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Electrochemical denitrification using carbon cloth as an efficient anode

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ABSTRACT

One main problem encountered in electrochemical denitrification is anode scarifying during the electrochemical process. To address this problem, carbon cloth (CC) was proposed as a stable, low-cost, and low level of corrosion anode. The highest nitrate removal efficiency (NRE) was achieved at the point of zero charge (pH_{PZC}) of the anode interface. The NRE slightly increased with increasing current in the range of 10–60 mA. Stereochemistry of the electrode corrosion indicates CC corrosion could produce CO_2, CO, and H_2 in the reactor, which H_2 gas can enhance denitrification by hydrogenation reactions. Faraday's equation showed 11.2 mg/L corrosion for an iron electrode when it was used instead of CC. It is expected that experimental results could be used as a reference for the utilization of CC electrodes in electrochemical denitrification.

Keywords: Denitrification; Carbon cloth; Stainless steel; Nitrate; Electrochemical; Kinetic

1. Introduction

Nitrate ion is a major type of water pollutant especially in the groundwater used in agricultural activity [1,2]. Serious health disorders result from the consumption of water containing nitrate, such as methemoglobinemia, the "blue baby syndrome" for infants aged less than 6 months, as well as reduced stomach acidity in pregnant women and adults [3,4]. The epidemiological studies have confirmed the carcinogenic potential of nitrate compounds present in water. To protect infants from methemoglobinemia syndrome, the maximum contaminant level for nitrate has been set to 10 mg/L by the US Environmental Protection Agency (US EPA) [5]. Available data show that no considerable nitrate removal efficiency (NRE) is achieved by conventional treatment processes for drinking water purification, including flocculation, sedimentation, and filtration [6]. Because of the low NREs using the methods, various alternatives have been developed, such as reverse osmosis [7], ion-exchange resins [8], electrochemical reduction [9], biological denitrification [10], catalytic, and electrocatalytic methods [11]. However, these denitrification processes result in secondary pollution, which can then lead to an increase in the overall cost of the process, making them unsuitable for small quantity and energy demand-sensitive applications. Among the proposed methods, the electrochemical denitrification is a notable technology that has several advantages, such as high treatment efficiency, low sludge production, small area requirement, and relatively lower investment costs. Hence, this method has been used by some researchers in recent years. In electrochemical nitrate reduction, especially in the cathode area, the nitrates are mainly converted to nitrites, ammonia, and nitrogen (Eqs. (1)–(3)) [3,12]. At the anode interface (Eqs. (4) and (5)), the nitrate is removed by oxidizing the cathode product to the initial nitrate and nitrogen (Eq. (6)). However, providing suitable conditions to simultaneously perform cathode reduction and anode oxidation to remove initial nitrate is a main problem that must be solved in the electrochemical denitrification process [12].

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$$NO_3^- + 2e^- + H_2O \rightarrow NO_2^- + 2OH^- \quad (1)$$

