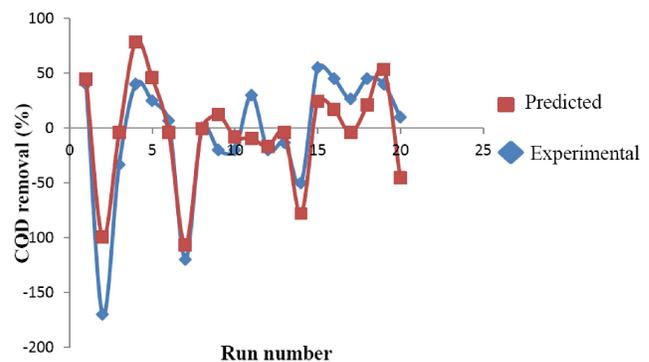


Figure 6 : Experimental data compared to predicted values given by the regression model for permeate flux

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in the reactor<sup>[38]</sup>. Figure 7 shows the non-filtrate COD removal (%) as well as corresponded sludge volume index (SVI). The COD/N ratio greater than 90 leads to excessive growth of filamentous, which damage settling properties<sup>[39]</sup>. SVI more than 150 mL·g<sup>-1</sup> is related to the filamentous growth<sup>[40]</sup> while the maximum COD/N ratio was 20 in this work. In current study, except run 1 and 18, the SVI was below 100 mg·L<sup>-1</sup>, which is acceptable for SBR<sup>[41]</sup>. So, the reason of emerging negative COD removal (%) is that when the influent COD had a sudden decrease, the effluent concentration was still influenced by the liquid present in the reactor due to high hydraulic retention time (HRT).

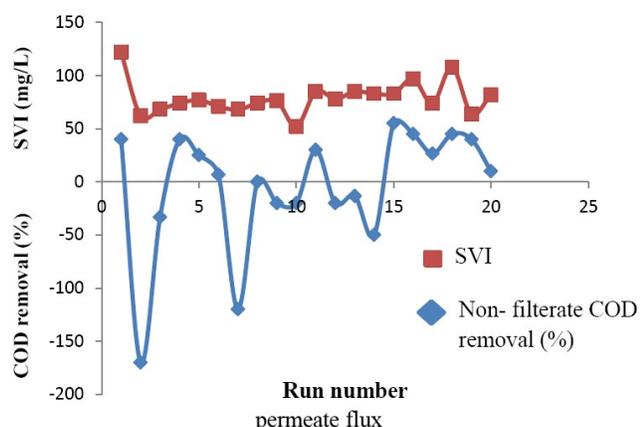


Figure 7 : Non-filtrate COD removal (%) and SVI vs. run number

## CONCLUSIONS

The effects of operative parameters on performance of a sequencing batch reactor (SBR) were investigated, which was applied for starchy wastewater treatment. The performance of reactor was evaluated using COD removal (%). For COD removal (%), correlation coefficient index ( $R^2$ ) is 0.8250, implying that 82.50% of response variability is achieved by a regression model. It is found from the ANOVA table that the aeration time and settling time have no considerable effect on reactor performance in treating the starchy wastewater. In current study, except run 1 and 18, the SVI was below 100 mg·L<sup>-1</sup>, which is acceptable for SBR. According to the regression model that obtained for COD removal (%), the maximum COD removal (%) was 78.33% that would be obtained at the COD of 400 mg·L<sup>-1</sup>, COD/N ratio of 5, COD/P ratio of 100, settling time of 90 min,

and temperature of 22 °C.

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