Techno-economical evaluation of nitrate removal using continuous flow electro-coagulation process: optimization by Taguchi model

E. Karamati Niaragh, M. R. Alavi Moghaddam and M. M. Emamjomeh

ABSTRACT

This study aims to investigate the effect of the main parameters on the performance of a continuous flow electro-coagulation (EC) process for nitrate removal efficiency and its operating costs. For this purpose, the Taguchi experimental design with orthogonal array L27 (3^{13}) was applied to analyze the effects of selected parameters, namely initial nitrate concentration, inlet flow rate, current density and initial pH. According to the analysis of variance results, the inlet flow rate and the current density were recognized to be the most effective factors playing a pivotal role in nitrate removal efficiency by using an EC process. The optimum conditions of initial nitrate concentration, inlet flow rate, current density and initial pH were found to be 100 mg/L, 50 mL/min, 80 A/m^2 and 8, respectively. As a result, the observed nitrate removal efficiency under these conditions was 61.70%. In addition, operating costs were evaluated as 1.278 US$/g NO_3-removed. Finally, a high correlation was observed between the experimental and predicted results indicating an appropriate accuracy of the Taguchi model for nitrate removal efficiency and its operating costs in an EC system.

Key words | continuous electro-coagulation (EC), nitrate removal efficiency, operating costs, optimization, Taguchi model

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INTRODUCTION

Nitrate contamination of groundwater resources emerged as a serious environmental problem due to the incremental usage of groundwater for drinking and agricultural purposes (Hosseinzadeh Talaee 2015). The extensive application of nitrogen fertilizers, the disposal of untreated domestic/industrial wastewater in an unsafe manner, and the leakage in septic systems has increased the nitrate contamination load in waterways. Further, excessive nitrate concentration in drinking water can also cause adverse human health problems such as blue-baby syndrome (methemoglobinemia) (Azadegan *et al.* 2012). Therefore, the World Health Organization (WHO 2013) and the Institute of Standard and Industrial Research of Iran (ISIRI 1996) have determined the acceptable level of 50 mg/L nitrate concentration as NO_3^- in drinking water in order to solve these problems. In order to remove excessive nitrate, efficient conventional methods are used, which are classified into biological decomposition and physico-chemical treatments such as ion exchange, chemical treatment and reverse osmosis (Lacasa *et al.* 2011; Lakshmi *et al.* 2013).

Among the physico-chemical methods, the electro-coagulation (EC) method was recognized as an eco-friendly, efficient and cost-effective process. Further, the successful application of this method was approved for removing various kinds of pollutants (Emamjomeh & Sivakumar 2009a) such as nitrate (Koparal & Öğütveren 2002; Emamjomeh & Sivakumar 2009b; Kumar & Goel 2010; Lacasa *et al.* 2011; Lakshmi *et al.* 2013; Yehya *et al.* 2014; Nazlabadi &

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Alavimoghaddam 2016). Lack of clear establishment of the cell geometry and scale-up procedure is regarded as one of the main demerits of EC. Systematically, EC reactors are classified into batch and continuous systems. In the continuous EC reactor, a continuous feed of water and wastewater is provided under (pseudo) steady/state conditions (Holt *et al.* 2005).

Generally, in the EC process, metallic hydroxide species are created by electro-dissolution of sacrificial anodes (usually made of iron or aluminum), and the pollutants adsorbed on the coagulants produced are removed by sedimentation or flotation (Emamjomeh & Sivakumar 2009a). The flotation of flocs occurs because of the production of hydrogen gas at the cathode, resulting from water reduction. The main cathodic and anodic reactions for the aluminum electrodes are as follows (Yildiz 2008):

$$\text{At the cathode: } 3H_2O + 3e \rightarrow 3/2\,H_2(g) + 3OH^- \quad (1)$$

$$\text{At the anode: } Al \rightarrow Al^{3+} + 3e \quad (2)$$

$$\text{In the solution: } Al^{3+}(aq) + 3H_2O \rightarrow Al(OH)_3 + 3H^+(aq) \quad (3)$$

The Taguchi method, as a statistical experimental design, is implemented to provide reliable data by reducing the number of experiments. Regarding the use of a matrix design in an orthogonal array, the Taguchi design is a preferable method in comparison with the conventional and classical experimental design. In an orthogonal array, all the parameters are varied at the same time. Their effects and performance interactions are studied simultaneously to determine those factors having more or less effect (Martinez-Villafane & Montero-Ocampo 2010). Some researchers applied the Taguchi experimental design for the batch EC process, in order to remove various kinds of pollutants (Yildiz 2008; Asadi Habib *et al.* 2012; Sadeghi *et al.* 2014; Ozyonar 2015; Taheri *et al.* 2015). However, to the best of our knowledge, the Taguchi method has not been used in the continuous EC process to evaluate the nitrate removal efficiency and its operating cost.

