Chapter 4
Simulation of Nitrate Removal in a Batch Flow Electrocoagulation-Flotation (ECF) Process by Response Surface Method (RSM)

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1 Introduction

Electrocoagulation-flotation (ECF) is one of the newest treatment methods, which has been used successfully to remove different kinds of pollutants (Behbahani et al. 2013). ECF is a process which consists of three main parts: (1) Creating metallic hydroxide flocs within the solution by electro-dissolution of soluble anodes, (2) Formation of coagulants in aqueous phase, and (3) Adsorption of pollutants on coagulants and then removal by sedimentation/flotation (Arroyo et al. 2009; Zaroual et al. 2009).

Nitrate is a stable and highly soluble ion with low potential for co-precipitation or adsorption (El-Shazly et al. 2011). Nitrate concentration is naturally a few milligrams in litre for groundwater, but different factors like inappropriate sewage treatment/disposal, unsuitable agricultural/stockbreeding activities, and the geological structures of each region, makes this concentration to grow up. High levels of nitrate can cause severe health problems (Moghaddasi et al. 2008; Azadegan et al. 2012; Nazlabadi and Alavi Moghaddam 2014). Conventional methods of removing nitrate include biological decomposition, ion exchange, chemical treatment, reverse osmosis and membrane separation techniques (El-Shazly et al. 2011; Lakshmi et al. 2012).

In recent years, ECF as a chemical treatment method has been focused on by a large number of researchers for removal of nitrate due to its high treatment efficiency, low sludge production, easy operation and relatively low capital cost. In particular, electrocoagulation has demonstrated an attractive alternative to the other traditional methods for treating nitrate contaminated water (Li et al. 2009;

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Emamjomeh and Sivakumar 2009; Kumar and Goel 2010; Kasiri and Khataee 2011; Lakshmi et al. 2012). However, the process is limited in practice due to formation of by-products like nitrite during treatment (Lakshmi et al. 2012; Nazlabadi and Alavi Moghaddam 2014).

The efficiency of ECF process is influenced by various factors such as initial pH, initial nitrate concentration, applied current, number of electrodes and reaction time. Simulation of these factors can be useful in order to achieve better nitrate removal efficiency. As it is well known, some limitations of classical study methods such as time consuming and high cost can be eliminated by statistical experimental design such as response surface methodology (RSM) (Zodi et al. 2010; Behbahani et al. 2013). RSM is only applicable for variables such as initial pH which is defined by a range of numbers and this technique is not useful for parameters like electrode material that are not numerical. Many research groups applied this method for removal of different pollutants by ECF (Aleboyeh et al. 2008; Krishna Prasad et al. 2008; Chavalparit and Ongwandee 2009; Sadri Moghaddam et al. 2010; Behbahani et al. 2011; Behbahani et al. 2013; Taheri et al. 2013; Radaei et al. 2014). However, to the best of our knowledge, RSM rarely has been used for nitrate removal (Koporal and Ogutveren 2002; Emamjomeh and Sivakumar 2009; Kumar and Goel 2010; Vasudevan et al. 2010; El-Shazly et al. 2011; Lacasa et al. 2011; Malakootian et al. 2011; Lakshmi et al. 2012; Sim et al. 2012).

The main objective of the present study is to simulate nitrate removal efficiency and remained nitrite as responses using an ECF unit operating in batch regime. For simulating this process, the relation between the responses and five quantitative variables (initial pH, initial nitrate concentration, reaction time, number of electrodes, applied current) is determined by a second order polynomial model.

2 Materials and Methods

2.1 ECF Reactor

A batch flow ECF reactor was made in the lab from Plexiglas with dimensions of 50 cm × 10 cm × 9 cm. Aluminum plate electrodes with the effective area of 42 cm^2 and thickness of 1 mm were used in this research. Inter-electrode distance was maintained at 10 mm and electrodes were connected to a DC power supply (Micro, PW4053R, 0–5 A, 0–40 V) in bipolar mode. Two hotplate magnetic stirrers (Labtech Hotplate Stirrer, LMS-1003, Korea) was applied for preparing complete mixed solutions in the EC reactor. The EC reactor used in this study is shown in Fig. 4.1.

