Introducing an applied reactor for treatment of wastewater containing propylene glycol

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Abstract

Propylene glycol (PG), classed as alcohol, has been used in many industrial additives. Leaching PG in aquatic environments would cause a significant decrease in dissolved oxygen, and this is the main reason for treatment of runoffs and wastewater contaminated with this pollutant. Previous researches on PG removal from wastewater indicate that biological methods are more economically suitable. Therefore, a new biological reactor was introduced and used for the treatment of wastewater containing PG. Two main approaches for increasing removal efficiency in presented research are utilizing two serial lab-scale Sequencing Batch Reactors and feed backward connection between these reactors. This novel reactor was named Feed Backward Serial Sequencing Batch Reactor. Moreover, response surface method was used for modeling of PG treatment and investigation of interactions and simultaneous effects of independent parameters. Retention time, influent COD, and flow recirculation percentage were considered as independent variables, where COD removal efficiency was the dependent variable. According to the results, the best COD removal efficiency was 47%, and it was achieved in 3.52 h retention time, 1667.76 mg/L influent COD, and 23.33% flow recirculation percentage. Based on the results of the presented research, PG treatment with the presented reactor is feasible.

Keywords Biological reactors · FBSSBR · Propylene glycol · Sequencing batch reactor

1 Introduction

In the contemporary world, different environmental problems have been caused as a result of constant development of additives and substances, most of which were supposed to enhance the quality of life [2, 4, 54]. Propylene glycol (PG) is one of these chemicals which is used in various industries including cosmetics, detergents, pesticides, foods, pharmaceutical, and most importantly aircraft deicing fluid, in tremendous amounts [53]. PG can affect organisms and the environment through penetrating groundwater [3] and leakage in surface water. PG significantly impacts marine life by reducing the dissolved oxygen [51]. Also it threatens human health with renal

inadequacy and hepatic debilitation [55]. Based on these risks, PG treatment in wastewater seems to be necessary.

There are series of ways for industrial wastewater treatment such as physical, chemical and biological processes [6, 12, 13, 19, 24, 25, 41, 42]. Studying about nano materials and using nano fluids is one of new research interests [33–40, 43–45]. Many ways are being used for PG contaminated wastewater treatment. Take, for instance, physical and chemical processes. The conclusion of these studies was that high cost is associated with chemical processes, and they produce secondary pollutants [30, 5]. Also, physical treatment methods showed low efficiency, and it only changed the ambiance of contamination [10, 14]. PG degradation with the Fenton process and FeSO_4 , H_2O_2 [51],

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and propylene glycol phenyl ether adsorption by activated carbon [52] are some physical and chemical procedures.

On the other hand, biological methods are immensely effective at a lower cost. Research on biological PG treatment has shown promising results. For instance, biological treatment of PG wastewater with methanogen bacteria in a semi-continuous reactor at a 35 °C temperature reached 95% of COD removal (Sezgin and Tonuk [31]). In another research, activated sludge achieved 85% PG removal [48]. Ethylene glycol treatment from paint industries wastewater in a continuous biological system showed 97% removal efficiency [18].

However, bio-treatment is not flawless either. In the past century, activated sludge has been widely used in all kinds of wastewater treatment; this method requires a great deal of energy, biomass byproduct, operation cost, and sludge management cost. Many alternatives have been recommended because of technological achievements in the past decades; one such alternative is the sequence batch reactor (SBR) system [20, 46].

Several studies on complicated compound degradation have underscored the importance of ambience conditions in SBR systems. These studies include research on hydraulic retention time (HRT) study on synthetic petroleum wastewater [32], and olive oil wastewater treatment with SBR [11].

