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Optimization of Nitrate Removal by Electro-Reduction Using a Ti/RuO_2 + IrO_2 Electrode: Process Modeling by Response Surface Methodology

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Abstract

Agricultural activities and the excessive use of fertilizers significantly contribute to nitrate contamination in the environment. These ions can cause a serious threat to both human health and ecosystems through the contamination of surface water and groundwater. It is therefore necessary to reduce their concentration before consumption. The general objective of this work is to propose new electrochemical treatment methods based on the Design of Experiments Methodology. Indeed, this approach allows to reduce the need for costly and lengthy experimental trials. Various parameters that could improve the efficiency of the process were studied, including initial concentration, pH, electrolysis time, and current intensity, in order to determine the optimal operating conditions. The results show that the process is effective at a reduction current intensity of −603 mA, a $NaNO_3$ concentration of 100 mg-N/L, and an electrolysis time of 10 min at a slightly basic pH, allowing a nitrate removal efficiency of 91%. The results highlight the importance of statistical modeling in improving and controlling electrochemical processes.

Keywords

Electrochemical Reduction, Water Treatment, Nitrate, Optimization, Modelling

1. Introduction

Water is a fundamental and essential resource for all living things life [1]. How-

ever, the extensive use of fertilizers in agricultural development can lead to the pollution of water resources by chemicals products through their infiltrating into water table [2] [3]. Groundwater contamination by nitrate ions, resulting from intensive agricultural practices, can compromise drinking water quality [4]. Nitrate becomes particulary toxic when reduced to nitrite; which can react with hemoglobin in the blood to form methemoglobin, thereby constituting a serious health risk, especially for children [5]. In Senegal, several studies carried out in agricultural regions have reported excessive nitrate concentration in groundwater [4] [6].

Consequently, the major challenge is to develop an effective and low-cost technique for a removal or reduction of nitrate concentrations in groundwater in order to preserve water quality [7]-[10]. In this context, various treatment methods have been investigated including coagulation/flocculation [11], membrane processes [12]-[16], chemical reduction [17] [18] and biological treatments [19]. Among these, electrochemical methods have emerged as one of the most promising approaches, offering ecological, technological, and economic advantages [20]-[23]. This method are gaining popularity due to transform nitrates into less harmful compounds such as nitrogen gas or ammonium [24].

Recently, our research group reported on the electrochemical denitrification using $Ti/RuO_2 + IrO_2$ electrodes [25]. The authors highlighted the importance of optimizing parameters such as time, current intensity, initial concentration, and pH. Therefore, the primary objective of the present study is to investigate the process through modeling based on Design of Experiments Methodology. This approach enables the evaluation of the individual effects of each factor as well as their potential interactions.

2. Materials and Methods

2.1. Chemicals and Apparatuses

All electrochemical manipulations were performed using $Ti/RuO_2 + IrO_2$ electrode. Electrolyses were carried out in a standard two-electrode cell, with a Voltalab 40 potentiostat, connected to an interfaced computer that employed Voltamaster 4 software. A stock solution, with a concentration of 2 g/L, was prepared by dissolving $NaNO_3$ solid (>99%, Sigma-Aldrich) in distilled water. The working solutions were subsequently obtained by successive dilutions to the desired concentrations. Nitrate concentrations (NO_3^-) before and after electrolysis were determined by UV-visible absorption spectroscopy using a Varian UV-vis spectrophotometer Cary-60. The pH of the solutions was adjusted before electrolysis by adding NaOH or HCl solutions and measured using a HI 2211 Ph/ORP Meter pH-meter. Nitrate ion concentrations were determined according to the NFT 90-012 standard method [26]. Sodium salicylate (>99.5%, Sigma-Aldrich), Sodium azide (>99.5%, Sigma-Aldrich) and EDTA disodium salt (Sigma-Aldrich) were used as received.

2.2. Methods

First, an UV-Vis calibration curve was established and subsequently used to determine the nitrate concentration in all solutions before and after electrolysis. For this purpose, four sodium nitrate solutions were prepared at concentrations of 0.5, 1.0, 2.5, and 5.0 mg/L. If the concentration is outside this range, the sample is diluted. Nitrate removal efficiency was obtained by Equation (1) [27]:

$$R = \frac{\left(C_i - C_f\right)}{C_i} \times 100 \tag{1}$$

where C_i and C_f are the initial and final concentration (mg-N/L) of NO_3^-.