The main aim of this study is to apply the Taguchi method for the techno-economical evaluation of nitrate removal using the continuous EC process. For this purpose, four parameters including initial nitrate concentration, inlet flow rate, current density and initial pH were selected. The experiments were also carried out based on the Taguchi orthogonal array L27 (3^{13}) in three levels.

MATERIALS AND METHODS

EC apparatus and experimental procedure

A continuous flow pilot-scale EC reactor was constructed from Plexiglas with an effective volume of 2.4 L. Aluminum plate electrodes, with a total effective area of 364 cm^2, were connected in monopolar parallel mode to a digital DC power supply (Micro, PW4053R, 0–5 A, 0–40 V). The inter-electrode distance was fixed at 1 cm for all the experiments. A digital peristaltic pump (Heidolph, PD 5201, Germany) was used to provide the continuous flow in the system. Figure 1 illustrates the EC system used in the present study.

Nitrate solutions were prepared by dissolving $NaNO_3$ (Merck) in tap water with the initial nitrate concentration of 100–200 mg/L as NO_3^-. Sodium sulfate (Na_2SO_4) (Merck, 99%) was used as the supporting electrolyte in order to increase the conductivity of the solutions. The electro-conductivity of the solutions was recorded by an EC meter (Cond 340i, WTW, Germany). The initial pH of solutions was adjusted by H_2SO_4 (2 N) and NaOH (5 N), and pH values were measured using a pH meter (340i, WTW, Germany). All experiments were accomplished at ambient temperature. The concentration of nitrate in the effluent and the influent samples was analyzed by UV-Vis spectrophotometer (Hach, DR4000, USA) at a wavelength of 500 nm. The percentage of nitrate removal was calculated using Equation (4):

$$\text{Nitrate removal efficiency}(\%) = \left[\frac{(C_r - C_t)}{C_r}\right] \times 100 \quad (4)$$

where C_r and C_t are the nitrate concentration in raw and treated solutions, respectively.

Calculation of operating costs

Costs of electrical energy, electrode materials, chemicals, maintenance, and disposal of sludge constitute the operating costs in the EC process (Ghosh *et al.* 2008; Behbahani *et al.*