$$2NO_3^- + 16e^- + 12H_2O \rightarrow 2NH_3 + 18OH^- \quad (2)$$

$$2NO_3^- + 10e^- + 6H_2O \rightarrow N_2 + 12OH^- \quad (3)$$

$$NO_2^- + 6H_2O \rightarrow NO_3^- + 2e^- + 2H^+ \quad (4)$$

$$2NH_3 \rightarrow N_2 + 6e^- + 6H^+ \quad (5)$$

$$2NO_3^- + 10e^- + 6H_2O \rightarrow N_2 + 12OH^- \quad (6)$$

The corrosion of electrodes in aqueous solution environments is another problem involved in using electrochemical systems [12,13]. The high cost of stable materials, such as boron-doped diamond has led to study for low-cost electrode materials [14]. The first requirement for using various metal electrode in the aqueous/drinking water environment is that harmful corrosion products such as metal ions should not be generated [12,15]. Corrosion causes electrode surface roughening that, provides suitable places where bacteria can grow, leading to related disinfection problems. Moreover, corrosion can release ions from the metallic materials into the treated water. Released ions lead to water discoloration, inducing allergic reactions in susceptible people; these ions can also reach toxic levels when concentrations are higher than the standard levels. Meanwhile, in terms of operation cost, corroded electrodes must be replaced with new ones, thus increasing electrode supply costs. One of the most used electrode materials in electrochemical nitrate reduction is metal. Stainless steel (SS) 304 mesh is a favorable material used in electrode fabrication as it has excellent mechanical and electrical properties. SS has been widely used as electrode because of high corrosion resistance, low cost, and environmental stability [16]. Furthermore, carbon cloth (CC), a type of carbonated material has been increasingly used in many applications due to its high surface area and good chemical stability [17]. Carbon has a low equilibrium potential for carbon corrosion ($E°$ = 0.207 V vs. SHE and $E°$ = 0.518 V vs. SHE for CO_2 and CO producing, respectively) [18]. Moreover, utilizing CC as anode in the electrochemical systems is relatively safe because CC corrosion have no harmful by-products released into treated water. Although mesh SS 304 cathodes have been proposed for electrochemical denitrification [19], the combination of CC anode and mesh SS cathodes has not been studied. The aim of this work is to study denitrification in an electrochemical system using CC electrode as efficient interface anode. The influences of applied currents, supporting electrolyte, chloride ions, pH values, oxidation–reduction potential (ORP), and electrical conductivity (EC) during the electrochemical process were investigated.

2. Experimental

2.1. Material and setup

The electrochemical system for this study consisted of a Plexiglass reactor with a thickness of 1 cm, effective capacity of 3.5 L, and 304 SS mesh (opening area percentage, 52%; weight, 2.1 kg/m^2; thickness, 1.168 mm; dimension, 34 cm × 31 cm) as cathode electrode. The CC (Torayca-T300B-3000-40B, 205% ± 5% g/m^2, 103 kg/m^3, tensile strength, 3,950 MPa; tensile modulus, 235 GPa; 45 cm × height 31 cm) electrodes were shaped in a cylindrical cross-section with a plastic support. A DC power supply (Atten APS 3005S-3, China) with operational options for controlling the constant voltage and current density were applied in the electrochemical denitrification system (Fig. 1). A UV/Vis spectrophotometer (550 SE; Perkin-Elmer, Waltham, MA, USA) and ORP meter (Eutech, Singapore) were used to measure ORP at different time intervals. Temperature, EC, and pH values were measured with HI 98129 (Hana Instruments, Ann Arbor, MI, USA) and HQ40d (Hach, Loveland, CO, USA), respectively.

2.2. Electrochemical denitrification

In the present study, the initial nitrate concentrations of 50–150 mg/L were inoculated in water to simulate real conditions for the electrochemical denitrification experiments. About 0–2 g/L NaCl and 0.5–1 g/L NaF were added into the solution to study the supporting electrolyte effects. Different applied currents (5–60 mA), initial nitrate concentrations (50–150 mg/L), and initial pH values (4, 7, and 9) on NRE were studied. At different time intervals, samples were drawn from the electrochemical system for further analysis. All analyses were conducted according to standard methods. The nitrate content was measured at λ_{max} = 220 and 275 nm using the spectrophotometric method:

$$\text{Nitrate}\left(\text{mg/L}\right) = \frac{\text{Abs}_{220} - 2\times\text{Abs}_{275}}{0.05} \quad (7)$$

where Abs is absorption, and 0.05 obtained from standard curve.

The nitrite content was analyzed with a colorimetric method using sulfanilamide and naphthylethylenediamine dihydrochloride reagents with absorption rate set at λ_{max} = 543 nm. Free chlorine was determined by N,N diethyl-p-phenylene diamine ferrous titrimetric method according to Standard Methods 4500-Cl.