Table 1 summarizes the experimental factors to be optimized and modeled using response surface methodology with Design-Expert software (version 13). The response surface methodology was used to evaluate the effects of the independent variables on the response. The experimental factors considered were electrolysis time (A), initial concentration (B), electrolysis current (C), and pH (D), while the response variable was the nitrate removal efficiency. Table 1 presents the coded levels assigned to each factor.

Table 1. Process factors and their levels.

Factors	Unit	Coded variables	Levels	
			Min	Max
Electrolysis time	min	A	10	120
NO_3^- concentration	mg-N/L	B	100	700
Reduction current	mA	C	−700	−100
pH		D	2	12

The matrix consisting of 4 factors, namely electrolysis time, NO_3^- concentration, reduction current and pH, is used to draw up a plan of experiments comprising 30 tests including 6 in the center.

The number of experiments N to be performed is given by the following relationship [28]:

$$N = 2k\left(k-1\right) + r \tag{2}$$

where r is the number of replicates in the centre and k is the number of factors. Thus, the mathematical equation below allowed the calculation of the response according to the factors:

$$Y = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} \left(x_i\right)^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} b_{ij} x_i x_j \tag{3}$$

In this model, Y represents the predicted response (degree of conversion); b_0 is the constant term; b_i are the linear coefficients; b_{ij} are the interaction coefficients; and b_{ii} are the quadratic coefficients, while x_i and x_j denote the coded values of the operating parameters.

The analysis of the different models tested (linear, two-factor interaction (2FI), quadratic, and cubic) indicates that the quadratic model is the most suitable for predicting the response. This is supported by its high statistical significance (p-value = 0.0001 < 0.05) and strong correlation coefficient (R^2 = 0.9551), along with a satisfactory predicted R^2 value of 0.8664.

Therefore, the quadratic model was selected by Design-Expert 13 to describe the relationship between the studied parameters and the response.

3. Results and Discussion

3.1. Diagnostic Model

The comparison between the predicted values (Y_pred) and the experimental values (Y_exp) is presented in Figure 1. The results indicate a good agreement between the model predictions and the experimental response.

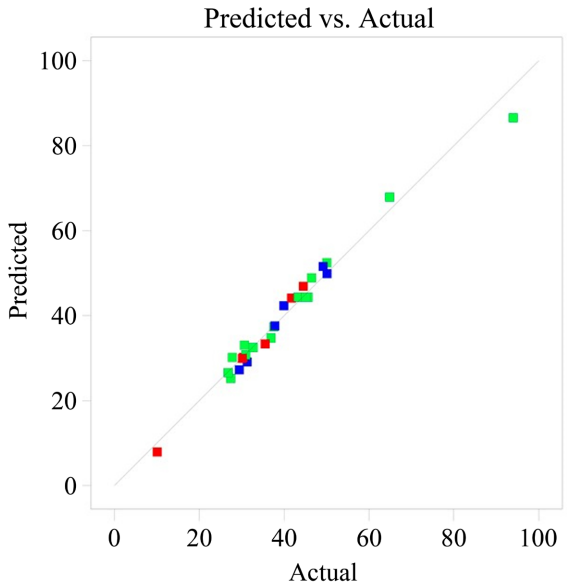

Figure 1. Experimental and predicted nitrate removal responses.

3.2. Analyse of Variance

Analysis of variance (ANOVA) was performed using Design-Expert software [29] [30]. The Desirability Function Approach (DFA) was applied to determine the optimal operating conditions for nitrate electroreduction within the selected experimental domain. ANOVA was used to assess the significance of the model and the curvature of the response at a 95% confidence level. The significance of the process variables was evaluated based on the F-value and P-value [31] [32] with differences considered statistically significant when the P-value was less than 0.05. The nitrate removal efficiency was evaluated according to the experimental design matrix, and the corresponding results are presented in Table 2.

As indicated in Table 2, the quadratic model for nitrate removal efficiency were statistically significant ($p \leq 0.0001$) [33] [34]. The results showed that only the

interaction term of BD was not significant for nitrate removal efficiency. After the removal of this term, the modified quadratic model for nitrate removal efficiency was obtained as presented in Equation (4):

$$\begin{aligned} \textit{Nitrate removal efficiency} = {} & 59.09430 - 0.607922A - 0.133785B \\ & - 0.129728C + 3.02922D + 0.001432AB \\ & + 0.000210AC - 0.022100AD + 0.000042BC \\ & - 0.004548CD + 0.001783A^2 + 0.000043B^2 \\ & - 0.000139C^2 - 0.296623D^2 \end{aligned} \quad (4)$$

In this final fitted quadratic equation, A, B, C and D represent the time, the NO_3^- concentration; the reduction current; and the pH, respectively; AB, AC, AD, BC and CD are the interaction terms; and A^2, B^2, C^2, and D^2 are the square terms.

