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Lab-scale optimization of propylene glycol removal from synthetic wastewater using activated sludge reactor

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ABSTRACT

Propylene glycol (PG) is not a toxic matter. However, it can dramatically increase BOD of water resources and that is why removal of PG is important. Removal of PG in synthetic wastewater was studied in a continuous activated sludge pilot-scale reactor. The influence of various factors (pH, nitrogen source, COD and wastewater feed salinity (conductivity)) on micro-organism growth as a measure of removal was determined, and the optimum condition for maximizing this response was obtained using Taguchi experimental design method. Primary micro-organisms were obtained from the return sludge line of the Shahrak-Gharb wastewater treatment system located in the city of Tehran. The micro-organisms have been adapted to high organic loads during five stages in 119 days. The maximum PG removal efficiency was equal to 85%. In the selected range of levels, the best pH was equal to 8 and the influent COD was 1300 mg/L. The best nitrogen source was urea, and salinity was obtained equal to 8%.

Keywords: Propylene glycol; Wastewater treatment; Biological removal; Optimization; Taguchi method

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1. Introduction

Increasing development of various industries leads to the release of highly pollutant effluents into the environment. Removal of hazardous materials resulting from these effluents is one of the most important responsibilities of environmentalists and other professionals in related fields. Propylene glycol (PG) is one of the products where various derivations are used for surfactant production in petrochemical industries. PG has wide applications in pharmacological, cosmetics, chemical and food-processing industries. After conversion to the poly (PG), it is used for polyurethanes production [1,2]. PG is also the main constituent of anti-freezing fluids which is widely used in airports [3].

Due to its wide application and as a result of its effluents discharge to the environment, PG may

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spread in soil, and contaminate underground and surface waters easily. PG consumes dissolved oxygen (DO) in water recourses and highly influences the water quality [4,5].

Biological treatment can be used to remove such pollutants effectively. Many researchers have studied the removal of glycol compounds. Evans and David [6] have carried out a study on mono-, di- and tri-ethylene glycol removal under controlled laboratory conditions. Although mono-ethylene glycol can be removed at 20˚C within 3 days, the full removal of this compound was not possible within 8 days at actual river temperature (8˚C) [6]. Zgola et al. (2006) have compared the removal of PG and ethylene glycol. They succeeded in removing a large amount of these compounds in their experiments in the liquid phase [1]. Zgola et al. (2007) also studied the bio-oxidation of PG in the activated sludge process, and found that PG in concentration more than 10 mg/L is extremely toxic for biologic systems [7]. Shupack et al. [8] surveyed the mineralization of PG in soil, which was done in temperatures ranging from 7 to 22˚C. Using this method, the toxic effect of PG was reduced effectively [8]. Some environmental parameters, such as pH, temperature, nitrogen source, phosphorus source, salinity and carbon source concentration can have a strong effect on biological processes efficiency. The use of an optimum amount of these parameters can lead biological processes to have high efficiency.

The most important subject in biological process is to optimize the process to achieve the maximum removal of pollutants by changing environmental conditions. Experimental design approaches including the Taguchi method have found wide applications in determining the effect of factors on the process responses and to find the optimum conditions of biological processes of organic pollutants [9]. Although some research has been conducted on the PG removal using an activated sludge bioreactor, the optimum condition has not been studied yet using a trustable method, such as Taguchi. The main objective of this study is to achieve a high PG removal efficiency by determining the optimum conditions in a completely mixed activated sludge process. Activated sludge has the advantages of simple operation and construction, low odour, no accumulation of insects and high efficiency compare to the other existing systems [10]. The ranges of pH, nitrogen source, carbon source concentration and salinity were first determined by reviewing the literature. Then, the optimal values of the selected factors and their effectiveness were found experimentally in a biological reactor loaded with adapted micro-organisms using Taguchi method.