To study the point of zero charge (pH_{PZC}) at the used electrode material, equilibrium technique was used by calculating H^+ and OH^-, as reported in the our previous works [20,21]. Briefly, a series of 20 mL 0.01 M KNO_3 electrolytes of different pH levels (2–12) were prepared. Then, 0.1 M of HCl and NaOH were added to adjust the pH levels. About 1 g of each electrode immersed (with shaking and constant temperature) in the electrolyte helped to adjust the pH. After 48 h, the final pH of the solution (drifted pH) was recorded and plotted (Eq. (8)) against the initial pH levels.

$$\Delta\text{pH} = \text{final pH} - \text{initial pH} \quad (8)$$

3. Results and discussion

3.1. Influence of the applied current

The effect of current density on nitrate removal during the electrochemical denitrification is shown in Fig. 2. As can be seen, the nitrate removal rate almost did not increase with

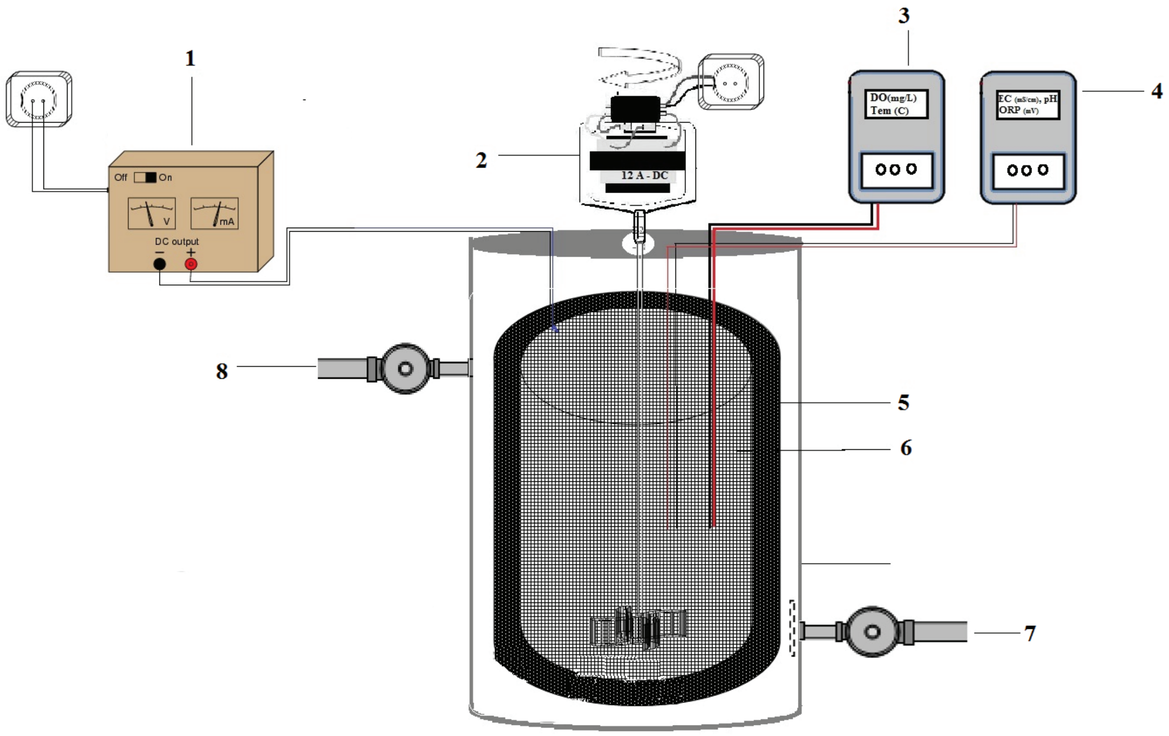

Fig. 1. Experimental apparatus: (1) DC power supply, (2) electromotor, (3) electrical conductivity (EC), (4) oxidation–reduction potential (ORP) and pH meter probe, (5) carbon cloth electrode with plastic mesh support (anode), (6) 12 Mesh T304 stainless electrode (cathode), (7) and (8) influent.

