

Investigation of the effect of different electrodes and their connections on the removal efficiency of 4-nitrophenol from aqueous solution by electrocoagulation

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Abstract

This study investigates the influence of variables on the removal efficiency of solution containing 4-NP (4-nitrophenol) by D. C. electrocoagulation (EC). The efficiency of different electrode connections and materials (steel 310, Fe, Al, graphite and steel 304) for 4-NP removal is compared. Current density, time of electrolysis, interelectrode distance, supporting electrolyte concentration and stirring rate of the solution were the variables that mostly influenced the 4-NP removal. Initially, a simple electrochemical cell was prepared with an anode and a cathode. Then the effect of each variable was studied separately using aqueous 4-NP in a batch mode. For a solution of 20 mg/L 4-NP + 300 mg/L NaCl with chemical oxygen demand (COD) of approximately 40 mg O₂/L, almost up to 99% 4-NP and 65% COD were removed, when the pH was about 9, time of electrolysis was approximately 10 min, current density was 100 A m⁻², interelectrode distance was 15 mm and stirring rate was 400 rpm. In the second series of experiments, the efficiency of EC cells with monopolar electrodes in series and parallel connections and an EC cell with bipolar electrodes was compared with that of a simple electrochemical cell. The best results obtained when steel 310 and Fe are used as anodes and employing Al and graphite as anodes would not be satisfactory. Also findings show that the types of sacrificial electrodes are not very significant in the removal of 4-NP. In the real wastewater obtained from Tabriz petrochemical plant 52% removal could be achieved after 10 min with using steel 310 as anode and steel 304 as cathode.

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Keywords: Electrocoagulation; Electrode connection; Electrode material; 4-nitrophenol

1. Introduction

Oil refineries, coke plants, chemical and plastic industries release phenolic compounds into the wastewater. These compounds are considered as one of the 65 major pollutants [1]. Nitrophenols are among the most common organic pollutants in industrial and agricultural wastewater [2]. These compounds are involved in the synthesis of many chemicals, particularly in the field of pesticides [3]. Also, nitrophenols are considered as hazardous wastes and priority toxic pollutants by the U.S. Environmental Protection Agency [4]. It is therefore important to assess the fate of these compounds in the environment and develop effective methods to remove them from water before discharge.

The most common treatment of industrial wastes are biological treatment, adsorption on activated carbon and chemical methods which are traditional methods [5,6] and cannot effectively destroy the pollutants.

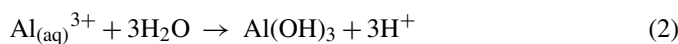
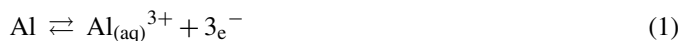
The electrocoagulation (EC) technique is considered to be potentially an effective tool for treatment of textile wastewaters with high removal efficiency [7–10]. This technique has recently attracted a great deal of attention. EC process provides a simple, reliable and cost-effective method for the treatment of wastewater without any need for additional chemicals. The following physicochemical reactions may also occur in the EC cell [11]:

- Cathodic reduction of impurities present in wastewater.
- Discharge and coagulation of colloidal particles.
- Electrophoretic migration of the ions in solution.
- Electroflotation of the coagulated particles by O₂ and H₂ bubbles produced at the electrodes.

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- Reduction of metal ions at the cathode.
- Other electrochemical and chemical processes.

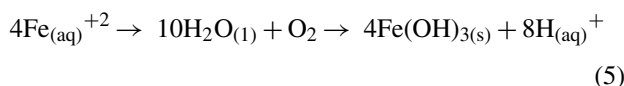
The electrolytic dissolution of aluminum anode yields the cationic monomeric species such as Al^{3+} and $\text{Al}(\text{OH})_2^+$ at low pH, which at appropriate pH values are converted into $\text{Al}(\text{OH})_3$ and eventually polymerized to $\text{Al}_n(\text{OH})_{3n}$ according to the following reactions:



In an electrolytic process iron is oxidized to iron hydroxide, $\text{Fe}(\text{OH})_n$, where n is 2 or 3 and two mechanisms have been proposed for the formation of $\text{Fe}(\text{OH})_n$ [12].

