Electrochemical removal of nitrate from wastewater

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Abstract. There are many natural elements that cause pollution of groundwater and surface water. Nitrate is one of those pollutants that could cause serious problems to the aquatic life and human health. Consequently, environmental organisations and most of the governments around the world limited nitrates in potable water to 50 and 10 mg/L for adults and babies, respectively. The current research explores the removability of nitrate from industrial effluents using an electrochemical cell (ESE). The latter is a laboratory-scale batch flow cell having steel electrodes. The experiments were conducted using nitrates solution having100 mg/L. This investigation also studied the impact distance between the electrodes (SBE), treatment period (TP), current density value (CDV) and the initial pH on nitrates removal. Box-Behnken design technique (BBD) has been utilized to optimize the impacts of TP, CDV, SBE and pH on the removal of nitrates by ESE and also to generate a regression model to simulate the removability of nitrates by the ESE. The results of this investigation indicated that the optimum removal of nitrates was 93.4% that achieved at TP of 60 minutes, SBE of 0.5 cm, CDV of 3 mA/cm² and initial pH of 6. Furthermore, the developed model's findings were in a respectable compatibility with the experimental findings (R²=0.904). In conclusion, the current investigation indicated that the ESE has good capacity to remove nitrate.

1. Introduction

Nitrate is an inorganic ion, which has chemical formula of NO3−, and it is an important component of the normal nitrogen (N) cycle. The nitrogenous compounds (mainly produced from human wastewater) are naturally digested in the aquatic environment by specialist microorganisms, in water or soil, producing ammonia, which then undergoes oxidizing reactions to yield nitrate and nitrite [1]. Regardless of the fact that nitrate is chemically categorized as unreactive and stable, but there is a wide variety of health and environmental issues related to its availability in the aquatic environment [2-4].

Owing to the man-made operations, the natural nitrate content in surface and ground water was substantially altered since the start of the industrial revolution period. For instance, the rapid growth in crop production was followed by substantial use of N-containing fertilizers, resulting in the development of highly contaminated nitrogen wastewater [5-9]. Furthermore, in order to meet the increasing demands for products of livestock and meat, the livestock farming has tremendously decreased during the last few years, and often generates substantial amounts N-containing wastewater, which naturally transformed into nitrates. In fact, wastewaters of many industrial sectors consider as severely contaminated with nitrates and nitrites. For example, food preservative, explosive materials, glasses,
oxidizing agents and potassium purification industries produce substantial amounts of N-containing wastewater [10, 11]. The existence of nitrate could cause negative impacts on environments and human health, where it was stated, in the recent researches, that nitrate causes respiratory disease outbreaks, asthma and reductions or prevents iodine absorption [10, 11]. Due to methaemoglobinemia, nitrites are categorised as hazardous elements for pregnant women and infants; the presence of nitrites in babies and infants’ stomachs leads to produce nitrates, which interacts with haemoglobins producing met-haemoglobin (methaemoglobinemia) that does possess the capacity to carry oxygen, see Equation 1 [12, 13]. It was rigidly proved, by the World Health Organization (WHO), that the majority of the methaemoglobinemia cases occur in the stomachs of infants (≤ 3 months old) [10].

\[
\text{Haemoglobin (Fe}^{2+}\text{)} + \text{NO}^{2-} \rightarrow \text{Methaemoglobin}
\]

Furthermore, a large number of unproven research findings linked nitrate pollution with deadly illnesses, like oesophageal, stomach cancer, and lymphoma of non-Hodgkin illnesses. Nitrate has negative effects on environment and animals besides its impacts on human health, where the literature proved relationships between nitrate concentrations and abortions in animals [4, 10]. Environmentally, specifically in the aquatic environment, nitrate is responsible for several issues. For instance, the existence of nitrate in water contributes to what known as algae revolution or eutrophication, which is defined as the development of algae mats that obstructs sunlight and speeds the consumption of dissolved oxygen, and consequently, massive death of aquatic life [14]. Dissolved oxygen is also depleted during the decomposition of \((\text{NH}_2)_2\text{CO}\) that forms ammonia, which in turn undergoes oxidising reactions (by some types of bacteria, like Nitrobacter and Nitrosomonas) yielding nitrite; these reactions deplete oxygen according to the following formulas:

\[
\text{NH}_3 + 3O^2 \rightarrow 2\text{NO}_2^- + 2H^+ + 2H_2O
\]

\[
2\text{NH}_2^- + O_2S \rightarrow 2\text{NO}_3^-
\]