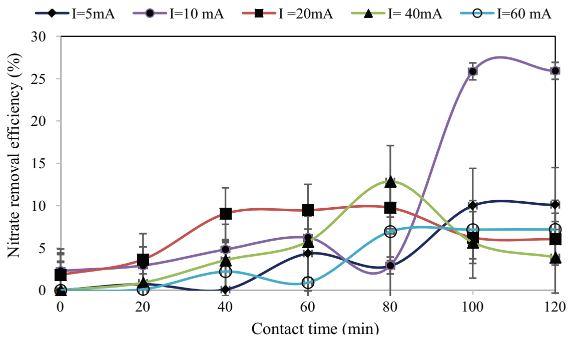

Fig. 2. Nitrate removal efficiencies under different currents and time intervals.

increasing current density in the range of 10–60 mA. The rate initially increased within the range of 5–10 mA, but it has not increased within the range of 10–60 mA. Hence, 10 mA was chosen because of energy saving. CC could have released hydrogen gas in the interface of the electrode, which led to the slow increase in nitrate removal rate. The hydrogen production was theoretically calculated using this equation:

$$n_{H_2,t} = \frac{\int_0^t I dt}{nF} \tag{9}$$

where I is the current, n (=2) is the number of electrons forming one hydrogen molecule, and F is the Faraday constant. The obtained results showed production of 0.009067358 H_2 L^{-1} and 0.10880829 mmol H_2 L^{-1} for the 5 and 60 mA currents, respectively. Hu et al. [18] proposed the following equations for carbon corrosion:

$$C(s) + 2H_2O \rightarrow O_2 + 4H^+ + 4e^- \tag{10}$$

$$H_2 \rightarrow 2H^+ + 2e^- \tag{11}$$

$$O_2 + 4H^+ + 4e^- \rightarrow 2H_2O \tag{12}$$

$$2H_2O \rightarrow O_2 + 4H^+ + 4e^- \tag{13}$$

$$2H_2 + O_2 \rightarrow 2H_2O \tag{14}$$

Previous study has reported that the CC corrosion probably leads to H_2 and electron production [22], which can then enhance denitrification through the hydrogenation reactions [23]. Hence, a current of 10 mA was chosen for the subsequent electrochemical denitrification experiments because it exhibited a relatively high reduction rate in the present experiments. A probable reason for this result is that the lower amount of produced hydrogen has led to the low selectivity of ammonia formation [12].

3.2. *Effect of initial nitrate concentrations*

The effect of initial nitrate concentrations (50–150 mg/L) on electrochemical nitrate reduction was studied. The samples were treated with a current of 10 mA following actual pH values for 150 min. As shown in Figs. 3(a) and (b), nitrate was reduced within the experimental time, leading to the

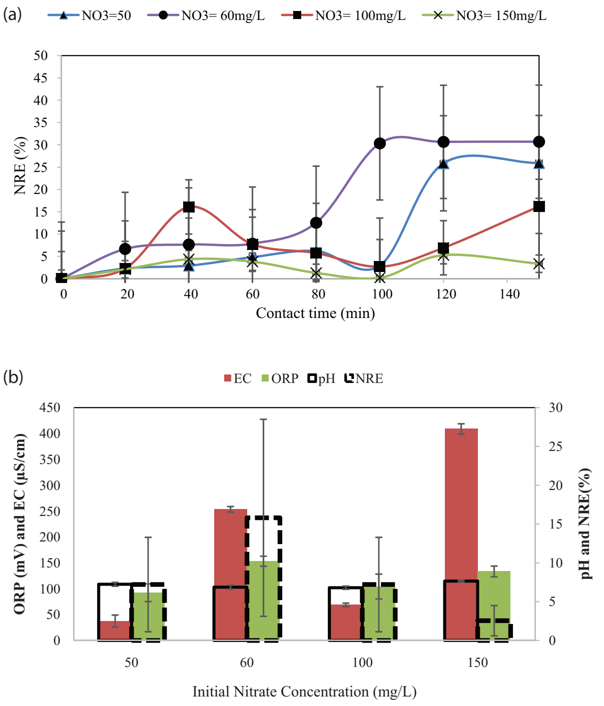

Fig. 3. (a) Nitrate removal efficiencies under different nitrate concentrations. (b) ORP, EC, nitrate removal efficiencies, and pH changes resulting from different initial nitrate concentrations.