2. Experiment

2.1. Pilot-scale bioreactor

A Plexiglas pilot-scale bioreactor (activated sludge) of 15 cm in diameter and 34 cm in height with the total volume of 6 L was used in this study. The effluent tube was placed fixed at 28 cm in height so that the effective volume of aeration tank was 5 L. The free board was 5.5 cm. The wastewater in the bioreactor was aerated and mixed with an air pump using an air diffuser. The flow rate of diffused air was measured with a flow meter and was adjusted by a valve. A secondary sedimentation tank was used for clarification and recycling the settled sludge to the aeration tank if found necessary. Secondary clarification was performed in a rectangular tank with the effective volume of 2 L. Feeding and recycling return sludge to the bioreactor was carried out with two peristaltic pumps. The pilot bioreactor was operated constantly at room temperature (20–25°C). Fig. 1 shows the schematic diagram of the bioreactor pilot plant.

2.2. Culture medium and synthetic wastewater

A mineral solution was used as the culture medium. This solution contained $MgSO_4$ 0.1 g/L, KH_2PO_4 0.5 g/L, $CaCl_2.2H_2O$ 0.01 g/L, $FeSO_4$ 0.001 g/L, NH_4Cl 1 g/L, K_2HPO_4 0.5 g/L and $MnSO_4$ 0.001 g/L. The pH of this solution was adjusted by NaOH in the range of 7 ± 0.5. This solution was also applied as the basis of the synthetic wastewater. The sole carbon source of the synthetic wastewater was a combination of glucose and PG which were added to the solution to prepare synthetic wastewater. Several biological experiments were performed with the wastewaters containing different concentrations of PG and glucose using the same micro-organisms. When the micro-organisms were adapted for consuming PG, the concentration of glucose decreased and the concentration of PG increased gradually in a series of treatment experiments. The concentration levels of other ingredients were kept constant in the solution for all experiments.

2.3. Bioreactor start-up

First, 5 liters of sludge was taken from the return sludge line of a full-scale municipal wastewater treatment plant in Ahvaz city of Iran and aerated for 2 weeks in batch conditions. The flow rate of air was adjusted to provide the adequate level of DO (1–3 mg/L). A mixture of PG and glucose was added to the synthetic wastewater to create the total COD of

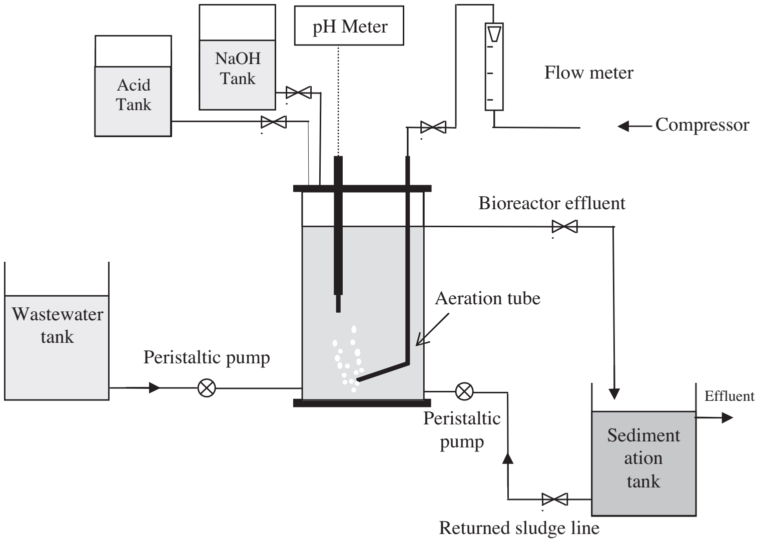

Fig. 1. Schematic diagram of bioreactor used in this study.

400 mg/L. The amount of added PG and glucose was such that 350 mg/L of total COD was created by glucose and the rest by PG. This solution was fed to the reactor in the start-up stage. During the operation, 1.5 L of supernatant was decanted from reactor and 1.5 L of fresh synthetic wastewater was added to the reactor daily. In addition, 100 mL of pure water was added to the solution to compensate for water evaporation. This procedure was repeated daily for 2 weeks. At the end of the second week, the reactor was operated continuously and the flow rate was adjusted to obtain the hydraulic retention time of 12 h. Then, the concentration of glucose was reduced and the concentration of PG was increased daily, with such a rate that only a total COD of 400 mg/L was created with the PG at the end of fourth week.