Furthermore, anaerobic bacteria could convert nitrate and nitrite to nitrogen monoxide and nitrogen gases as shown in the following formula:

\[
\text{NO}_2^- \text{ or NO}_3^- \rightarrow \text{denitrifications} \rightarrow N_2 + 2H^+ + N_2O
\]

Remediation of water from nitrate and other types of nutrients therefore had become a predominant necessity to avoid these harmful consequences. The widely accepted techniques to remove nitrate are biological and physicochemical methods [15-17]. In addition to that, other techniques like reverse osmosis were practiced, but on limited scales due to the investment costs and the need for post and pre-treatment procedures [4, 15-18]. The electrocoagulation method is known for its efficacy in the elimination of diverse pollutants at short treatment time [19, 20], needless for chemical additives [21, 22], cost-effectiveness [23, 24] and low production of sludge [8, 25]. Because it produces minor amount of sludge with low water content [3], it does not required large dumping places for sludge or expensive handling machines and management strategies [26-28]. Additionally, the EC units could be easily automated or integrated with other methods [3, 21], which confirms the possibility of using the sensing technology [29-33] in the monitoring and controlling of the performance of the EC units. Furthermore, many recent studies confirmed that the sludge of the EC units could be recycled in manufacturing of building blocks [34-37], which makes the EC method more cost-effective. Consequently, the EC technique was utilized in the present research to get rid of nitrate from wastewater.

2. Methodology

2.1. Setting up of experimental works

A batch flow ESE was manufactured by the authors, according the stated specifications in the related literature, from Perspex sheet in the laboratory [7, 19, 23]. This cell was manufactured in a rectangular shape with total volume of 1400 cm³. The electrodes, utilized in the ESE, have been made from iron
with a total area of 797.2 cm². An attached peristaltic water pump to ESE was utilized to circulate solutions during the treatment process. Each experiment was initiated by switching on the DC power supply to flow the electric current through the ESE, which triggers the production of coagulants from iron anode. Samples were collected at a specified depth from the ESE (outlet at 1 cm from the bottom of the ESE) during the experiments; these samples are approximately 10 mL in size and were obtained at the required intervals (according to the outcomes of the Box-Behnken design, which is described later in this article). Since these samples are normally containing some flocs that developed during the treatment process, they have been filtered before measuring nitrate concentrations.

All tests have been conducted at normal laboratory temperature (about 20°C). NaCl was utilized to control the conductivity of solution, which was kept around 0.320 mS/cm. The applied electric current was supplied using the rectifier (DC source), whereas the initial pH was controlled using NaOH or HCl. The remediation of nitrates by the ESE was optimised for the influences of TP, SBE, CDV and initial pH.

The efficiency of removal was calculated utilizing the following formula [18]:

\[
\text{Removal} \, (\%) = \frac{C_0 - C}{C_0} \times 100
\]

Whereas, the initial and measured nitrate concentration are \(C_0\) and \(C\), respectively.

### 2.2. Contaminated wastewater samples

A concentrated nitrate solution (1 g/L) was produced, at the beginning of research, by adding potassium nitrates in deionized water. The following equation (Eq. 6) was used to calculate the required weight of potassium nitrates for preparing the concentrated nitrate solution [10]:

\[
\text{Weight of nitrate} = V \times C_{st} \times \frac{M_{\text{wt}}}{A_{\text{wt}}}
\]

Whereas:

- \(V\): Specimen’s sample.
- \(C_{st}\): essential nitrate concentration.
- \(M_{\text{wt}}\): \(\text{KNO}_3\) molecular weight.
- \(A_{\text{wt}}\): the nitrate atomic weight.

Depending on Eq.6, the required nitrate weight to produce 1g/L solution was 1.64 grams per litter of deionized water.

### 2.3. Electrolyzing process

The ESE experiments have been conducted at various operating conditions, which involved SBE, CDV, TP and initial pH. The studied limits of these factors are tabulated in Table 1. Box-Behnken design (BBD) has been utilized to optimize the impact of these factors on the treatability of nitrate solution by the ESE.