Fig. 4.1 Photograph of the ECF's set-up

2.2 Experimental Procedure

Coagulation, flocculation, settling and flotation were taking place within the ECF reactor. All the experiments were carried out at room temperature. Nitrate solutions were prepared synthetically by dissolving proper amounts of $NaNO_3$ (Merck, solubility 874 g/l) in the range of 300–500 mg/lit and Na_2SO_4 (Merck, 99 %) as supporting electrolyte in 3.7 L of distilled water. The amounts of Na_2SO_4 added in each experiment are depending on the applied currents. The initial pH of the solution was adjusted before the experiment by H_2SO_4 and NaOH, and pH values were measured using pH meter (340i, WTW, Germany). All effluent samples for nitrate and some of them for nitrite were analyzed using a UV–vis spectrophotometer (DR/4000, HACH, USA) by method of 8039 (nitrate) and 8507 (nitrite). Percentage of nitrate removal was calculated by Eq. (4.1):

$$\text{Nitrate removal efficiency } (\%) = (C_r - C_t) \times 100/C_r \quad (4.1)$$

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

2.3 Experimental Design and Data Analysis

RSM is a well-known up-to-date approach for developing approximation models based on either physical experiments or computer experiments (simulations) with minimum number of experiments, as well as analyzing the interactions between selected parameters (Hameed et al. 2009; Raissi and Eslami Farsani 2009). The most widely used class of second-order designs called central composite design (CCD) was applied for the RSM (Nazlabadi and Alavi Moghaddam 2014). In the present study, the CCD was selected for experimental design of the removal efficiency of nitrate and remained nitrite. Five factors, including initial pH, initial nitrate concentration, current, electrode number and reaction time with five-levels were employed for response surface modelling in the ECF process. A total of 57 experiments were carried out according to a 2^5 full factorial CCD, consisting of 32 factorial experiments, 10 axial experiments on the axis at a distance of $\pm\alpha$ from the centre, and 15 replicates at the centre of the experimental domain. The value of α for rotatability depends on the number of points in the factorial portion of the design, which is given in Eq. (4.2):

$$\alpha = (N_F)^{\frac{1}{4}} \tag{4.2}$$

where N_F is the number of points in the cube portion of the design ($N_F = 2^k$, k is the number of factors) (Behbahani et al. 2011). Therefore, α is equal to $(2^5)^{1/4} = 2.4$ according to Eq. (4.2).

The statistical software "Minitab", version 16.1.0 was also used for CCD and developing a simulation model. Several experiments were initiated as a preliminary study for determining the range of parameters prior to designing the experimental runs. Five-level factors were used to build models as shown in Table 4.1. In the case of remained nitrite, among the 57 run, the 22 critical runs were selected. The critical runs include: minimum level (five runs) and maximum level (five runs) of each factor, one factor in minimum level and other in maximum (five runs), one factor in maximum level and other in minimum (five runs), and two of replicate runs.

Nitrate removal efficiency and remained nitrite of the ECF process were taken as the responses of the experiments (Y_i) according to Eq. (4.3):

$$Y_i = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} b_{ij} x_i x_j, \tag{4.3}$$

where Y_i is the response, b_0, b_i, b_{ii}, b_{ij} are the constant coefficient, the linear coefficients, the quadratic coefficients and the interaction coefficients, respectively, and x_i and x_j are the coded values of the variables.

Table 4.1 Experimental range and levels of independent variables according to RSM design

Variables	Factor	Unit	Levels				
			–α	−1	0	+1	+α
Initial pH	X_1	–	1.9	4	5.5	7	9.1
Applied current	X_2	Ampere	0.95	2	2.75	3.5	4.55
Initial concentration of nitrate	X_3	mg/L as NO_3^-	160	300	400	500	640
Electrode number	X_4	–	5	8	10	12	15
Reaction time	X_5	min	61	110	145	180	229

3 Results and Discussion

3.1 Development of Regression Model Equation

In order to study the effect of selected variables, experiments were performed for different combinations of variables using statistically designed experiments. The P and the F values for nitrate removal efficiency and remained nitrite are listed in Tables 4.2 and 4.3, respectively.