The industrial biological processes were performed in the environment temperature, and this reactor was operated at room temperature (20–25˚C).

2.4. Micro-organisms adaptation

Several methods have been developed to adapt micro-organisms for living with toxic materials [11]. For adapting biomass with high concentrations of PG, COD was increased from 400 mg/L to 700, 900, 1,100 and 1,300 mg/L, respectively. Total stages of experiments lasted for 119 days. During this period, pH was adjusted by NaOH and hydrochloric acid at 7 ± 0.3.

2.5. Experimental design

The first important step in the design of this experiment is the proper selection of factors and their levels. In this study, four environmental factors (pH, nitrogen source, influent COD (mg/L) and salinity percentage) were considered (Table 1). These factors and their levels were chosen according to the literature review and based on previous publications, the practical aspects, and some screening experiments. For the Taguchi design of experiments with four factors, a standard L_9 orthogonal array was employed (Table 2), using Qualitek-4 (Nutek Inc.) software. Each row of the matrix represents one run at a specified condition. In order to avoid the systematic bias, the sequence in which these runs were carried out was randomized [12]. In the selected coordinate system, the codes 1, 2 and 3 represent the low, medium and high levels, respectively.

To perform the designed experiments, reactor was conducted in four experiments with different conditions as in Table 2. Each experiment was conducted to achieve study-state condition. The amount of carbon source was controlled according to $NaNO_3$, NH_4Cl and $CO(NH_3)_2$ concentrations so that the C/N ratio

Table 1
Selected factors and their levels

	Levels		
Factors	Low (1)	Intermediate (2)	High (3)
	6	7	8
Nitrogen source	$NaNO_3$	NH_4Cl	$CO(NH_3)_2$
Influent COD (mg/L)	400	900	1,300
Salinity percentage	4%	6%	8%

Table 2
Designed experiments and resulted PG removal

	Factors				Percentage of PG removal		
Run	pH	Nitrogen source	Influent COD (mg/L)	Percentage of salinity	Replication #1 (%)	Replication #2 (%)	S/N
1	1	1	1	2	5	9	15.821
2	1	2	2	3	41	36	31.654
3	1	3	3	1	85	80	38.317
4	2	1	2	1	37	33	30.838
5	2	2	3	2	63	62	35.916
6	2	3	1	3	82	79	38.111
7	3	1	3	3	65	60	35.896
8	3	2	1	1	70	66	36.638
9	3	3	2	2	64	70	36.495

could be kept at 100/5 throughout the experiment [10]. The pH of synthetic wastewater was adjusted using HCl and NaOH.

2.6. Analytical methods

Salinity was measured by a salinity meter that worked in the range of 0–100%. Measurement of DO demand was performed by Winkler method, and the COD was monitored in each experiment according to standard methods [13]. pH was measured by digital pH meter.

3. Results and discussion

3.1. Bioreactor start-up and micro-organisms adaptation

It is indicated in Fig. 2 that the COD removal efficiency at the end of the adaptation period for COD feeding of 400 mg/L has been 98% (Std ± 1.28). In order to invigorate micro-organisms to survive in presence of PG and to consume it as the main carbon source, COD was increased to 1,300 mg/L in five steps, as shown in Fig. 2. The average of COD removal for influent COD concentrations of 700, 900, 1,100 and 1,300 mg/L was 95% (Std ± 5.3), 93% (Std ± 3.65), 90% (Std ± 7.58) and 85% (Std ± 12.37), respectively. It can be implied from this trend that the micro-organisms could adapt themselves well with COD increasing in the system. The bioreactor performance decreases slightly as the influent COD concentration increases. After 119 days, at the highest value of loading (1,300 mg/L), the COD removal is up to 85%. Although this is a reasonable performance, it is expected to be improved by optimization of bioreactor.