formation of nitrite as a main by-product. NRE showed an uptrend and then a downtrend at different time intervals for each initial nitrate concentration. However, these occurred at a slower rate because of the further conversion to nitrate and other by-products, as shown in Eqs. (15)–(17) [19]:

$$NO_3^- + 2e^- + H_2O \leftrightarrow NO_2^- + 2OH^- \tag{15}$$

$$NO_2^- + 6e^- + 5H_2O \leftrightarrow NH_3 + 7OH^- \tag{16}$$

$$NO_2^- + 3e^- + 2H_2O \leftrightarrow 1/2N_2 + 4OH^- \tag{17}$$

Furthermore, the fluctuation in denitrification within the treatment time resulted from rapid nitrite oxidization to nitrate (Eq. (15)). As shown in Fig. 3(b), initial pH values for all solutions were around 7.14 ± 0.21. The initial values increased rapidly by as much as 0.6 owing to the production of OH^- during nitrate and nitrite reduction, as indicated in the previous reactions (Eqs. (15)–(17)). Additionally, the pH value of the effluent increased 0.5–0.9 units because of the hydroxide ions generated by the electrolysis of water [15].

3.3. Effect of supporting electrolyte

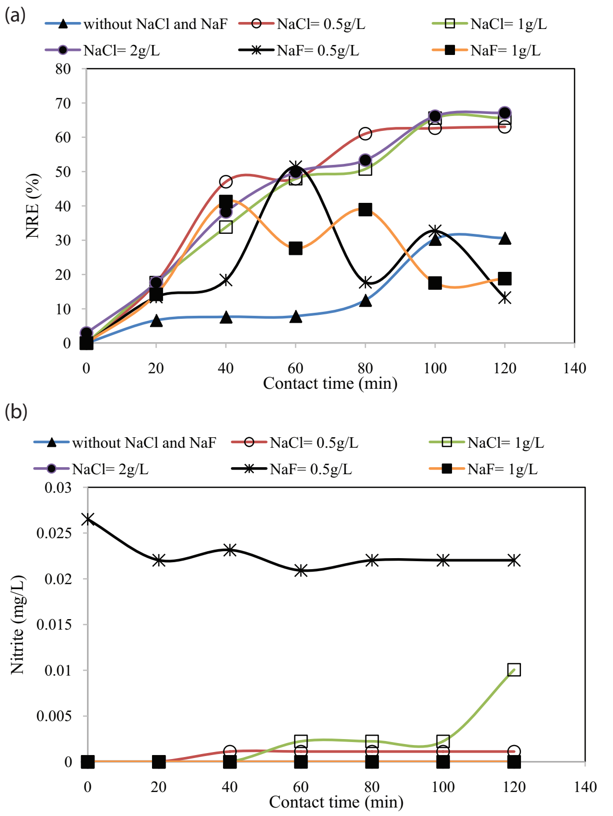

Fig. 4. (a) and (b) Nitrate removal efficiencies under different supporting electrolyte.

Figs. 4(a) and (b) show the variations of nitrate and nitrite, respectively, during electrolysis in the presence of NaCl and NaF. As can be seen, nitrate concentrations sharply decreased compared with the control sample (without the added NaCl). As shown in Fig. 4(a), the highest NRE was obtained with the addition of NaCl, while the lowest NRE was attain when NaF was added. The NREs increased with respect to treatment time in all cases, but decreased from 13.27% to 5.41% and from 41.2% to 18.8% in 120 min after addition of 0.5 and 1 g/L NaF, respectively. Meanwhile, with the addition of NaCl, NREs increased by increasing treatment time and minimal concentrations of nitrite were detected in the treated solution. Although the NaCl dosage increased from 0 to 2 g/L, the NREs at the end of the treatment (120 min) were similar for 0.5, 1, and 2 g/L of NaCl. Moreover, the nitrate reduction rate at higher NaCl dosage was not higher than that at lower NaCl addition (2 vs. 1 g/L) (Fig. 4). This can be attributed to the fact that nitrate removal was decreased in the presence of chloride ions. A previous study has reported the effectiveness of the chloride on the nitrate reduction, which is due to the potential of these anions to be specifically adsorbed on the electrode surface [2]. In the present study, because of the pH_{PZC} of SS 304 and CC (5 vs. 5.5) and pH of aqueous environment (pH = 7), this assumption is rejected. This can be explained by the assumption that specifically adsorbed anions inhibited the reduction of nitrate because of the decreased active surface area of the electrode.