Prediction is crucial in environmental engineering and was studied in previous researches [7, 8, 21, 26–29, 47]. In past studies, modeling, multivariate optimization, simultaneous effects of independent parameters, and synergistic and antagonistic effects of variables in biological treatment of contaminations were not considered. In this research, SBR was upgraded, and a new biological reactor called Feed Backward Serial Sequencing Batch Reactor (FBSSBR) was introduced. Application of FBSSBR in PG wastewater treatment was evaluated, for the first time. Modeling, multivariate optimization, simultaneous effects of independent parameters, and synergistic and antagonistic effects of variables in biological treatment of PG wastewater were done based on response surface methodology (RSM).

2 Materials and methods

2.1 Reactor design

According to Figs. 1 and 2, the FBSSBR reactor system had two similar reactors with 10 cm diameter, a height of 35 cm, and volume of 2.75 L each. Furthermore, the indicator was placed 30 cm from the bottom. Accordingly, the effective height was 30 cm, and effective

Fig. 1 3D scheme of feed backward serial sequencing batch reactor (A: reactor 1, B: reactor 2, C: feed container, D: aeration pump, E&F: cycling pump, H&G: sampling valve)

volume was 2.335 L. The connection between two reactors for having an FBSSBR was with regular PVC tubes.

Each time the initial concentration of PG in wastewater was changed, the following steps were followed:

- At first, both reactors were full of wastewater, and then aeration units were turned on until the end of the retention time. Then aeration was stopped, and each reactor had 30 min time for settling time. Afterward, reactor two was emptied and used for concentration measurement, and reactor two was filled with the content of reactor 1. Finally, reactor one was filled with raw wastewater from the feed container.
- Secondly, the two reactors were again aerated during their retention time, and in this stage, the wastewater was in circulation between two reactors through the connection pipes. So that, during the retention time, a certain percentage of each reactor volume was transferred to the other (the noted volume percentage in the adaptation phase was 40% and in the main experiment were as “flow recycling percent” column in Table 1). Then, as at the end of Step 1, reactor 2’s content was discharged after the retention time and used for testing. Reactor 1’s wastewater was pumped to reactor two and then, reactor one was filled with raw wastewater from the feed container.
- Finally, for each concentration of PG, step 2 was repeated until reaching steady-state conditions (less than 2% difference in treatment efficiency). The removal efficiency in steady-state conditions was reported in this paper.

Fig. 2 2D scheme of feed backward serial sequencing batch reactor **Table 1** RSM main experiments details

Run	Retention time (h)	Influent COD (mg/L)	Flow recirculation percentage (%)
1	5.00	1500.00	27.50
2	3.00	900.00	20.00
3	5.00	1500.00	27.50
4	5.00	1500.00	40.11
5	3.50	2100.00	20.00
6	5.00	1500.00	27.50
7	7.52	1500.00	27.50
8	3.50	900.00	35.00
9	2.48	1500.00	27.50
10	6.50	2100.00	35.00
11	5.00	1500.00	14.89
12	3.50	2100.00	35.00
13	5.00	1500.00	27.50
14	6.50	900.00	35.00
15	6.50	900.00	20.00
16	5.00	1500.00	27.50
17	6.50	2100.00	20.00
18	5.00	1500.00	27.50
19	5.00	490.92	27.50
20	5.00	2509.08	27.50

2.2 Dependent and independent parameters and RSM

In this research, a completely new reactor (FBSSBR) was introduced for the biological treatment of propylene glycol. The primary purposes of this research were statistical analysis, modeling, and determination of optimum

conditions. For these objectives, the experiments were designed with the response surface methodology.

Response surface methodology has been used in environmental studies, previously [1, 17, 22, 23, 49, 50]. The foundation of RSM is mathematical and statistical techniques. In RSM, linear or square polynomial functions are used for modeling, optimization, and system study [9].

RSM modeling requires effective factors selection. The next step in modeling is determining each factor's maximum and minimum for experimental matrix design. This process shows the number of main experiment tests. The main goal of the experimental design is to obtain statistically reliable results. The code levels (+1) and (−1) indicate the maximum and minimum limits of each parameter that these two levels should determine based on the main idea of the study. Finally, there is a third level or central (0) between the maximum and minimum.