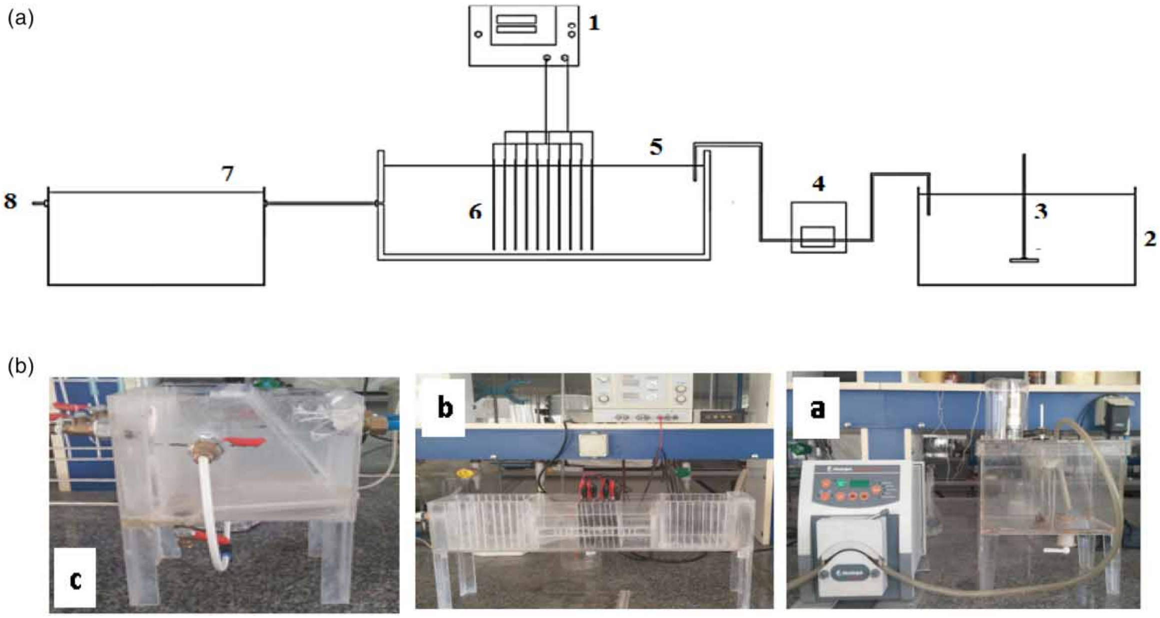

Figure 1 | Continuous monopolar EC system used in this study. (a) The schematic diagram: (1) DC power supply; (2) Reservoir tank; (3) Mechanical stirrer; (4) Peristaltic pump; (5) EC cell; (6) Aluminum electrodes; (7) Sedimentation tank and (8) Effluent. (b) The photographs: (a) Reservoir tank; (b) EC cell; (c) Sedimentation tank.

2011). In this research, an economic evaluation of electrical energy consumption, electrode material and chemical costs were calculated as the major costs in terms of operating costs using Equation (5):

$$\text{Operating costs} = aC_{energy} + bC_{electrodes} + cC_{chemicals} \quad (5)$$

where C_{energy} (kWh/ g NO_3-removed), $C_{electrodes}$ (kg Al/ g NO_3-removed) and $C_{chemicals}$ (g chemicals/ g NO_3-removed) are quantities consumed in the experiments on nitrate removal. The a, b and c are the coefficients of the Iranian market in 2016, as follows:

Coefficient a: industrial electrical energy price = 0.0222 US$kWh^{-1}.
Coefficient b: wholesale electrode material (Aluminum) price = 1.56 US$/kg Al.
Coefficient c: industrial Na_2SO_4 (as major chemicals) price in Iran = 18.867 US$/kg Na_2SO_4.

The energy consumption in the EC process is calculated by Equation (6):

$$E = \int_0^t V.I.dt = VIt \quad (6)$$

where *E* is the energy consumption (kWh), *V* is applied voltage (V), *I* is the current intensity (A) and *t* is the EC time (h) (Martinez-Villafane & Montero-Ocampo 2010; Behbahani *et al.* 2011; Hooshmandfar *et al.* 2016). The energy consumption of the pump was also calculated. Mechanical power for mixing purposes was neglected. In order to obtain the mass depletion of the electrodes, their weight difference was calculated in each experiment by subtracting their weights at the beginning and end of the experiments. The amount of the dissolved aluminum theoretically relies on current intensity and reaction time, which is calculated according to Faraday's law (Equation (7)) (Ghosh *et al.* 2008; Hooshmandfar *et al.* 2016):

$$m = \frac{ItM}{zF} \quad (7)$$

where *I* is the current (A), *t* is the reaction time, *M* is the molecular mass of aluminum ion (26.98 g/mol), *z* is the number of electrons transferred in the reaction ($z = 3$) and *F* is Faraday's constant (96,487 C/mol).

Experimental design and data analysis

The Taguchi method is based on four main steps, including: (1) to determine the factors and their levels, (2) to conduct

Table 1 | Levels of the independent parameters and experimental ranges from the Taguchi design (constant inter-electrode distance of 1 cm in monopolar parallel mode connection)

		Levels		
Parameter, unit	**Symbol**	**1**	**2**	**3**
Initial nitrate concentration, (mg/L)	A	100	150	200
Flow rate, (mL/min)	B	50	100	150
Current density, (A/m^2)	C	30	50	80
Initial pH	D	4	6	8

the designed experiments, (3) to analyze the data statistically and finally, (4) to validate the data (Roy 2001). The selected parameters and their levels are presented in Table 1. In the present study, the Taguchi orthogonal array L27 (3^{13}) was applied to analyze the impact of the selected parameters on nitrate removal efficiency and its operating costs.