A similar study reported that the denitrification rate in the presence of 250 mg/L NaCl was retarded with Ti cathode, whereas NaF had little effect on the nitrate reduction [2]. In the present study, the NREs were 67.17% and 63.05% with the addition of 2 and 0.5 g/L NaCl, respectively. Furthermore, in the presence of 2 g/L NaCl (Fig. 4(b)), no nitrite was detected in the treated solution. Hu et al. [2] demonstrated that hypochlorite ions were formed in the presence of chloride ion during electrolysis. They also reported that the by-products of ammonia and nitrite were oxidized to form nitrogen gas and nitrate, respectively. However, this pathway was not confirmed in the present study. Meanwhile, nitrite was detected only in the presence of NaF (0.5 g/L) and NaCl (1 g/L). Nitrite increased at the first time intervals, and then decreased to zero. This finding is in agreement with that reported by previous study [12], which proved that nitrite was a by-product of nitrate reduction, and that it was probably further reduced into nitrogen gas or ammonia or oxidized into nitrate again at the anode.

3.4. Influence of chloride concentration on electrochemical denitrification

The effect of the indirect chloride-mediated denitrification was experimentally checked in specific experimental runs, in which nitrate feed solutions were prepared with different initial chloride concentrations. As shown in Fig. 5(a), nitrate was reduced during the experimental time for the three initial chloride concentrations of 700, 1,400, and 2,500 mg/L, respectively. Chloride concentration did not have a significant influence on the kinetics of denitrification during the process, although the rates were slightly lower when a higher chloride concentration was used. This can be attributed to the faster oxidation kinetics of the formed nitrite to nitrate as the concentration of chloride ions increased. According to Fig. 5(b), chloride concentration did not decrease with time, and it was not completely removed in all the studied cases. Furthermore, free chlorine disappeared since the first moment; its maximum concentration was 0.5 mg/L and it did not depend on the initial chloride concentration at all. In a reported pathway for effect on chloride ion on NRE, it has been assumed that nitrate is reduced because of the hypochlorite reaction with ammonia consequence of chlorine generated at the anode, as expressed below.

$$2NH_4^+ + 3HClO \rightarrow N_2 + 3H_2O + 5H^+ + 3Cl^- \quad (18)$$

According to Fig. 5(b), chloride ion was not removed at high level during treatment, so this pathway was not confirmed in this study. Chloride ion concentrations decreased with initial pH levels; specifically from 600 to 200 mg/L and from 220 to 150 mg/L for pH levels 4 and 9, respectively. We assumed that the mentioned pathway took place for the pH levels. Free chlorine in pH = 9 decreased from 1.5 to 0.5 mg/L, while it was decreased from 1 mg/L to zero for pH = 4 at the end of the treatment time. Free chlorine at the end of treatment time was only detected in pH = 9.

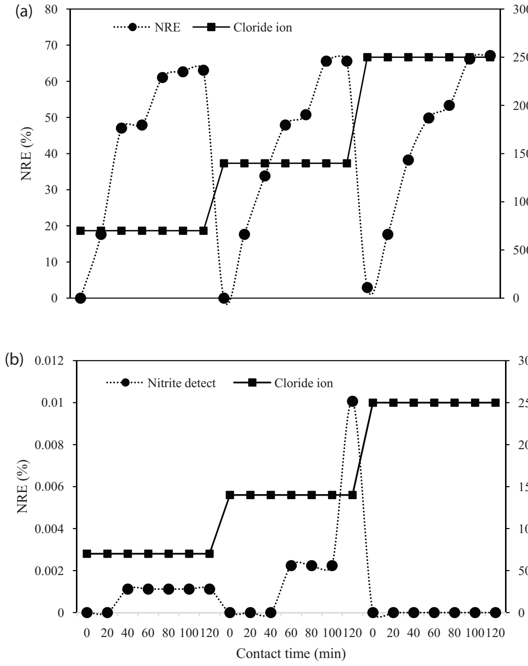

Fig. 5. (a) and (b) Influence of chloride concentration on the denitrification and chloride ion removal.