Table 2. Analysis of variance (ANOVA) for the quadratic response surface model.

Source	Squares	DF	Mean Square	F-value	p-value	Remarks
Model	5877.73	14	419.84	44	<0.0001	Significant
A	63.19	1	63.19	6.62	0.0212	
B	595.67	1	595.67	62.43	<0.0001	
C	417.13	1	417.13	43.71	<0.0001	
D	171.16	1	171.16	17.94	0.0007	
AB	2234.16	1	2234.16	234.14	<0.0001	
AC	47.89	1	47.89	5.02	0.0406	
AD	147.74	1	147.74	15.48	0.0013	
BC	58.22	1	58.22	6.1	0.026	
BD	0.0121	1	0.0121	0.0013	0.9721	
CD	186.19	1	186.19	19.51	0.0005	
A^2	199.58	1	199.58	20.92	0.0004	
B^2	102.76	1	102.76	10.77	0.005	
C^2	1075.6	1	1075.6	112.72	<0.0001	
D^2	377.08	1	377.08	39.52	<0.0001	
Residual	143.13	15	9.54			
Lack of Fit	138.64	10	13.86	15.44	0.0037	
Pure Error	4.49	5	0.8978			
Cor Total	6020.86	29				

DF: Degree of freedom.

3.3. Optimization of Physico-Chemical Parameters

The experimental results obtained at room temperature (27˚C) are presented in Table 3. This design is based on the Box-Behnken response surface methodology.

The optimization results indicate that the most favorable conditions for nitrate removal are achieved through a specific combination of the four studied factors.

Table 3. Nitrate removal efficiency of electrolysis as a function of four parameters.

Time (min)	Ci (mg-N/L)	Current (mA)	pH	R (%)
65	100	−700	7	50.07
65	700	−700	7	30.96
65	100	−100	7	30.65
65	700	−100	7	26.8
10	400	−400	2	39.94
10	400	−400	12	44.54
120	400	−400	2	50.12
120	400	−400	12	30.41
10	100	−400	7	94
10	700	−400	7	27.42
120	100	−400	7	36.92
120	700	−400	7	64.8738
65	400	−700	2	29.45
65	400	−100	2	31.3
65	400	−700	12	35.54
65	400	−100	12	10.1
65	100	−400	2	49.19
65	700	−400	2	37.82
65	100	−400	12	41.75
65	700	−400	12	30.16
10	400	−700	7	46.49
10	400	−100	7	27.78
120	400	−700	7	37.59
120	400	−100	7	32.72
65	400	−400	7	43.5
65	400	−400	7	44.65
65	400	−400	7	45.21
65	400	−400	7	44.8
65	400	−400	7	43.9
65	400	−400	7	44.68

3.3.1. Influence of Current Intensity and pH

Figure 2 represents the evolution of the nitrate removal efficiency as a function of the cathodic current intensity between −100 and −700 mA and the pH varying

from 2 to 12. These results reveal a significant interaction between these two parameters, indicating that the effect of current intensity on nitrate removal strongly depends on the pH of the solution.

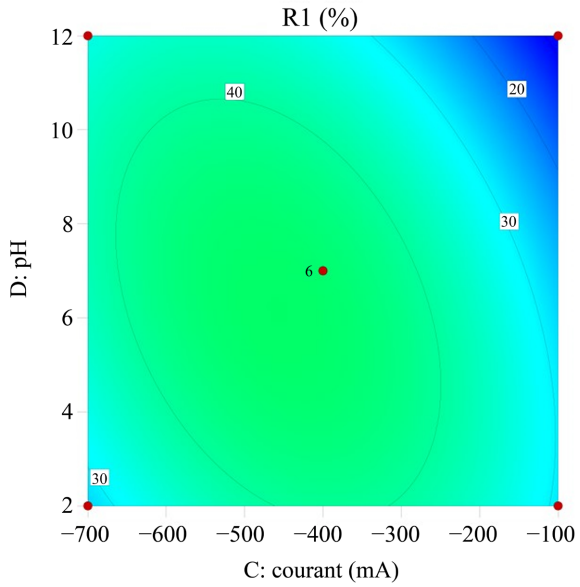

Figure 2. Effect of pH and cathodic current on the nitrate removal efficiency.