The second order polynomial equations for nitrate removal efficiency (Y_1) and remained nitrite (Y_2) in terms of coded factors are given by Eqs. (4.4) and (4.5), respectively.

Table 4.2 Analysis of variance for nitrate removal efficiency

Source	df	F value	p-value Prob > F
Model	24	49.72	<0.0001
X_1-pH	1	19.23	0.0002
X_2-Current	1	2.98	0.0974
X_3-Ini. Conc.	1	328.50	<0.0001
X_4-Elec.	1	1.67	0.2089
X_5-Time	1	160.08	<0.0001
X_1X_2	1	6.24	0.0198
X_1X_3	1	25.67	<0.0001
X_1X_4	1	2.19	0.1517
X_1X_5	1	66.98	<0.0001
X_2X_3	1	60.56	<0.0001
X_2X_4	1	6.24	0.0197
X_2X_5	1	6.85	0.0151
X_3X_4	1	1.68	0.2076
X_3X_5	1	19.40	0.0002
X_1^2	1	0.51	0.4816
X_2^2	1	25.55	<0.0001
X_3^2	1	3.02	0.0949
X_5^2	1	34.20	<0.0001
$X_1X_2X_3$	1	1.84	0.1877
$X_1X_2X_4$	1	15.44	0.0006
$X_1X_2X_5$	1	13.29	0.0013
$X_1X_3X_5$	1	36.37	<0.0001
$X_1^2X_2$	1	43.87	<0.0001
$X_1^2X_5$	1	74.27	<0.0001
Residual	24		
Lack of fit	10	0.99	0.50
Pure error	14		

Table 4.3 Analysis of variance for remained nitrite

Source	df	F value	p-value Prob > F
Model	13	40.34	0.0004
X_1-pH	1	7.54	0.0405
X_2-Current	1	58.20	0.0006
X_3-Ini. Conc.	1	260.72	<0.0001
X_4-Elec.	1	9.92	0.0254
X_5-Time	1	4.07	0.0998
X_1X_2	1	1.76	0.2416
X_1X_3	1	1.25	0.3141
X_2X_3	1	29.16	0.0029
${X_1}^2$	1	2.93	0.1474
${X_2}^2$	1	12.72	0.0161
${X_3}^2$	1	39.96	0.0015
${X_4}^2$	1	3.12	0.1377
${X_5}^2$	1	0.06	0.8118
Residual	5		
Lack of fit	4	–	–
Pure error	1		

$$\begin{aligned}Y_1 = {} & 76.24 + 3.28\,X_1 + 1.49\,X_2 - 10.27\,X_3 + 0.68\,X_4 + 10.96\,X_5 \\ & + 1.67\,X_1X_2 + 3.81\,X_1X_3 + 0.96\,X_1X_4 + 6.15\,X_1X_5 + 5.95\,X_2X_3 \\ & + 1.60\,X_2X_4 + 2.00\,X_2X_5 - 0.87\,X_3X_4 - 3.18\,X_3X_5 - 0.40\,X_1^2 \\ & + 1.87\,X_2^2 - 0.64\,X_3^2 - 2.16\,X_5^2 + 1.06X_1X_2X_3 - 2.52\,X_1X_2X_4 \\ & - 2.84\,X_1X_2X_5 + 4.54\,X_1X_3X_5 + 7.20\,X_1^2X_2 - 9.62\,X_1^2X_5 \end{aligned} \tag{4.4}$$

$$\begin{aligned}Y_2 = {} & 12.33 + 1.99\,X_1 - 9.9\,X_2 + 11.68\,X_3 - 2.28\,X_4 - 2.61\,X_5 + 1.7\,X_1X_2 - \\ & 1.52\,X_1X_3 - 6.92\,X_2X_3 - 0.91\,X_1^2 + 2.84\,X_2^2 + 3.36\,X_3^2 + 0.94\,X_4^2 + 0.22\,X_5^2 \end{aligned} \tag{4.5}$$

Table 4.4 presents the observed nitrate removal efficiency and remained nitrite for the 57 and 22 experiments, respectively.