According to the Taguchi method, the values of the experimental observations were transformed into signal-to-noise (S/N) ratios. The S/N ratio is a quality indicator which evaluates the effect of changing a particular

Table 2 | The observed and the predicted values of the conducted experiments corresponding to Taguchi design using continuous EC process

	Parameters				Nitrate removal efficiency (%)		Operating cost (US$/g NO_3-removed)	
Run	**A**	**B**	**C**	**D**	**Observed**	**Taguchi predicted**	**Observed**	**Taguchi predicted**
1	1	1	1	1	40.00	36.33	1.9723	2.1385
2	1	1	2	2	45.00	46.96	1.57181	1.4484
3	1	1	3	3	60.00	61.70	1.3209	1.2781
4	1	2	1	2	41.00	43.56	2.4637	2.6695
5	1	2	2	3	54.00	49.67	2.0538	1.6748
6	1	2	3	1	45.00	46.78	1.4820	1.6552
7	1	3	1	3	40.00	41.11	3.8252	3.4533
8	1	3	2	1	24.00	26.37	2.2877	2.7900
9	1	3	3	2	55.00	51.52	1.5348	1.4044
10	2	1	1	2	38.66	39.92	1.9000	1.8326
11	2	1	2	3	54.66	54.70	1.2894	1.0675
12	2	1	3	1	56.00	54.70	1.1213	1.4105
13	2	2	1	3	18.00	16.52	3.1323	3.7181
14	2	2	2	1	12.00	14.11	2.4055	2.5867
15	2	2	3	2	33.00	32.37	1.8167	1.0497
16	2	3	1	1	6.00	6.22	6.1697	5.6513
17	2	3	2	2	21.00	18.85	2.7800	2.8206
18	2	3	3	3	40.00	41.93	1.5322	2.0010
19	3	1	1	3	38.00	40.41	1.3889	1.2901
20	3	1	2	1	39.00	37	1.1506	1.4958
21	3	1	3	2	51.00	50.59	0.9128	0.6664
22	3	2	1	1	6.00	4.93	5.7376	4.9461
23	3	2	2	2	18.00	20.22	2.5571	2.749
24	3	2	3	3	30.00	28.85	1.5440	2.1378
25	3	3	1	2	20.00	18.67	2.2858	3.1761
26	3	3	2	3	26.00	25.78	1.9208	1.3778
27	3	3	3	1	25.00	26.56	1.7728	1.4255

parameter on process performance (Roy 2001). In this study, the S/N ratios of 'the larger is the better' and 'the smaller is the better' were used for the efficiency of the nitrate removal and the operating costs, respectively. Two performance characteristics were evaluated for 'the larger is the better' and 'the smaller is the better' by Equations (8) and (9), respectively (Roy 2001):

$$\frac{S}{N} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \quad (8)$$

$$\frac{S}{N} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}y_i^2\right) \quad (9)$$

where n and y_i are the number of observations and observed data, respectively. The impacts of the selected parameters were evaluated through analysis of variance (ANOVA) with 95% confidence level. The statistical software 'Minitab' (version 16) was also used to analyze the obtained data.

RESULTS AND DISCUSSION

Experimental results

The experiments were conducted based on the Taguchi orthogonal array matrix. In Table 2, the observed and predicted values of the conducted experiments are presented for the nitrate removal efficiencies and the operating costs. According to the data achieved by Minitab software, the coefficient of R^2 was 96.8% and 93.6% for nitrate removal efficiency and its operating costs, respectively. Further, high correlation between the experimental data and the predicted results indicates acceptable accuracy of the Taguchi model for both responses in the continuous EC process.

ANOVA and evaluation of the effects of the parameters

The ANOVA test for S/N ratios was also performed in order to calculate the statistical significance of the selected parameters for the nitrate removal efficiency and the operating costs. Table 3 presents the ANOVA results of S/N ratios. Generally, the F ratio and ρ (contribution ratio) indicate the importance of the factors in the EC process. According to Table 3, independent parameters

Table 3 | F, P, and ρ values from ANOVA for S/N ratios in the Taguchi design for the nitrate removal efficiency (%) and the operating cost (US$/g NO_3 removed) using continuous EC process

		Nitrate removal efficiency (%)			Operating cost (US$/g NO_3-removed)		
Source	**DF**	**F**	**P**	**ρ^a**	**F**	**P**	**ρ^a**
A	2	17.56	0.003	18.79	0.89	0.458	1.91
B	2	23.58	0.001	25.36	16.19	0.004	34.71
C	2	17.65	0.003	18.79	18.93	0.003	40.57
D	2	14.27	0.005	15.26	1.49	0.298	3.19
A × B	4	5.72	0.030	12.24	2.28	0.176	9.76
A × C	4	2.07	0.203	4.43	0.18	0.939	0.78
B × C	4	0.90	0.518	1.93	0.62	0.667	2.64
Error	6			3.2			6.44
Total	26			100			100