The analysis of the response surface reveals an elliptical region where nitrate removal efficiency reaches approximately 40% when the cathodic current ranges between −500 and −600 mA and the pH is between 6 and 8. However, under highly acidic conditions, increasing the absolute value of the current results in a decrease in nitrate removal efficiency, not exceeding 30%. A similar limitation is observed at low currents around −100 mA. In highly basic media, the nitrate removal efficiency is also limited to about 30%, particularly at low cathodic currents (around −200 mA). Within the optimal pH range of 6 to 8, increasing the cathodic current from −100 to −700 mA leads to a significant improvement in nitrate removal efficiency. However, at extreme pH values below 3 or above 10, the same increase in current leads in only a low nitrate removal efficiency, indicating an antagonistic interaction between extreme pH conditions and high cathodic currents. This result appears to indicate that H^+ and OH^- ions compete with nitrates and affect the nitrate removal efficiency. H^+ ions are reduced at approximately 0.00 V, and a high concentration of OH^- ions may hinder the mobility of nitrate ions, limiting their migration toward the cathode.

3.3.2. Influence of Time and pH

Figure 3 shows the evolution of the nitrate removal efficiency as a function of the electrolysis time between 10 and 120 min.

Contrary to the effect observed for the couple (I, pH), the evolution of the nitrate removal efficiency as a function of the couple (time, pH) indicates a planar representation, with efficiency variations of up to 50%. This uniformity suggests

that the effects of pH and time are largely independent. As illustrated in Figure 3, the reaction exhibits high efficiency in a minimal time at pH values between 6 and 8. This result suggests good mobility of nitrate ions in a neutral medium. In acidic conditions, the nitrate removal efficiency is limited to 40%. Beyond this maximum value, any increase in electrolysis time becomes unfavorable to the process. In agreement with our previous work, this result indicates a competition between proton reduction and the desired reaction.

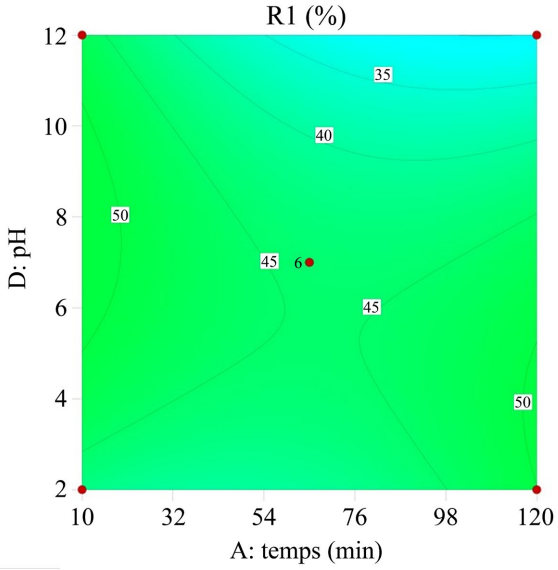

Figure 3. Effect of time and pH on the nitrate removal efficiency.

3.3.3. Influence of Time and Concentration

The following figure presents the evolution of the nitrate removal efficiency as a function of nitrate concentration.

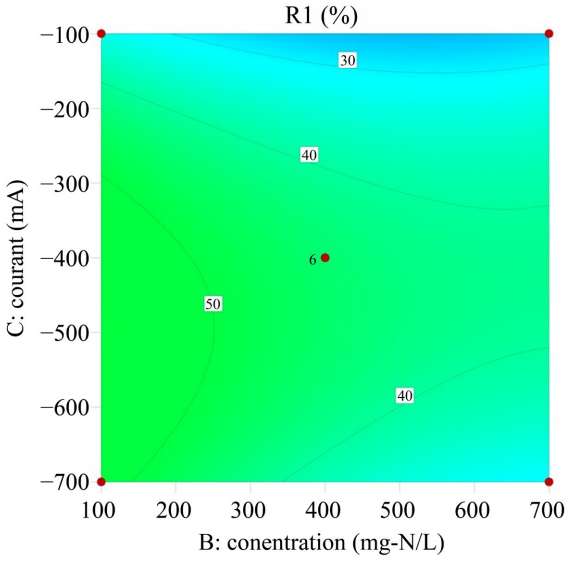

Figure 4. Effect of time and concentration on the nitrate removal efficiency.