The most important parameters, which affect the nitrate removal efficiency are initial pH, initial nitrate concentration and time. Moreover it was found that square terms of current and time and interaction terms except X_1X_4 and X_3X_4 were significant to the response. Triple interaction terms of $X_1X_2X_4$, $X_1X_2X_5$, $X_1X_3X_5$ and $X_2X_3X_5$ were also significant to the response. In case of remained nitrite, initial nitrate concentration and current are the most effective parameters. Also interaction and square terms except of X_2X_3, ${X_2}^2$ and ${X_3}^2$, respectively, have negligible effect.

Table 4.5 shows the coefficient of determination (R^2) for nitrate removal efficiency and remained nitrite. High R^2 values of 98 % and 99 % for nitrate removal efficiency and remained nitrite, respectively, express a high correlation between the observed and predicted values. Also the "Predicted R^2" is in reasonable agreement with the "Adjusted R^2" in both response.

Table 4.4 RSM design and its observed values for nitrate removal efficiency and remained nitrite

Std	Run	Initial pH	Applied current (Ampere)	Initial concentration of nitrate (mg/L)	Electrode number	Reaction time (min)	Nitrate removal efficiency (%)	Remained nitrite (mg/L)
7	1	4.00	3.50	500	8	110.00	72.98	–
55	2	5.50	2.75	400	10	145.00	73.43	12.6
13	3	4.00	2.00	500	12	110.00	54.82	–
17	4	4.00	2.00	300	8	180.00	83.02	10.18
48	5	5.50	2.75	400	10	145.00	82.16	12.6
8	6	7.00	3.50	500	8	110.00	80.07	–
44	7	5.50	2.75	400	10	145.00	67.89	–
12	8	7.00	3.50	300	12	110.00	83.76	–
36	9	5.50	4.55	400	10	145.00	90.56	5.68
6	10	7.00	2.00	500	8	110.00	44.20	–
45	11	5.50	2.75	400	10	145.00	78.96	–
24	12	7.00	3.50	500	8	180.00	89.37	10.77
37	13	5.50	2.75	160	10	145.00	97.23	2.67
1	14	4.00	2.00	300	8	110.00	76.38	–
43	15	5.50	2.75	400	10	145.00	76.61	–
56	16	5.50	2.75	400	10	145.00	76.61	–
2	17	7.00	2.00	300	8	110.00	83.76	15.32
57	18	5.50	2.75	400	10	145.00	76.61	–
34	19	9.10	2.75	400	10	145.00	81.73	13.78
32	20	7.00	3.50	500	12	180.00	96.90	–
14	21	7.00	2.00	500	12	110.00	45.08	–
26	22	7.00	2.00	300	12	180.00	91.88	–
18	23	7.00	2.00	300	8	180.00	84.50	–
40	24	5.50	2.75	400	15	145.00	78.41	11.38

(continued)

Table 4.4 (continued)