Design-Expert 7.0.0 software was used for experimental design and analysis of the results. Based on the conditions mentioned in Table 2, three independent variables (retention time, influent COD, and flow recirculation percentage) and a dependent variable of COD removal were selected, and RSM model suggested twenty experiments for the main experiments.

2.3 Microorganisms preparing and adaptation; nutrient compound details

Microorganisms were prepared from returned sludge to activated sludge tank in a municipal wastewater treatment plant. Around 40% of the reactors' volume was filled with sludge (microorganisms), and the rest of the effective

Table 2 RSM design, experiments condition summary

Factor	Units	Type	Low actual	High actual	Low coded	High coded	Mean
Retention time	h	Numeric	3.50	6.50	−1.000	1.000	5.00
Influent COD	mg/L	Numeric	900.00	2100.00	−1.000	1.000	1500.00
Flow recirculation percentage	%	Numeric	20.00	35.00	−1.000	1.000	27.50

Table 3 RSM main experiments details

Parameter	Measure
PH	7.2
Temperature (°C)	20
Dissolved oxygen (mg/L)	1.8
Total suspended solid (mg/L)	3500

volume was filled with wastewater. Table 3 indicates some necessary details of used sludge.

The aeration pump adjusted to mix the reactor content slowly by aeration and keep the dissolved oxygen near 1.5–2.5 mg/L. In specific periods, pH was kept fixed in the range of 6.8–7.2. The experiments were conducted at an ambient temperature between 21 and 25 °C. Inlet COD concentration was 200 (mg/L) during the adaptation period. At the beginning of each cycle, reactors were fed with 100:5:1, C:N:P ratio wastewater. In the adaptation phase, carbon was from glucose and propylene glycol, and in the main experiments, it was from propylene glycol.

The adaptation nutrients order from glucose and PG feed were in accordance with Fig. 3. In the feeding period, retention time was 7.5 h and 40% of reactors volume could circulate between two reactors. For each input, this would

be repeated until FBSSBR reached the steady-state condition. In all of the situations in Fig. 3, removal efficiency was 100%. After microorganism adaptation, in the main experiments (Table 1), PG was used as carbon source. Urea was the primary nitrogen source, and KH₂PO₄ was the phosphor source. C:N:P ratio was fixed in the range of 100:5:1 during all experiments. Table 4 represents other details about micronutrients, which were used to increase the efficiency and activity of microorganisms.

2.4 Experiments instructions

In this research, all experiments were conducted in line with *Standard Methods for the Examination of Water and Wastewater* [16].

3 Results and discussions

As mentioned, three main factors (retention time, influent COD, and flow recirculation percentage) were used as independent variables. For each run, removal efficiencies were determined, and they are presented in Table 5. According to Table 5, maximum removal efficiency was

Fig. 3 Order of nutrient compound input from glucose and PG for adaptation feeding

Table 4 details of used micronutrients

Ingredients	Compound name	Chemical formula	Adaptation concentrate (mg/L)
Carbon source	Glucose	$C_6H_{12}O_6 \cdot 6H_2O$	0–200
	Propylene glycol	$C_3H_4O_2$	0–200
Nutrients	Urea	H_2NCONH_2	10
	Potassium hydrogen phosphate	K_2HPO_4	1
	Potassium dihydrogen phosphate	KH_2PO_4	1
	Magnesium sulphate	$MgSO_4 \cdot 7H_2O$	5
Small nutrients	Calcium chloride	$CaCl_2$	3.75
	Iron(III)chloride	$FeCl_3 \cdot 6H_2O$	1
	Sodium molybdate	$Na_2MoO_4 \cdot 2H_2O$	1.26