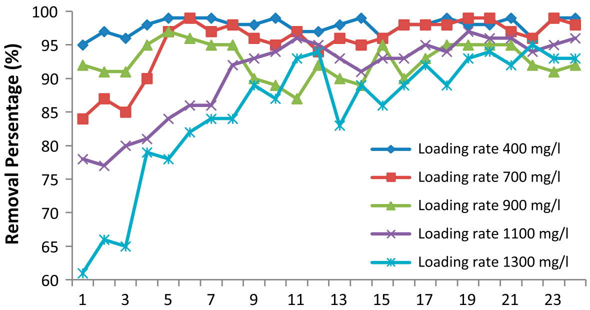

Fig. 2. Percentage of COD removal due to PG depletion at five organic loading rates during the adaptation of micro-organisms.

Relation between the PG concentration and the removal efficiency of PG can be described by following liner equation (Fig. 3). This equation is results of liner regression between the removal efficiency and different PG concentrations. This equation has a high correlation (R^2) coefficient which is equal to 0.96.

$$y = -ax + b$$

where y is the PG removal efficiency (in %), x is the concentration of PG; and a and b are constant coefficients and defined as biological removal rate coefficient (in percentage per pollutant concentration) and maximum reactor efficiency coefficient (in %), respectively. Based on the results of experiments as shown in Fig. 3 constant coefficients a and b are equal to 0.013 and 104.1, respectively. It was found that, under similar conditions, when the PG concentration is 315 mg/L it can be completely removed using this reactor.

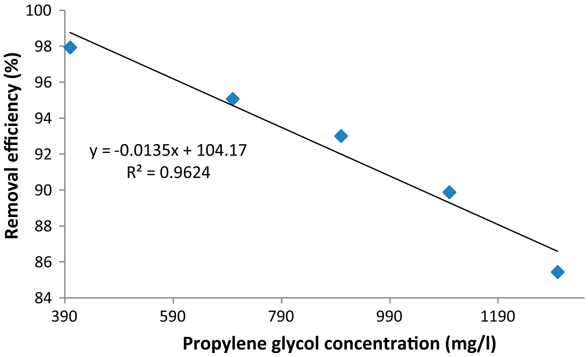

Fig. 3. Relation between PG concentration and removal efficiency.

3.2. Taguchi transformed response

When micro-organisms were adapted to the system, the Taguchi experimental design was applied in order to investigate the influences of environmental factors on PG consumption by micro-organisms or equivalently COD removal. The results of repeated COD measurements at different experimental runs are reported in Table 2.

In Taguchi method, a transformed response called signal-to-noise (S/N) ratio is usually used for the analysis of experimental results. The signal-to-noise ratio indicates the magnitude of changes in response to the variation of controlled factors with respect to that of errors. Therefore, the higher value of S/N ratio is desirable in all quality characteristics (QCs). The QC used in this study was "bigger is better"; i.e. the higher the COD (or PG) removal, the better the bioreactors performance is. In order to perform the statistical analysis for this QC, the S/N ratio was calculated from the following equation [7,12]:

$$\frac{S}{N} = -10 \log \frac{(1/y_1^2 + 1/y_2^2 + \ldots + 1/y_n^2)}{n}$$

where y_i is the COD measured at reactor exit for ith experiment, and n is the number of measurements carried out for each run. The unit of S/N ratio is decibel, which is frequently used in communication engineering [9]. The S/N ratio obtained for each experiment is presented in Table 2.

3.3. Main effects

The trends of an influence of each factor on S/N (corresponding to COD removal) are discussed in the following sections. It should be noted that the interpretation of these results is valid just for the range of levels considered for the factors in this study.

3.4. Effect of pH

The pH or hydrogen ion concentration of environment is extremely effective on microbial growth [7]. The range of pH, over which the organism grows, is defined by three cardinal points: (1) the minimum pH, below which the organism cannot grow; (2) the maximum pH, above which the organism cannot grow; and (3) the optimum pH, at which the organism grows best. For most micro-organisms, there is an orderly increase in the growth rate between the minimum and the optimum and a corresponding orderly decrease in the growth rate between the optimum and the maximum pH, reflecting the general effect of changing hydrogen ion concentration on protein and the rate of enzymatic reaction [10,11] and therefore the micro-organisms cannot be efficient to remove pollutants from environment.