Box-Behnken design (BBD) is a common technique of optimization that is widely utilized in the researches that investigates treatment of water. This wide usage of BBD in optimization of the impacts of treatment parameters on water treatability is due to its acknowledged ability to estimate the impacts of many parameters at the same time. Statistical optimisation and modelling was adopted in this study because it saves time of experiments, decreases number of experiments and gives the ability to predict the efficiency of the process under different conditions [17, 38-41]. In this investigation, the BBD was utilized to improve the impacts of four factors on nitrate removability from contaminated solution, namely TP (up to 50 minutes), the SBE (up to 1.5 cm), CDV (up to 3 mA/cm²) and initial pH (at three values, 4, 6 and 8). The BBD was worked utilizing SPSS 20 packaged. It is remarkable to note that the investigated parameters were coded prior to operating the BBD, see Table 1, to shorten the form of the produced model.

| Parameter | Initial pH | CDV(mA.cm⁻²) | SBE(mm) | TP(minutes) |
|-----------|------------|--------------|---------|-------------|
|           |            |              |         |             |

### Table 1. Codes and ranges of the studied factors.
that the predicted removals were compared to the experimental research were also utilized to create a regression model (prediction model) that reproduces the removability of nitrates by the ESE at various operating conditions.

| Code of parameter | Studied range | F1  | F2  | F3  | F4  |
|-------------------|---------------|-----|-----|-----|-----|
| Minimum           | 4.0           | 1.0 | 5.0 | 10.0| 10.0|
| Maximum           | 8.0           | 3.0 | 15.0| 15.0| 50.0|

BBD's outcomes, tabulated in Table 2, demonstrated that 27 runs are needed to optimize and simulate the impacts of the studied factors on the removability of nitrates by the ESE. Initially, percentage of nitrate removal, in each run, was experimentally measured, and then the obtained removals were utilized to create a regression model (prediction model) that reproduces the removability of nitrates by the ESE.

| Runs | F1  | F2  | F3  | F4  |
|------|-----|-----|-----|-----|
| 1    | 4.0 | 1.0 | 10.0| 30.0|
| 2    | 8.0 | 1.0 | 10.0| 30.0|
| 3    | 4.0 | 3.0 | 10.0| 30.0|
| 4    | 8.0 | 3.0 | 10.0| 30.0|
| 5    | 6.0 | 2.0 | 5.0 | 10.0|
| 6    | 6.0 | 2.0 | 15.0| 10.0|
| 7    | 6.0 | 2.0 | 5.0 | 50.0|
| 8    | 6.0 | 2.0 | 15.0| 50.0|
| 9    | 4.0 | 2.0 | 10.0| 10.0|
| 10   | 8.0 | 2.0 | 10.0| 10.0|
| 11   | 4.0 | 2.0 | 10.0| 50.0|
| 12   | 8.0 | 2.0 | 10.0| 50.0|
| 13   | 6.0 | 1.0 | 5.0 | 30.0|
| 14   | 6.0 | 3.0 | 5.0 | 30.0|
| 15   | 6.0 | 1.0 | 15.0| 30.0|
| 16   | 6.0 | 3.0 | 15.0| 30.0|
| 17   | 4.0 | 2.0 | 5.0 | 30.0|
| 18   | 8.0 | 2.0 | 5.0 | 30.0|
| 19   | 4.0 | 2.0 | 15.0| 30.0|
| 20   | 8.0 | 2.0 | 15.0| 30.0|
| 21   | 6.0 | 1.0 | 10.0| 10.0|
| 22   | 6.0 | 3.0 | 10.0| 10.0|
| 23   | 6.0 | 1.0 | 10.0| 50.0|
| 24   | 6.0 | 3.0 | 10.0| 50.0|
| 25   | 6.0 | 2.0 | 10.0| 30.0|
| 26   | 6.0 | 2.0 | 10.0| 30.0|
| 27   | 6.0 | 2.0 | 10.0| 30.0|

3. Results and discussion
Optimization of impacts of TP, CDV, SBE and the initial pH on the removability of nitrates using SES experimental work was conducted, as mentioned above, using the BBD technique. The latter showed that 27 runs are needed to optimize the impacts of the initial pH (F1), CDV (F2), SBE (F3) and TP (F4). The experiments were conducted depending on the parameter values specified in Table 1. The obtained removal efficiencies are illustrated in Table 3, and Figures 1-6. The efficiencies gained from the experimental research were also utilized to create a regression model, Eq.7, to simulate the removability of nitrates using the ESE.