Table 5 Removal efficiency of RSM main experiments

Run	Retention time (h)	Influent COD (mg/L)	Flow recirculation percentage	Removal efficiency percentage
1	5	1500	27.50	67
2	3	900	20	70
3	5	1500	27.50	67
4	5	1500	40.11	78
5	3.50	2100	20	30
6	5	1500	27.50	67
7	7.52	1500	27.50	83
8	3.50	900	35	85
9	2.48	1500	27.50	36
10	6.50	2100	35	74
11	5	1500	14.89	64
12	3.50	2100	35	38
13	5	1500	27.50	67
14	6.50	900	35	96
15	6.50	900	20	93
16	5.00	1500	27.50	67
17	6.50	2100	20	56
18	5	1500	27.50	67
19	5	490.92	27.50	98
20	5	2509.08	27.50	45

98% in 5 h retention time, 490.92 mg/L of initial COD concentration, and 27.5% recycling flow rate.

3.1 Individual effect of each parameter

One dimensional diagram represents each factor's effect on the removal efficiency percentage separately. In each discussion, two out of three independent variables were considered as a fixed value, and the removal efficiency diagram was illustrated based on effect of one variable.

3.1.1 The effect of retention time

Figure 4 shows the removal efficiency in different retention times. According to the results, change of retention time between 3.5 and 6.5 h would cause removal efficiency to increase from 51% to nearly 77%. Hence, it can be concluded that in the FBSSBR system, removal efficiency and retention time are directly related. Previous researchers could confirm this augment; for example, in a fixed bed activated hybrid sludge reactor, there was a similar trend of increase in removal efficiency after increasing retention time [15]. This is in that microorganisms have more consumption and more contact with the contamination within the longer retention time.

3.1.2 The effect of influent COD concentration

Figure 5 shows the removal efficiency percentage in different influent COD concentrations. The inverse relationship between inlet COD and removal efficiency is apparent because toxicity increases when COD increases. In other words, in high PG concentrations, microorganisms will lose their metabolic. This research attempts to improve the ability of microorganisms in the FBSSBR system with the adaptation phase.

In Fig. 5, influent COD concentration changes in the range of 900–2100 mg/L. Based on the outcomes, the removal efficiency was 87% for 900 mg/L initial COD concentration, and close to 50% for 2100 mg/L initial COD.

Synthetic PG wastewater treatment in an activated sludge reactor showed the same trend [48]. A Fixed Bed Activated Sludge Hybrid Reactor also showed the reduction of removal efficiency by increasing in influent COD [15]. Wastewater containing PG (Paint industries' wastewater) treatment with submerged attached growth reactor (SAGR) in both batch and continuous conditions validated this research result.

Fig. 4 The effect of retention time on COD removal efficiency (Influent COD = 1500 mg/L, flow recirculation percentage = 27.5%)

Fig. 5 The effect of influent COD concentration on COD removal efficiency (Retention time = 5 h, flow recirculation percentage = 27.5%)

3.1.3 The effect of flow recirculation percentage

As seen in Fig. 6, removal efficiency and flow recirculation percentage have a direct relation. By increasing the flow recirculation percentage from 20 to 35%, removal efficiency also increased from 64% (540 mg/L output COD) to 73% (405 mg/L output COD). Flow recirculation percentage was investigated for the first time in this research by introducing FBSSBR reactor, which has a positive effect on removal efficiency.

3.2 Simultaneous effect of parameters

3.2.1 The effect of retention time and influent COD concentration

The simultaneous effect of retention time and influent COD concentration on COD removal efficiency was shown in Fig. 7. With influent COD concentration changing between 900 and 2100 mg/L and retention time between 3.5 and 6.5 h, removal efficiency was improved from 32% (for 2100 mg/L COD and 3.5 h retention time) to 89.75%

Fig. 6 The effect of flow recirculation percentage on COD removal efficiency (Retention time = 5 h, Influent COD = 1500 mg/L)

Fig. 7 The effect of retention time and influent COD concentration (flow recirculation percentage = 27.5%)

(for 900 mg/L COD and 6.5 h retention time). The output COD in 89.75% removal efficiency was 99 mg/L, which complies with environmental regulations such as USEPA and CEPA standards.