[a]ρ = contribution ratio.

including inlet flow rate, initial nitrate concentration, current density and initial pH had a significant effect on the nitrate removal efficiency with a contribution ratio of 25.36, 18.79, 18.79 and 15.26%, respectively. The contribution ratio of the current density, inlet flow rate, initial pH and initial nitrate concentration for the operating costs also reached 40.57, 34.71, 3.19 and 1.91%, respectively. Moreover, the results clearly indicate that the selected interactions had no significant effect on both the nitrate removal efficiency and its operating costs in this study.

Given the obtained results, the inlet flow rate was considered as the most important parameter for the nitrate removal efficiency when using the continuous EC process. The results are in agreement with those of batch EC, which indicates that a longer duration of reaction time leads to a high removal efficiency of nitrate (Koparal & Öğütveren 2002; Emamjomeh & Sivakumar 2009b; El-Shazly *et al.* 2011). Also, it was found that the current density and the flow rate were significant parameters for the operating costs of the EC process. However, initial pH and initial nitrate concentration had no significant effect on operating costs in the present study. The findings of the current study do not support the results of the study carried out by Nazlabadi & Alavi Moghaddam (2014), who found that the initial nitrate concentration (300–500 mg/L) and initial pH were significant parameters for the operating costs of the batch EC process.

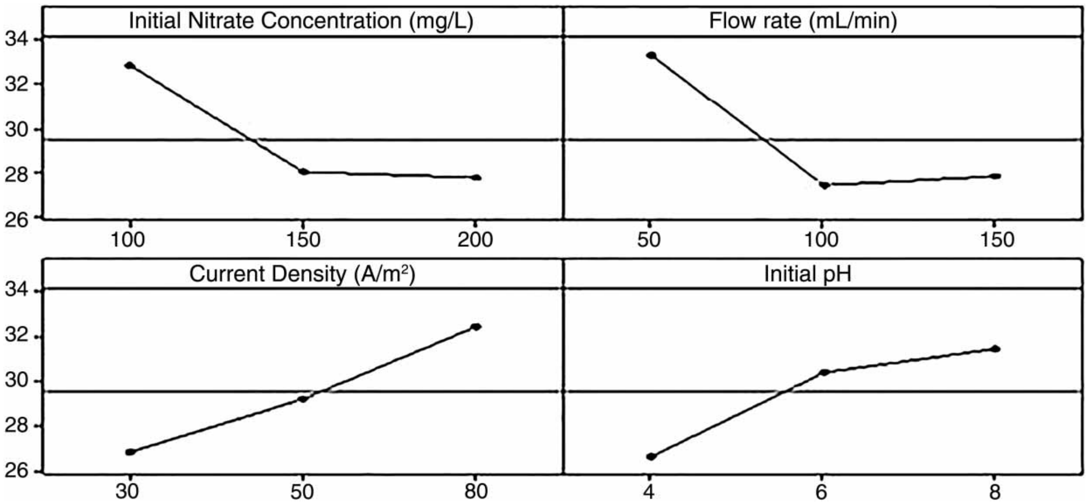

Figure 2 | The main effects of S/N ratios plots for the nitrate removal efficiency (%) in the Taguchi method using the continuous EC process (Signal-to-noise: Larger is better).

The main effects of the parameters for the nitrate removal efficiency and the operating costs using the Taguchi method are illustrated in Figures 2 and 3, respectively. As shown in Figure 2, nitrate removal efficiency was improved by increasing the current density, which is consistent with the results of other researchers indicating that an increase in current intensity results in improving the removal efficiency (El-Shazly *et al.* 2011).

The maximum nitrate removal efficiency was achieved with the flow rate, the initial nitrate concentration and the initial pH of 50 mL/min, 100 ppm and 8, respectively. Based on the results, the removal efficiency could improve in the highest initial pH, which could confirm other researchers' results (Emamjomeh & Sivakumar 2009b; Yehya *et al.* 2014). Regarding the operating cost, as illustrated in Figure 3, an increase in the current density led to an increase in the operating cost. Similar results were also reported by Nazlabadi & Alavi Moghaddam (2014).