Figure 4 illustrates the combined effect of electrolysis time and nitrate concentration on the nitrate removal efficiency. At elevated NO_3^- concentrations, a decrease in removal efficiency is observed. This may be due to the inefficiency of the process above a maximun concentration value. Although, an increase in efficiency is observed at low concentration, a decrease in removal efficiency is observed with increasing electrolysis time, suggesting that excessively long treatment under these conditions may reduce nitrate removal efficiency.

3.3.4. Influence of Current and Concentration

Figure 5 illustrates the evolution of the current varying between −100 to −700 mA in the concentration range 100 - 700 mg-N/L.

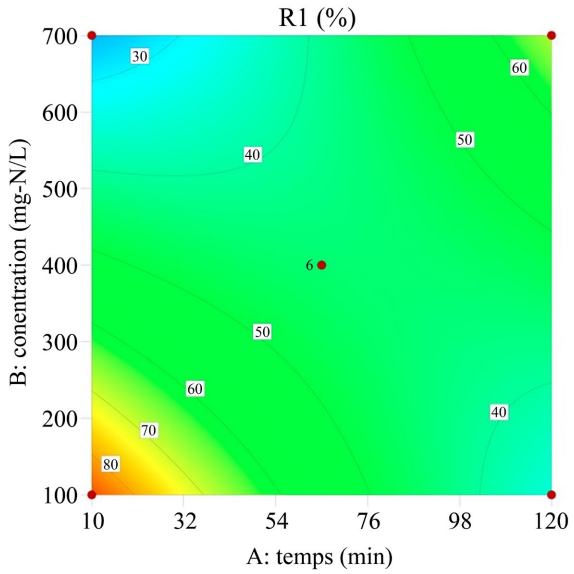

Figure 5. Effect of current and concentration on the nitrate removal efficiency.

The graph shows that the surface exhibits a relatively uniform ondulation and gradual progression as a function of concentration and current intensity, which facilitates the identification of optimal operating conditions. As for the current intensity, it is moduled between different values in order to evaluate a wide range of reduction current. Although, the increase in the absolute value of the electric current allows a higher electron production. This behavior can be attributed to the use of two $Ti/RuO_2 + IrO_2$ electrodes in a single-compartment cell, facilitating nitrate reduction. However, as illustrated in Figure 5, at high concentrations, the removal efficiency remains low even at high applied potentials and with a reduced electrode gap, indicating that other limiting factors may govern the process under these conditions.

3.3.5. Optimal Model Results

The optimization indicates that the initial nitrate concentration should be maintained very close to its minimum value, around 100 mg-N/L, which leads more efficient pollutant reduction. The optimal current intensity is strongly negative,

reflecting an intense electro-reductive regime, essential for activating the effective reduction of nitrates on the Ti/RuO_2 + IrO_2 electrode. The optimal pH, around 9.46, confirms the essential role of slightly alkaline conditions, which limit the formation of undesirable by-products such as nitrites. Furthermore, the optimal electrolysis time of 10 min shows that high nitrate removal efficiency can be achieved within a relatively short period, thereby limiting energy consumption and improving process sustainability.

3.3.6. Model Validation

To validate the developed model, four additional experiments were conducted under the previously determined optimal conditions: a $NaNO_3$ concentration of 100 mg-N/L, an electrolysis time of 10 minutes, and a reduction current intensity of −603 mA at a pH around 9.4. The experimental results at optimum conditions are shown in Table 4. The result shows good agreement between the average experimental values and the predicted values obtained from models. This result highlights a valid and applicable model for predicting the response.

Table 4. Verification of experimental results at optimum conditions.

Optimum conditions	Nitrate removal efficiency (%)
Mean experimental value	91.34
predicted value	91
gap	0.34

4. Conclusion

The objective of this study was to optimize nitrate removal by electrochemical process using the Design of Experiments methodology. Statistical analyses, particularly ANOVA, confirmed the relevance of the quadratic model, which exhibited a high coefficient of determination, indicating strong agreement between the experimental values and those predicted by the model. The results showed that current intensity and pH are the most influential parameters in the electrochemical process, while electrolysis time and concentration also affect overall treatment efficiency. The optimal operating conditions identified in this study were a reduction current intensity of −603 mA, a $NaNO_3$ concentration of 100 mg-N/L, and an electrolysis time of 10 min at a slightly basic pH. Method validation tests under these conditions showed a nitrate removal efficiency of 91%, confirming the validity of the approach. The results of this work open several promising perspectives for further development and improvement of the nitrate removal process by electro-reduction. Future studies could focus on the detailed analysis of intermediate products, such as nitrites and ammonium, to enhance the selectivity and sustainability of the process.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

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