A closer look at Fig. 7 indicates the more significant effect of retention time in comparison with influent COD concentration. This would indicate the importance of finding an optimum level between these two variables.

3.2.2 The effect of retention time and flow recirculation percentage

Figure 8 shows the effect of retention time and flow recirculation percentage on removal efficiency. The retention time ranged from 3.5 to 6.5 h, and flow recirculation percentage ranged from 20 to 35%. These changes caused decline in the COD removal efficiency in the range of 35% (975 mg/L output COD) to 84%

Fig. 8 The effect of retention time and flow recirculation percentage (influent COD = 1500 mg/L)

(240 mg/L output COD). As shown in Fig. 8, FBSSBR, COD removal efficiency in the system increased by the increase of these two factors. By using the FBSSBR system idea, some non-degraded pollutants entered reactor one again. Therefore, microorganisms had more chances to adapt and remove the COD. Increasing the retention time also gave more exposure time to microorganisms and improved COD removal efficiency.

3.2.3 The effect of influent COD concentration and flow recirculation percentage

Figure 9 shows the effect of changing in influent COD concentration and flow recirculation percentage. The influent COD concentration range was 900–2100 mg/L, and flow recirculation percentage range was 20–35%. In these conditions, the removal efficiency changed from 48% (1092 mg/L output COD) to 84% (72 mg/L output COD). It seems reduction in influent COD concentration positively

Fig. 9 The effect of influent COD concentration and flow recirculation percentage (retention time = 5 h)

Fig. 10 The effect of three independent variables

affects removal efficiency. This effect is much greater than the flow recirculation percentage effect.

3.3 Cubic diagram

A cubic diagram determines all three variables' effects on removal efficiency. The effects of Retention time, influent COD concentration, and flow recirculation percentage on COD removal efficiency are shown in Fig. 10.

The maximum removal efficiency (97.82%) occurred within the 6.5 h of retention time, 900 mg/L of influent COD concentration, and 35% of flow recirculation percentage. Minimum removal efficiency (27.86%) occurred within

the 3.5 h of retention time, 2100 mg/L of influent COD concentration, and 20% of flow recirculation percentage. Output COD in maximum removal was 72 mg/L.

3.4 Interaction effects of parameters

Interaction diagrams show the interactive effect of two variables on removal efficiency. These effects can be synergistic (where two variables make a positive effect on each other for removal efficiency) or antagonistic (where two variables tend to weaken positive effects of each other on the removal efficiency).

Fig. 11 Interactive effect of retention time and flow recirculation percentage (influent COD = 1500 mg/L)

Fig. 12 Interactive effect of retention time and influent COD concentration (flow recirculation percentage = 27.5%)

3.4.1 Interactive effect of retention time and flow recirculation percentage

Figure 11 shows the interaction between retention time and flow recirculation percentage. In this figure, when retention time is 3.5 h, by changing inflow recirculation percentage from 20 to 35%, change in removal efficiency is 47–60%. With 6.5 h retention time and the same situation, there was a shift from 73 to 84% in removal efficiency. The diagram slope in 3.5 h of retention time is 0.87, and 6.5 h of retention time is 0.73. This minimal difference indicates the little effect of these two factors on each other.

3.4.2 Interactive effect of retention time and influent COD concentration

Figure 12 depicts the interactive effect of influent COD concentration and retention time. In this figure, by applying changes in retention time between 3.5 and 6.5 h, removal efficiency changed from 75 to 92% when the influent COD was 900 mg/L. Moreover, the change in removal efficiency

was 33–65% when influent COD was 2100 mg/L. The diagram slope of 900 mg/L of influent COD concentration is 5.67, and in 2100 mg/L of influent COD concentration is 10.67. This difference indicates the antagonistic effect of these two factors on each other. Despite less removal efficiency in higher influent CODs, raise in retention time has stronger effect on samples with higher initial COD.