• Mechanism 1.

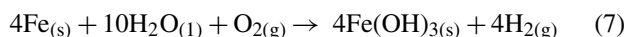
Anode:



Cathode:

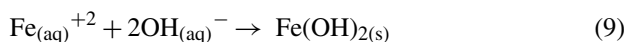


Overall:

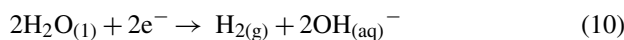


• Mechanism 2:

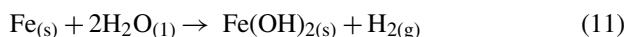
Anode:



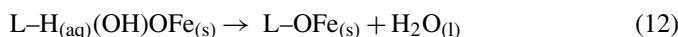
Cathode:



Overall:



The produced $\text{Fe}(\text{OH})_{n(\text{s})}$ remains in the aqueous stream as a gelatinous suspension, which can remove the pollutants from wastewater either by complexation or by electrostatic attraction followed by coagulation [12]. In the case of surface complexation the pollutant acts as a Ligand (L) to bind to hydrous iron:



Furthermore, in many wastewaters which contain chlorides, another strong oxidant of hypochlorite may be formed as given in Eq. (13) [13,14].

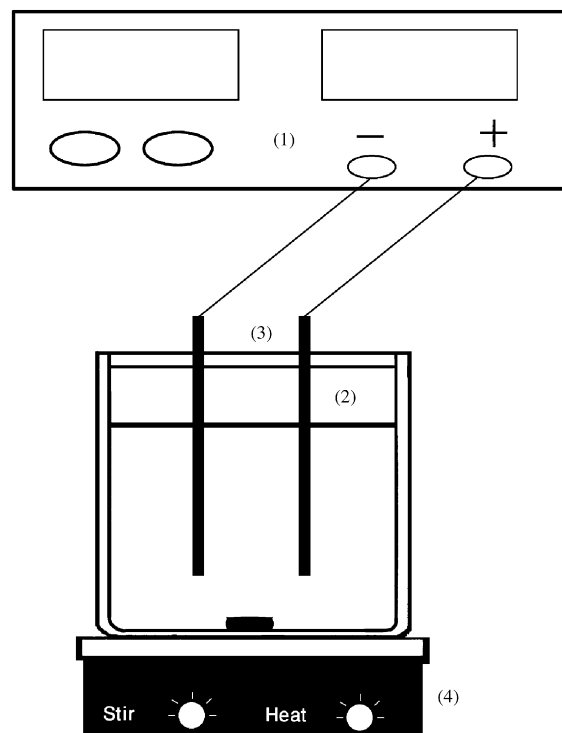
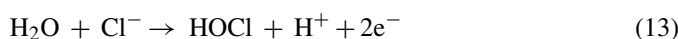


Fig. 1. An apparatus cell. (1) DC power, (2) Electrolysis cell, (3) Electrodes pair and (4) Magnetic stirrer.

In our previous work electrocoagulation process using iron and aluminum electrodes were used to investigate the removal of dye solution [12]. In this study electrocoagulation process employing different electrodes and different connections have been investigated to degrade 4-NP, which is mainly used in the manufacture of dyestuffs [15].