3.5. Effect of pH

In this study, the effect of pH was studied by performing electrochemical processes at pH levels 4, 7, and 9, using the same experimental conditions as those in the previous steps. The results of different initial pHs on the nitrate reduction are shown in Fig. 6(a). As can be seen, the tendencies of denitrification were similar at different initial pH levels; the NREs increased to 54.4% in 120 min for pH = 4, which had the highest NRE among the initial pH levels. At pH = 7, no nitrite was detected in the treated solution. As the solutions became more alkaline in the presence of NaCl, of the pH did not increase due to the production of H^+, which matched the production of OH^- ion. Cattarin [24] found that the electrochemical reduction of nitrate in highly alkaline solutions with Cu electrode mainly produced ammonia at a very negative potential. This finding is not in agreement with our experimental data. Such a disagreement can be due to the different conditions in our experiments, especially the electrode materials used. In our experiments, we clearly demonstrated through the obtained data that nitrate was mainly reduced to nitrite and finally to N_2. NH_3 was not produced, so data were not shown. At the initial pH = 4.0, unlike other electrochemical nitrate reduction methods, the deposited corroded anode on the cathode was not observed during the electrolysis. We assumed that the cathode passivation in the used electrochemical system did not occur. If an iron electrode was used instead of a CC electrode, the theoretical amount of electrode dissolved can be obtained by using Faraday's law given by Eq. (19) [25]:

$$C = \frac{I.t.M}{Z.F.V} \quad (19)$$

In this equation, C (g/L) is the corroded iron electrode, I (A) is the current amount (A), t is the contact time (s), M (g/mol) is the molecular weight of the electrode, Z is the chemical equivalence, F is the Faraday constant (96,500 C/mol), and V (L) is the volume of the treated solution. The amount for the iron electrode would be 11.2 mg/L for I = 10 mA, whereas no amount would be recorded for CC.

Owing to the fact that pH depended on initial pH levels, we can say that the surface charge of SS 304 and CC is pH dependent. Depending on the adjusted initial pH value, the electrode surface can be protonated (positive charge of the protons) or deprotonated/hydroxylated (negative charge of hydroxyl ions). Point of zero charge (pH_{PZC}) for a given mineral surface is the pH value at which this surface has a net neutral charge (H^+ is equal with the HO^- number). In this study, pH_{PZC} for SS 304 and CC were 5 vs. 5.5, respectively (Fig. 6(b)). Both electrodes have a positive charge at an initial pH = 4, which led to the adsorption of nitrate ion with negative charge. This may be the main reason why a higher denitrification rate was recorded for the initial pH = 4. Meanwhile, for pHs = 7 and 9, due to the higher pH_{PZC}, the electrode surface tend to have a negative charge, leading to repulsive force enhancement instead of adsorptive force among the electrodes and nitrate ions. This can be a reasonable explanation for the lower NREs in these initial pHs.

3.6. Electrical energy consumption

Electrical energy is an important parameter in evaluating the overall performance of a technology, such as an electrochemical process [15] for treating an aqueous solution. Using this equation, the electrical energy consumption in the studied electrical currents and voltages was calculated [25,26].

$$E\left(\text{kWh/m}^3\right) = \frac{U(v).I(A).t(h)}{V(L)} \tag{20}$$

where E is the electrical energy consumption; U and I are the applied electrical voltage and current; respectively; t is the contact time; and V is the treated solution volume.

According to Fig. 7, there was an uptrend in the electrical consumption as the applied current increased. Results showed that the highest denitrification rate was obtained at 10.28 kWh/m³ energy consumption at a current of 10 mA. Hence, a reasonable solution to decrease energy consumption is to reduce voltage by enhancing electrolyte strength (data not shown); however, this can lead to an increase in electrode corrosion.