Fig. 4 shows the influence of pH of wastewater on PG removal. It can be seen from Fig. 4 that the pH increases the PG removal due to enhancement of micro-organisms' growth. This effect is more pronounced when pH is shifting from acidic to neutral values, and the performance is just improved slightly when pH is increased from 7 to 8. The highest PG removal was obtained at a neutral pH value. At lower pH, PG removal decreased due to the inhibition of microbial growth. Majority of the micro-organisms in this study were considered neutrophilic, since they were obtained from a municipal wastewater treatment plant to inoculate in the reactor [10]. Therefore, it can justify why the best PG removal can be achieved in

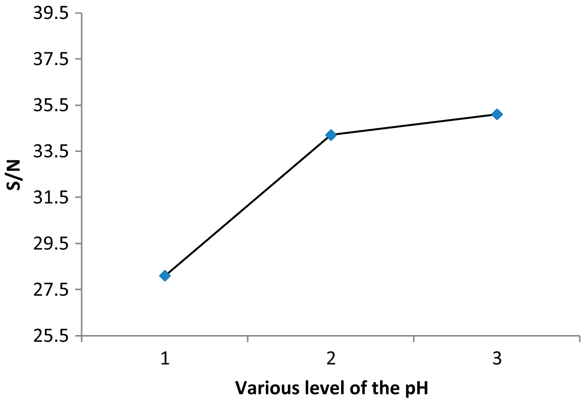

Fig. 4. Effect of wastewater pH on the PG removal and micro-organisms concentration.

neutral pH. Fulazzaky et al. [14] showed that micro-organisms can be adapted to be active under unusual pH conditions (pH less than 3 or more than 9) after a long adaptation period of time. This adaptation could be partially improved in order to provoke the removal efficiency of pollutants by micro-organism. However, the amount of this improvement in removal efficiency was not observed very high during the study.

3.5. Effect of nitrogen source

Nitrogen is undoubtedly a vital element for growth as well as for the metabolism of micro-organisms, and is an essential element in enzymes. Bancroft et al. [15] showed that heterotrophic bacterial populations in a biological process hve a faster growth by the presence of special nitrogen sources and therefore finding the best nitrogen source can be useful to achieve higher efficiency of biological reactors. In this study, three different nitrogen-containing compounds were examined. The influence of nitrogen source in wastewater on the PG removal can be observed in Fig. 5. The best result has been obtained for urea, which is also the most readily available and the cheapest compound amongst the three nitrogen sources examined. In addition, urea contains both carbon and nitrogen elements and can be considered as a dual source for micro-organisms.

Although urea incorporates an additional COD content to the environment, observation indicates the high activity and capability of micro-organisms to reduce the COD contents altogether. The better response of system with urea may be attributed to higher biodegradability of this compound than that of PG. The urea can be degraded early in a bioreactor and it results in an accelerated micro-organism growth. This in turn leads to consumption of PG molecules with a populated numbers of micro-organisms. In comparison to urea, NH_4Cl and $NaNO_3$ cannot play well the roll of micro-organism procreative in our system. This however does not correspond to results reported by Li et al. [16] for a different wastewater treatment system, in which these two compounds were found as a suitable nitrogen source. Mancinelli and McKay [17] found that nitric oxide (NO) and nitrogen dioxide (NO_2) are very suitable for a group of micro-organisms containing *Staphylococcus aureus*, *Micrococcus luteus*, *Micrococcus roseus*, *Serratia marcescens*, *Bacillus subtilis*, *Bacillus circulans*, *Bacillus megaterium* and *Bacillus cereus*. However, these nitrogen sources could not accelerate the growth of micro-organisms. The type of isolated micro-organisms as well as other environmental conditions, such as pH, carbon source concentration, and temperature, is probably responsible for different observations.

3.6. Effect of entering COD concentration

Three different concentrations of PG were used to supply the COD levels fixed at 300, 900 and 1,300 mg/L, in the bioreactor influent. It is indicated in Fig. 6 that the related S/N ratio increases as the entering COD is increased. A greater concentration of PG means a higher amount of nutrient available for micro-organisms. This leads to extend the logarithmic period of micro-organisms growth, and therefore the acceleration of biomass generation. A limitation is expected however, as a very high amount of PG may result in the toxicity condition for micro-organisms, and thus has an adverse effect. In this paper, the PG concentration has been controlled below the toxic limits at all levels.