As shown in Figure 3, the operating costs are highly dependent on the current density and the flow rate values. As is well known, the flow rate determines the reaction time, thus an increase in the current density and the flow rate leads to higher energy consumption (Equation (6)) and electrode corrosion (Equation (7)). Further, reducing the

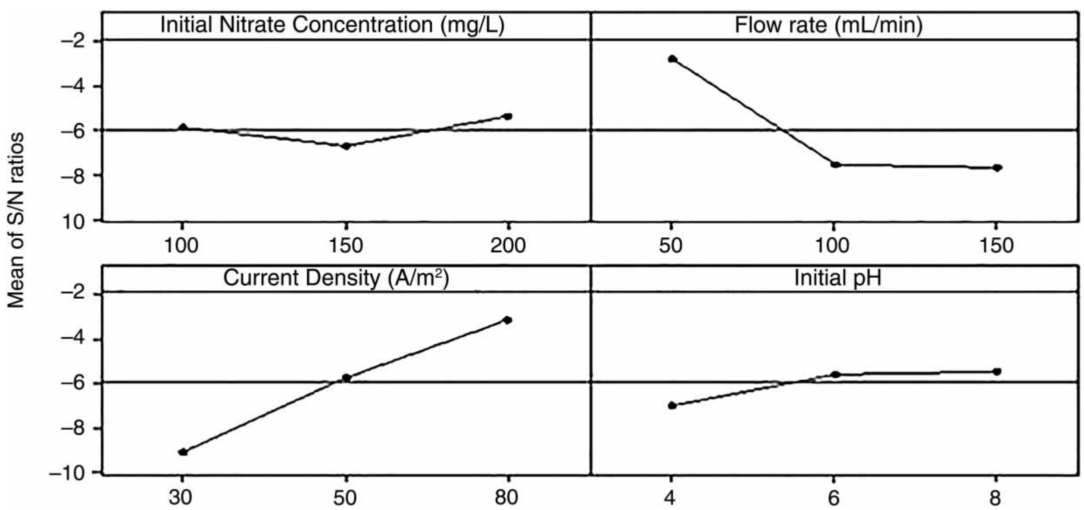

Figure 3 | The main effects of S/N ratios plots for the operating cost (US$/g NO_3- removed) in the Taguchi method using the continuous EC process (Signal-to-noise: Smaller is better).

energy consumption and electrode corrosion will make the continuous EC process more affordable in terms of operating costs.

The interaction plots for the nitrate removal efficiency and its operating costs are shown in Figure 4. As is evident, the interaction between initial concentration and the flow rate is the most important interaction with regard to the nitrate removal efficiency and the operating cost.

The higher S/N ratios indicate the highest removal efficiency. Therefore, the optimum conditions achieved by the Taguchi model for initial nitrate concentration, flow rate, current density and initial pH were: 100 mg/L,