2. Experimental

2.1. Materials and methods

The experiments were carried out on 4-NP (obtained from Fluka) solutions with concentration of 20 mg/L which was freshly prepared. At the beginning of each run 250 ml solution of the desired concentration was poured into the reactor and NaCl was used as an electrolyte to enhance the ionic strength of the solution. In all experiments pH was kept constant at about 9 using 0.1 N NaOH because at this pH, 4-NP can be easily measured with spectrophotometer at 400 nm. The electrodes were placed inside a Pyrex glass reactor with a distance of 15 mm between anode and cathode in all EC cells with different connections. The dimensions of electrodes were 40 mm × 70 mm × 2 mm. The total submerged area of each electrode was 18.7 cm². In order to allow easy stirring the sufficient distance between the bottom of the electrodes and the bottom of the cell was allowed. All experiments were carried out in the above reactor as shown in Figs. 1–4 [11], and a DC-power supply (REC-P-6, (0–50) V and 0–5 A) and magnetic stirrer, flat-plate anode and cathode were employed. For comparative purpose, we worked out and applied in practice an electrocoagulator with Fe, steel 310, Al

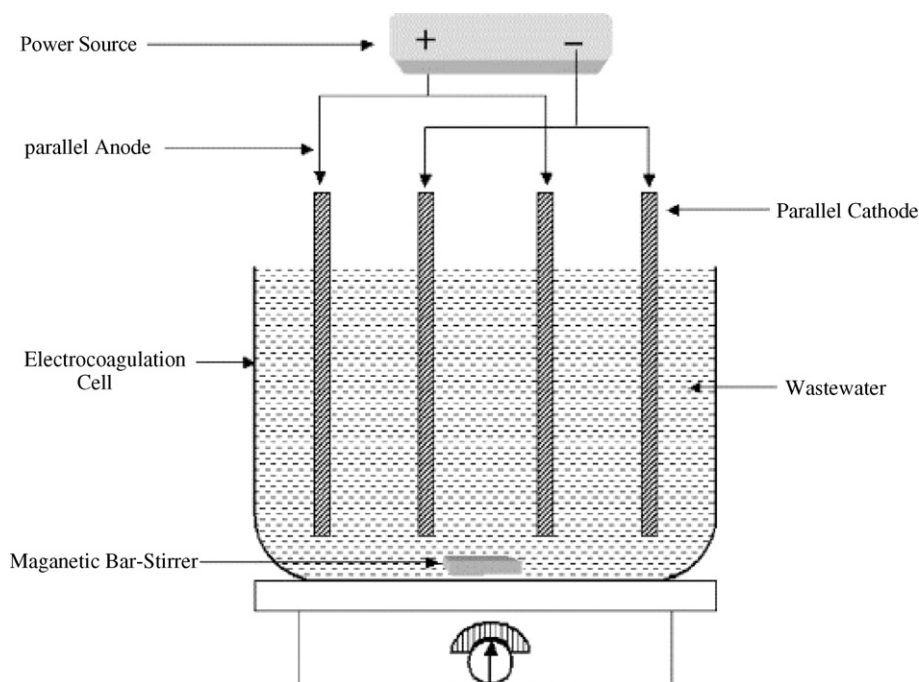


Fig. 2. Bench-Scale EC reactor with monopolar electrodes in parallel connections.

and graphite anodes, and also steel 304, graphite, Fe and Al cathodes. Before each experiment the electrodes were washed for 2 min with 0.1 N NaOH, H₂SO₄ solutions and distilled water. The solution was poured into a cylinder and left for 25 min. Then it was filtered using Ø 125 mm filter paper purchased from Schleicher & Schull, and the absorbance was read. All experiments were performed at room temperature. A real wastewater obtained from Tabriz petrochemical plant containing phenol and its derivatives such as ortho hydroxyphenol and experi-

mental solution was prepared using the above waste along with 4-NP.

2.2. Chemical analysis

The 4-NP concentration was determined by spectrophotometer at $\lambda_{\text{max}} = 400 \text{ nm}$ according to Beer–Lambert's law, using a Ultrospec 2000 Biotech Pharmacia UV/vis spectrophotometer. Conductivity (κ) was measured using a conductivity