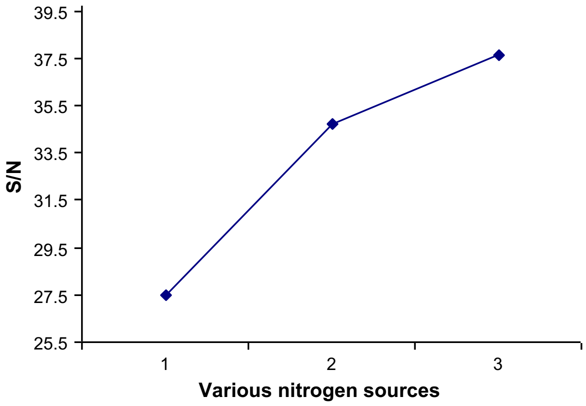

Fig. 5. Effect of various nitrogen sources on the PG removal.

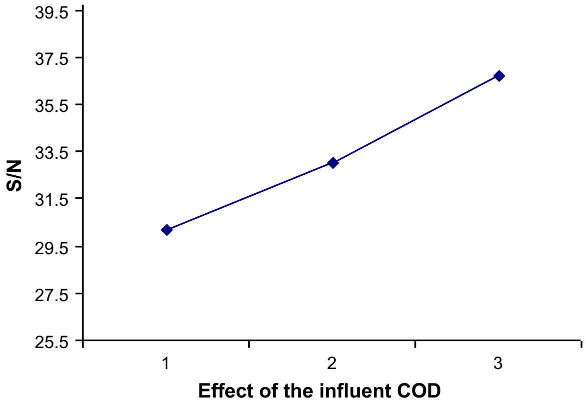

Fig. 6. Effect of wastewater COD on the PG removal.

According to Eiler et al. [18] the micro-organism growth rate in the exponential growth phase exhibits a hyperbolic response to the carbon source concentration. Increasing microbial concentration can be the reason for higher substrate removal in biological reactors. These results are similar to the results obtained during this study. Based on previous studies, carbon source until a limited concentration has a greater influence on micro-organism growth rate. This influence can be seen in the results of this experiment (Fig. 6). The carbon source concentration had also a major influence on the composition of the micro-organism communities that develop in the reactor [19]. Therefore, the main impacts of carbon source concentration in a biological rector are (a) micro-organism growth rate, (b) composition of the micro-organism communities, and (c) removal of substrate.

3.7. Effect of salinity

All organisms with a semi-permeable membrane are subjected to osmotic pressure, or the effect of water moving in and out of the cell. Bacteria must live in an aqueous environment which can be a hypotonic environment [20]. In these type of environment, the concentration of water outside the cell is greater than the concentration of water inside the cell. This causes the net movement of more water into the cell than outside. If the bacterium did not have a cell wall, this could cause the cell to burst. When the outside environment around a cell is salty, then the concentration of water in the solution is less than that inside the cell and water tends to leave the cell. This causes the cell to dehydrate, which eventually kills the cell [21,22]. By subjecting bacteria to a salty environment, it keeps them from growing. Some bacteria, however, have adapted to living in salty environments (halophiles) [23].

In Fig. 7, the effect of salinity on Taguchi transformed response is indicated. When the salinity is changed from 4 to 6%, a high impact is observed in PG removal. This behaviour is justified by the effect of salinity on the uptake of some vital ions by pumping mechanism through the cellular membranes. The uptake of cations like Ca^{2+}, Mg^{2+}, Na^{+} and K^{+} by cells depends to the conductivity of water. The salinity indeed affects the conductivity, and therefore the capability of micro-organisms to receive the necessary ions.