3.4.3 Interactive effect of influent COD concentration and flow recirculation percentage

Figure 13 shows the interactive effect of flow recirculation percentage and influent COD concentration. In this figure, where the flow recirculation is 20%, by changing influent COD concentration (between 900 and 2100 mg/L COD), removal efficiency changed from 84.5 to 47%, and when there was 35% flow recirculation percentage, removal efficiency deduced from 90.2 to 59%.

3.5 Mathematical model

After results analysis, Eq. 1 was obtained.

$$\begin{aligned}
 \text{Removal Efficiency percent} = & +86.52052 + 14.73595 \times \text{Retention Time} - 0.068207 \text{ Influent COD} \\
 & - 1.02736 \text{ flow recirculation percentage} + 3.88889\text{E} - 003 \times \text{Retention Time} \\
 & \times \text{Influent COD} - 0.022222 \text{ Retention Time} \times \text{flow recirculation percentage} \\
 & + 2.22222\text{E} - 004 \text{ Influent COD} \times \text{flow recirculation percentage} \\
 & - 1.14133 \times \text{Retention Time}^2 + 4.65181\text{E} - 006 \times \text{Influent COD}^2 \\
 & + 0.026629 \times \text{flow recirculation percentage}^2
 \end{aligned} \quad (1)$$

Fig. 13 Interactive effect of influent COD concentration and flow recirculation percentage (retention time = 5 h)

Based on Eq. 1, the effect of retention time is more than the other two factors. The comparison between effectiveness of each factor is as the following:

Retention Time > flow recirculation percentage > Influent COD

3.6 Analysis of variance

The ANOVA test strategy is based on using data to calculate the effect of each factor on the results. Table 6 indicates the ANOVA analysis result. The mean square and Fisher value are some of the factors of this table, but the important column in this table is the *P* value column.

The *P* value of the model was below 0.0001, which shows that the presented mathematical model is statistically significant.

3.7 Optimum condition and validation investigation

The optimum condition occurred in the 3.52-h of retention time, 1667.76 mg/L of influent COD concentration, and 23.23% of flow recirculation percentage. Due to this condition, based on the mathematical equation, the optimum removal efficiency was 47%. Based on experimental

Table 6 Analysis of variance results

Source	Sum of squares	df	Mean squares	F value	<i>P</i> value prob > F
Model	6918.37	9	768.71	76.54	< 0.0001
A-retention time	224,360	1	2243.60	223.40	< 0.0001
B-influent COD	4048.41	1	4048.41	403.11	< 0.0001
C-flow recirculation percentage	334.07	1	334.07	33.26	0.0002
AB	98.00	1	98.00	9.76	0.0108
AC	0.50	1	0.50	0.050	0.8279
BC	8.00	1	8.00	0.80	0.3931
A ²	95.04	1	95.04	9.46	0.0117
B ²	40.42	1	40.42	4.02	0.0726
C ²	32.33	1	32.33	3.22	0.1030
Residual	100.43	10	10.04		
Lack of fit	100.43	5	20.09		
Pure error	0.000	5	0.000		
Cor total	7018.80	19			

Fig. 14 Predicted values versus actual values

results, this removal efficiency had less than 5% error, and this error showed the model's accuracy. The experimental results confirmed the model results ultimately, as seen in Fig. 14.

4 Conclusions

This research studied FBSSBR's practical factors simultaneously. The RSM model was successfully applied to investigate the effect of three independent factors. The retention time was the most effective factor. Influent COD concentration by changing the environment's toxicity was the next effective factor. Approximately 97.82% was the highest removal efficiency. Among the other biological reactors, FBSSBR was successful in PG treatment.

Compliance with ethical standards

Conflict of interest The authors declare they have no conflict of interest.

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