Removal(%) = −511.4 + 90.1 × F1 + 73.5 × F2 + 27.09 × F3 + 6.05 × F4 − 6.60 × F1 × F1 − 12.97 F2 × F2 − 0.611 F3 × F3 − 0.0511 F4 × F4 + 0.86 F1 × F2 − 0.968 F1 × F3 + 0.061 F1 × F4 − 1.35 F2 × F3 + 0.064 F2 × F4 − 0.2752 F3 × F4

To validate this model, it was applied to predict nitrate removing using the stated conditions in Table 2, and the predicted removals were compared to the actual ones. The predicted nitrate removals are tabulated in Table 4, which are very comparable to the observed removals. Additionally, it was indicated that the model’s determination coefficient (R²) was 0.827 that means this model could reliably estimate
about 83% of the impacts of the investigated factors on the removability of nitrates using the ESE technique.

It could be observed that the increase in nitrate removability is compatible with increase of TP, CDV, and initial pH. For example, increasing initial pH from 4 to 8 caused an increase in the removal of nitrate by about 15.0% (from 27.14% to 42.78%, respectively) (at TP of 30 minutes, SBE of 10 mm and CDV of 1 mA/cm²). The reason for this increase is related to the increase in the number of the positively charged coagulants in the basic pH values, which helps to remove nitrates that naturally carried a negative charge. While at acidic pH levels, negatively charged coagulants became the predominant, which repel the negatively charged nitrate ions [4, 42].

Table 3. Observed nitrate removal.

| Runs | F1 | F2 | F3 | F4 | Observed removal (%) |
|------|----|----|----|----|-----------------------|
| 1    | 4.0| 1.0| 10.0| 30.0| 27                    |
| 2    | 8.0| 1.0| 10.0| 30.0| 42                    |
| 3    | 4.0| 3.0| 10.0| 30.0| 55.2                  |
| 4    | 8.0| 3.0| 10.0| 30.0| 77.1                  |
| 5    | 6.0| 2.0| 5.0 | 10.0| 36.8                  |
| 6    | 6.0| 2.0| 15.0| 10.0| 70                    |
| 7    | 6.0| 2.0| 5.0 | 50.0| 93                    |
| 8    | 6.0| 2.0| 15.0| 50.0| 16.1                  |
| 9    | 4.0| 2.0| 10.0| 10.0| 18.1                  |
| 10   | 8.0| 2.0| 10.0| 10.0| 25.1                  |
| 11   | 4.0| 2.0| 10.0| 50.0| 56.3                  |
| 12   | 8.0| 2.0| 10.0| 50.0| 73                    |
| 13   | 6.0| 1.0| 5.0 | 30.0| 44.9                  |
| 14   | 6.0| 3.0| 5.0 | 30.0| 93.4                  |
| 15   | 6.0| 1.0| 15.0| 30.0| 43.5                  |
| 16   | 6.0| 3.0| 15.0| 30.0| 65                    |
| 17   | 4.0| 2.0| 5.0 | 30.0| 33.8                  |
| 18   | 8.0| 2.0| 5.0 | 30.0| 80.5                  |
| 19   | 4.0| 2.0| 15.0| 30.0| 33.1                  |
| 20   | 8.0| 2.0| 15.0| 30.0| 41.1                  |
| 21   | 6.0| 1.0| 10.0| 10.0| 23.1                  |
| 22   | 6.0| 3.0| 10.0| 10.0| 45.1                  |
| 23   | 6.0| 1.0| 10.0| 50.0| 63.1                  |
| 24   | 6.0| 3.0| 10.0| 50.0| 90.2                  |
| 25   | 6.0| 2.0| 10.0| 30.0| 89.4                  |
| 26   | 6.0| 2.0| 10.0| 30.0| 89.7                  |
| 27   | 6.0| 2.0| 10.0| 30.0| 89.3                  |
Table 4. Predicted nitrate removal.