Std	Run	Initial pH	Applied current (Ampere)	Initial concentration of nitrate (mg/L)	Electrode number	Reaction time (min)	Nitrate removal efficiency (%)	Remained nitrite (mg/L)
46	25	5.50	2.75	400	10	145.00	76.61	–
11	26	4.00	3.50	300	12	110.00	86.71	–
47	27	5.50	2.75	400	10	145.00	76.61	–
19	28	4.00	3.50	300	8	180.00	94.83	–
21	29	4.00	2.00	500	8	180.00	33.57	–
4	30	7.00	3.50	300	8	110.00	81.54	–
50	31	5.50	2.75	400	10	145.00	76.61	–
15	32	4.00	3.50	500	12	110.00	81.40	–
41	33	5.50	2.75	400	10	61.00	37.44	41.82
51	34	5.50	2.75	400	10	145.00	76.61	–
53	35	5.50	2.75	400	10	145.00	76.61	–
30	36	7.00	2.00	500	12	180.00	65.90	40.10
39	37	5.50	2.75	400	5	145.00	72.32	24.8
16	38	7.00	3.50	500	12	110.00	82.28	17.34
31	39	4.00	3.50	500	12	180.00	88.48	6.98
22	40	7.00	2.00	500	8	180.00	90.69	–
25	41	4.00	2.00	300	12	180.00	80.20	–
49	42	5.50	2.75	400	10	145.00	76.61	–
29	43	4.00	2.00	500	12	180.00	75.64	–
33	44	1.90	2.75	400	10	145.00	5.89	1.11
9	45	4.00	2.00	300	12	110.00	86.71	8.81
38	46	5.50	2.75	640	10	145.00	47.76	61.39
20	47	7.00	3.50	300	8	180.00	93.35	–
27	48	4.00	3.50	300	12	180.00	96.31	–
54	49	5.50	2.75	400	10	145.00	76.61	–

42	50	5.50	2.75	400	10	229.00	90.03	8.02
10	51	7.00	2.00	300	12	110.00	77.85	–
23	52	4.00	3.50	500	8	180.00	58.81	–
5	53	4.00	2.00	500	8	110.00	71.21	52.22
35	54	5.50	0.95	400	10	145.00	83.39	9.72
52	55	5.50	2.75	400	10	145.00	76.61	–
28	56	7.00	3.50	300	12	180.00	93.35	4.57
3	57	4.00	3.50	300	8	110.00	73.42	5.09

Table 4.5 Coefficient of determination (R^2) for nitrate removal efficiency and remained nitrite

	Value	
Parameter	Nitrate removal efficiency	Remained nitrite
R^2	0.98	0.99
Adjusted-R^2	0.96	0.96
Predicted-R^2	0.89	0.82

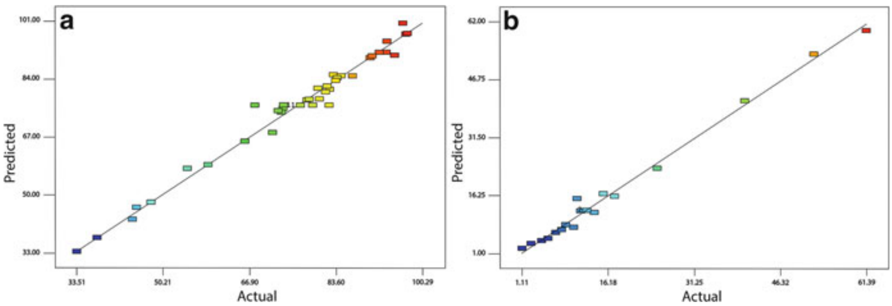

Fig. 4.2 Actual versus predicted values: (**a**) nitrate removal efficiency and (**b**) remained nitrite

Figure 4.2a, b illustrates the actual versus predicted values for nitrate removal efficiency and remained nitrite, respectively. The figure indicates a good agreement between the observed and predicted values.

4 Conclusions

In the present study, the effects of five main parameters including initial pH, initial nitrate concentration, applied current, number of electrodes and reaction time on nitrate removal efficiency by ECF, as a response, were investigated using RSM. Also, due to the formation of by-products like nitrite during treatment process, remained nitrite was also considered as a second response. According to the ANOVA results, the model indicated high R-squared value of 98 % and 99 % for nitrate removal efficiency and remained nitrite, respectively. The predicted R-squared of 89 % is in reasonable agreement with the adjusted R-squared of 96 % for removal efficiency of nitrate. Similarly, predicted R-squared of 96 % is in reasonable agreement with adjusted R-squared of 82 % for remained nitrite. Therefore the applied model showed an acceptable accuracy. In addition, it can be concluded that ECF is a very efficient technology for treatment of nitrate wastewaters and RSM is a powerful tool for simulation of nitrate removal efficiency and remained nitrite by ECF process.

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