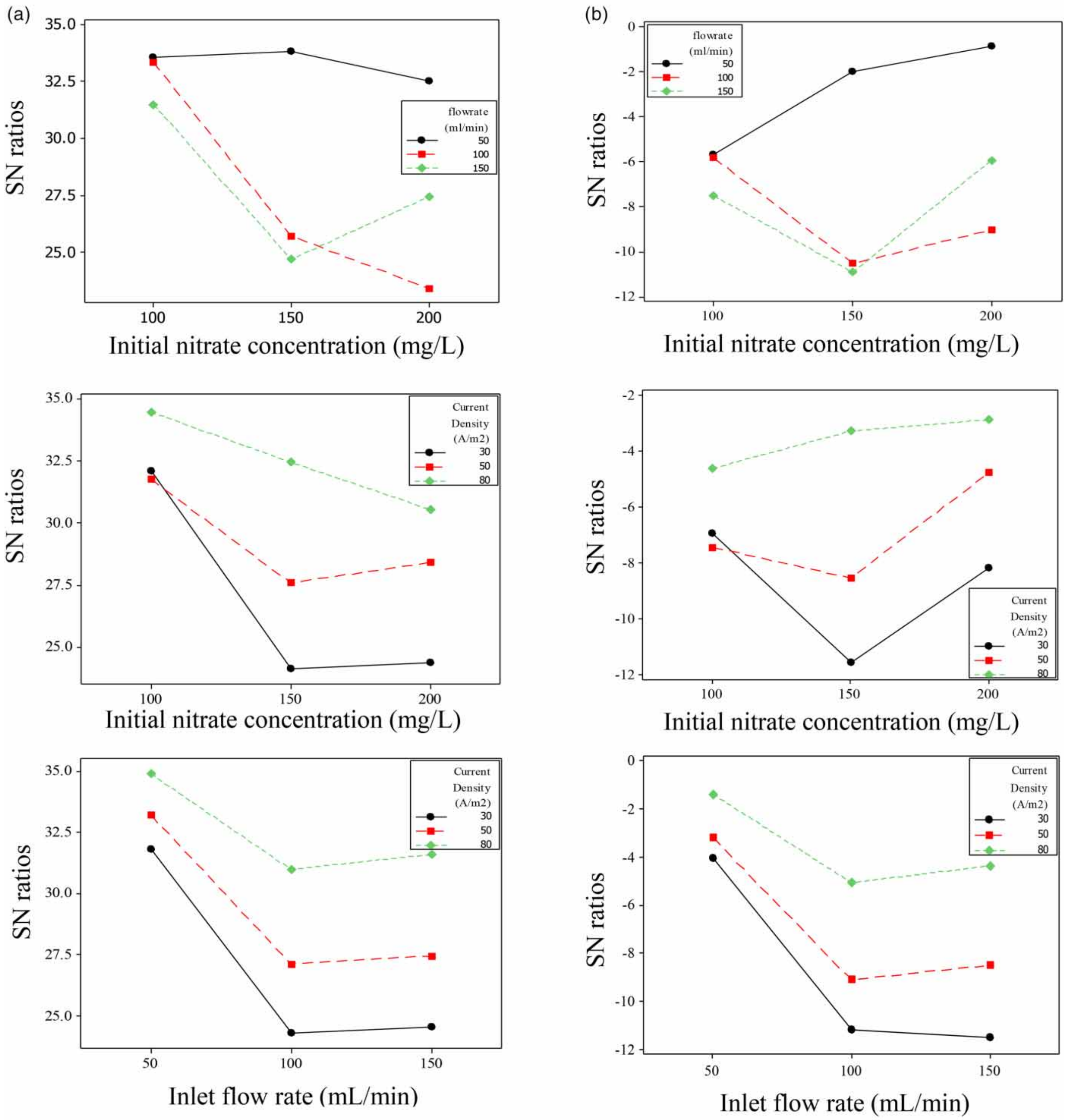

Figure 4 | The interaction plots for (a) nitrate removal efficiencies (%) and (b) operating costs (US$/g NO_3-removed) in the Taguchi method using the continuous EC process.

50 mL/min, 80 A/m^2 and 8, respectively. Under these conditions, the efficiency of nitrate removal and the estimated operating costs achieved were 61.70% and 1.2781 US$/g NO_3-removed, respectively. Altogether, a detention time of about 60 minutes is essential to reach a nitrate concentration lower than the guideline value of 50 mg/L NO_3 (ISIRI 1996; WHO 2013) using a current intensity of 2.91 Amperes for an initial nitrate concentration of 100 mg/L.

CONCLUSIONS

In the present study, the Taguchi orthogonal array L27 (3^{13}) was employed as an experimental design tool to evaluate the effect of the main parameters on the removal of nitrate and its operating costs, through using a continuous EC process. For this purpose, four main parameters including initial nitrate concentration, inlet flow rate, current density and initial pH were selected. The ANOVA results revealed that the first three of the above-mentioned parameters (with a contribution ratio of 25.36, 18.79 and 18.79%, respectively), could markedly influence the nitrate removal efficiency, while the current density (40.57%) and the flow rate (34.71%) were regarded as significant parameters for the operating costs. Based on the optimum operating conditions, suggested by the Taguchi model, 61.70% nitrate removal with an operating cost of 1.278 US$/g NO_3-removed was achieved at initial concentration, flow rate, current density and initial pH of 100 mg/L, 50 mL/min, 80 A/m^2 and 8, respectively. Regarding the nitrate removal efficiency and the operating cost terms, the coefficients of R^2 were found to be 96.8% and 93.6%, respectively. The high correlation between the experimental and predicted results proved that the Taguchi model was a powerful tool to evaluate the continuous EC process for nitrate removal efficiency and its operating costs.

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