| Runs | F1 | F2 | F3 | F4 | Predicted removal (%) |
|------|----|----|----|----|------------------------|
| 28   | 4.0| 1.0| 10.0| 30.0| 27.14 |
| 29   | 8.0| 1.0| 10.0| 30.0| 42.78 |
| 30   | 4.0| 3.0| 10.0| 30.0| 54.1 |
| 31   | 8.0| 3.0| 10.0| 30.0| 76.62 |
| 32   | 6.0| 2.0| 5.0 | 10.0| 21.245 |
| 33   | 6.0| 2.0| 15.0| 10.0| 57.345 |
| 34   | 6.0| 2.0| 5.0 | 50.0| 100* |
| 35   | 6.0| 2.0| 15.0| 50.0| 31.345 |
| 36   | 4.0| 2.0| 10.0| 10.0| 21.07 |
| 37   | 8.0| 2.0| 10.0| 10.0| 35.27 |
| 38   | 4.0| 2.0| 10.0| 50.0| 45.23 |
| 39   | 8.0| 2.0| 10.0| 50.0| 69.19 |
| 40   | 6.0| 1.0| 5.0 | 30.0| 48.805 |
| 41   | 6.0| 3.0| 5.0 | 30.0| 92.705 |
| 42   | 6.0| 1.0| 15.0| 30.0| 43.365 |
| 43   | 6.0| 3.0| 15.0| 30.0| 60.265 |
| 44   | 4.0| 2.0| 5.0 | 30.0| 38.105 |
| 45   | 8.0| 2.0| 5.0 | 30.0| 76.545 |
| 46   | 4.0| 2.0| 15.0| 30.0| 38.525 |
| 47   | 8.0| 2.0| 15.0| 30.0| 38.245 |
| 48   | 6.0| 1.0| 10.0| 10.0| 27.68 |
| 49   | 6.0| 3.0| 10.0| 10.0| 55.52 |
| 50   | 6.0| 1.0| 10.0| 50.0| 54.16 |
| 51   | 6.0| 3.0| 10.0| 50.0| 87.12 |
| 52   | 6.0| 2.0| 10.0| 30.0| 89.53 |
| 53   | 6.0| 2.0| 10.0| 30.0| 89.53 |
| 54   | 6.0| 2.0| 10.0| 30.0| 89.53 |

*The predicted value was more than 100%, thus it was capped at 100%

In previous researches, the positive influence of the CDV on the ability of the ESE to remove nitrates from solution was linked to the increase in the generation of coagulants from the anodes, thereby the removability of nitrate improved [43]. Likewise, for the impact of the TP, it has been shown that increasing the TP leads to more production of coagulants and, therefore, better nitrate removal was observed [43]. The impact of SBE was different from the impact of other factors (TP, CDV and initial pH), where it was observed that expanding the SBE reduced nitrate removal. Expanding the SBE, according to the previous researches, lead to an increase in the electrical resistance of the ESE (resists the flow of the DC current), which causes a reduction in the production of coagulants, and consequently it decreases the removal efficiency [4].

In general, the outcomes of the conducted experiments indicated that the optimum nitrate removal (93.40%) was achieved at TP, SBE, CDV and initial pH of 30 minutes, 5.0 mm, 3 mA/cm$^2$ and initial pH of 6.

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In general, the outcomes of the conducted experiments indicated that the optimum nitrate removal (93.40%) was achieved at TP, SBE, CDV and initial pH of 30 minutes, 5.0 mm, 3 mA/cm$^2$ and initial pH of 6.

**Conclusions**

As a result of the experimental work, the following key facts were concluded:

1. The steel-based ESE demonstrated good ability to remediate wastewater from nitrates.
2. The ability of the ESE to remove nitrates depends on many factors, such as SBE, TP, CDV and initial pH.
3. Some factors have a positive effect on the removability of nitrate, which are the initial pH, TP and CDV, while the SBE negatively impacts the removability of nitrates.
4. The performance of the ESE was found to be reproducible using a regression model suitable technique such as the BBD. It is recommended to conduct more investigations about the applicability of the electrochemical techniques to treat water or wastewater using different electrode materials.

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