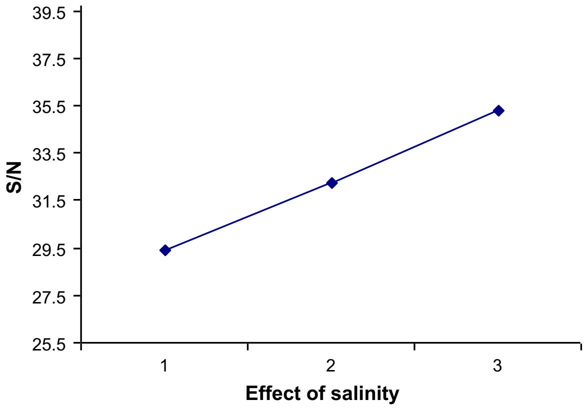

Fig. 7. Effect of wastewater salinity on the PG removal.

Due to osmosis effect that decays the micro-organism growth, a very high salinity may however lead to lesser amount of water in the cell [10]. The two opposite effects may probably justify the less influence of salinity on micro-organism activities at higher levels. The ability of micro-organisms to be active at high salinity conditions is of importance as many wastewater produced in oil and gas industries contains high amounts of different salts [24]. Many researchers have tried to find out the effects of salinity on biological activity and the optimum concentration of saline depends on many factors like type of carbon source, micro-organism strains and previous exposure of micro-organisms with high salinity [21–23].

3.8. Analysis of variance

The analysis of variance (ANOVA) is a powerful technique in Taguchi method that explores the percent contribution of factors affecting the response. The strategy of ANOVA is to extract from the results how much variations each factor causes relative to the total variation observed in the result. The statistical analysis of the results was carried out using Qualitek-4 (Nutek Inc.) software. Table 3 shows the ANOVA statistical terms representing the significance of four environmental factors affecting the PG removal. It is implied from the contribution percents in the last column of ANOVA table that all of the factors are more

Table 3
ANOVA for factors affecting the PG removal

Factor	DOF	Sum of squares	Variance	Percent contribution
pH	2	102.353	51.176	25.746
Nitrogen source	2	162.992	81.496	41.000
Concentration of entering COD	2	64.171	32.085	16.142
Salinity	2	68.020	34.010	17.110
Total	8	70.945	—	100

Table 4
The optimum condition for maximum removal of PG

Factors	Best levels	Contribution (*S*/*N*)
pH	8	3.044
Nitrogen source	$CO(NH_3)_2$	4.342
Influent COD (mg/L)	1,300	3.411
Percentage of salinity	8%	1.965
Total contribution from all factors		12.762
Current grand average of performance		33.298
Expected result at optimum condition (S/N)		46.060

or less important and significantly affect the response. In the range of levels considered for any factors in this study, the following order is observed for significance of factors:

Nitrogen source > pH > salinity > concentration of entering COD.

3.9. Optimum conditions

From Figs. 3–6 and the data in ANOVA table, one can estimate the relative optimum conditions at which the maximum rate of micro-organism growth or the PG removal will be attained. Table 4 indicates the optimum conditions obtained via Taguchi approach. Using urea as nitrogen source, it was observed that all of the factors should be kept at their highest level, in order to obtain the maximum response. However, more experiments are required however around the best levels of these factors in a narrower range, in order to find the exact optimum condition for a defined target.

In Table 4, the percent of improvement in response (S/N) with respect to current average of results is also calculated. It is predicted that application of optimum conditions would improve the transformed response by 38% over the current grand average of performance.

4. Conclusions

The influence of four environmental variables on the rate of micro-organism growth or PG removal in an activated sludge bioreactor was statistically analysed using Taguchi experimental design. The main conclusions that are valid in the range of levels considered in this study are as follow:

The results indicated that the application of Taguchi method was suitable for the optimization of biological removal of PG removal using activated sludge. Results obtained also showed that the activated sludge process have a high efficiency to remove PG from wastewater. The PG removal was dependent on the PG concentration, pH, nitrogen source and salinity. All of the factors examined in this study had significant effect on the PG removal. The nitrogen source had the largest effect and contribution in PG removal. Based on the analysed results using Taguchi method, the relative optimum conditions are estimated as follows: The best nitrogen source: urea; Concentration of entering COD: 1,300 mg/L; pH: 8; and salinity: 8%. These optimized results can be applied to conduct an activated sludge bioreactor to remove PG with maximum efficiency.

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