3.7. Reaction kinetics

The rate of kinetic was studied for the electrochemical denitrification process. To obtain kinetic value, $\log_{10}\left(\frac{C_t}{C_i}\right)$ is plotted against time, where C_t is the concentration of nitrate at time t, and C_i is the initial nitrate concentration. From the slope of the obtained straight line and comparing with $\log_{10}\left(\frac{C_t}{C_i}\right) = -\frac{kt}{2.303}$, the k value was calculated in h^{-1} (Table 1).

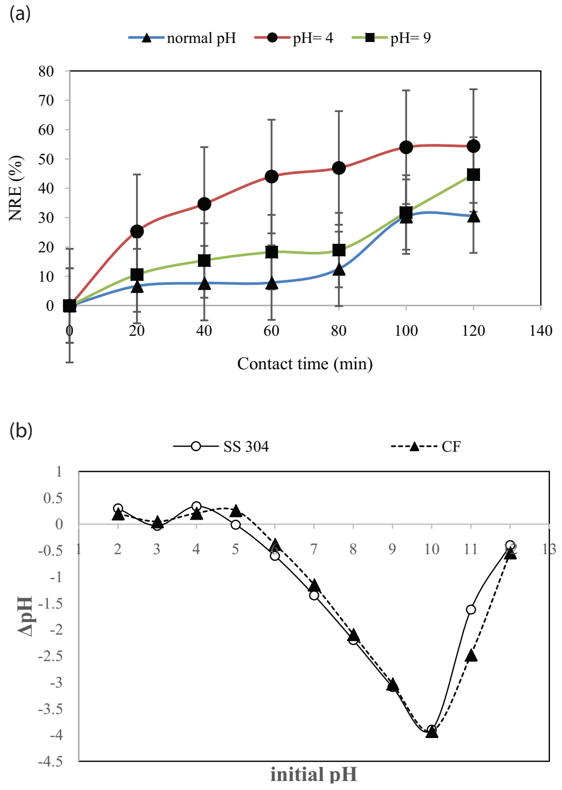

Fig. 6. (a) Nitrate removal efficiencies under different initial pH levels. (b) Zero point charge pH determination of SS 304 and CC electrodes.

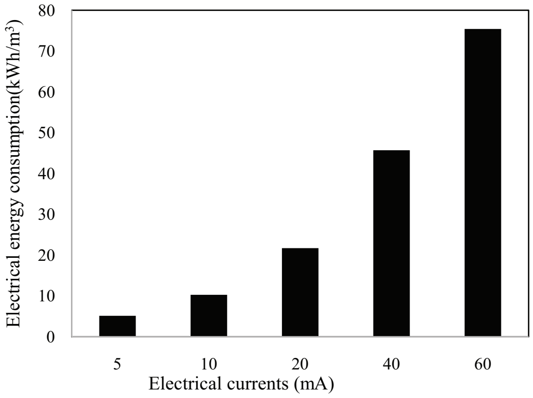

Fig. 7. Electrical energy consumption.

Table 1
Rate constants of different conditions in the kinetic study of electrochemical denitrification

Concentration (mg/L)	k (h^{-1})	NaCl (g/L)	k (h^{-1})	NaF (g/L)	k (h^{-1})	I (mA)	k (h^{-1})	pH	k (h^{-1})
50	0.000434216	0	0.000695	0.5	0.000347	5	0.00026053	4	0.001563
60	0.000694746	0.5	0.002432	1	0.000261	10	0.000694746	7	0.000695
100	0.000434216	1	0.002605			20	7.81589E–05	9	0.000955
150	3.47373E–05	2	0.002692			40	0.000173686		
						60	0.000173686		

4. Conclusion

In the present study, the nitrate was removed by electrochemical process using a CC as an anode electrode. The obtained results showed that very low electrode corrosion was observed. Based on the results, we have reached the following conclusions:

- The maximum nitrate reduction rate was obtained with current of 10 mA.
- Reaction kinetics among the studied parameters was highest at pH = 4, NaCl = 2 g/L, initial nitrate concentration of 60 mg/L, and current of 10 mA.
- Chloride concentration did not have a significant influence on the kinetics of denitrification during the reduction process.
- The less amount of hydrogen produced led to the low selectivity of ammonia formation and a high denitrification rate with a current of 10 mA.

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