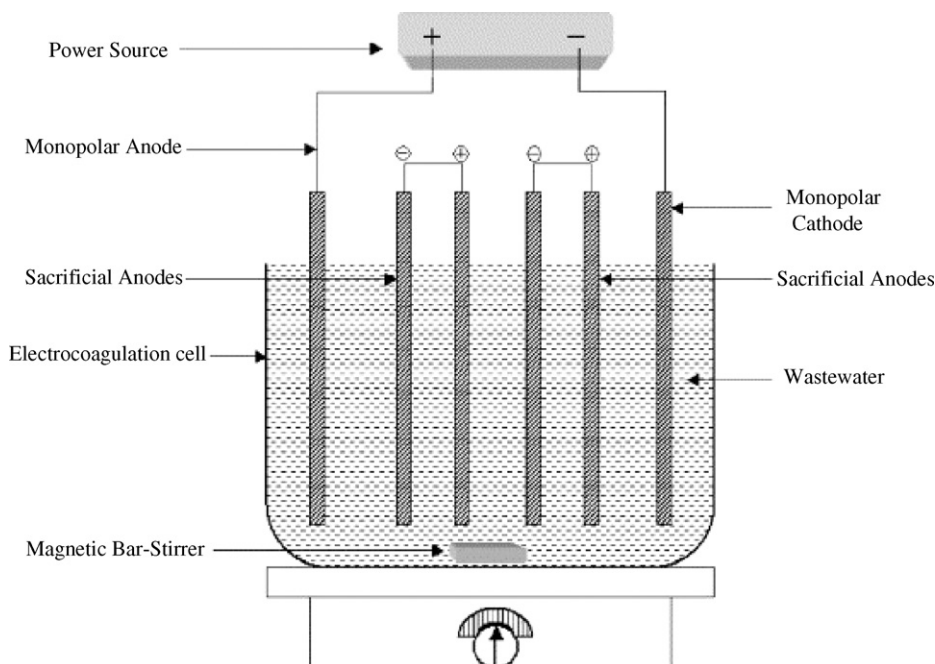


Fig. 3. Bench-scale EC reactor with monopolar electrodes in series connections.

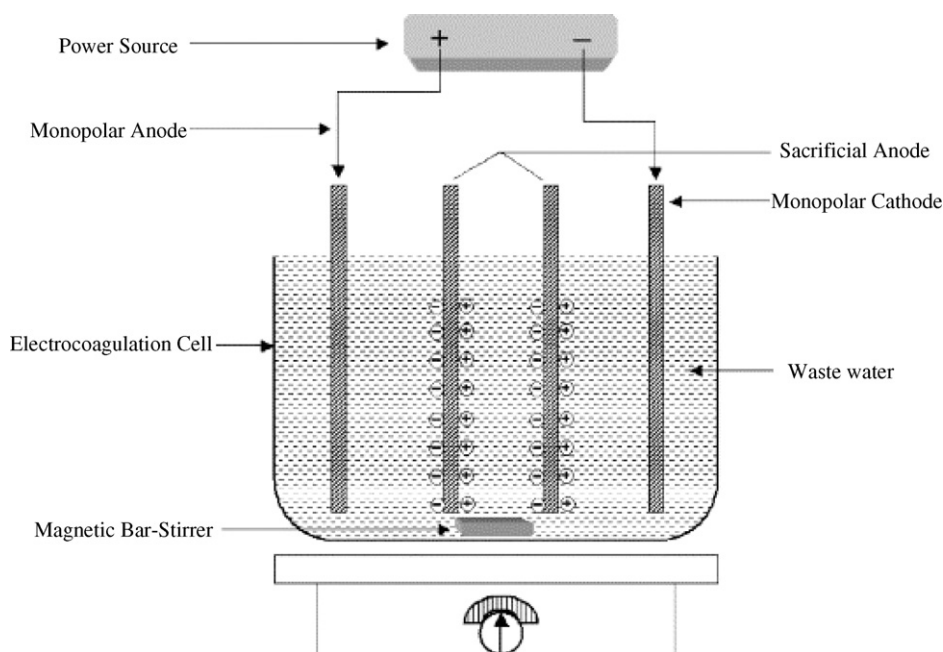


Fig. 4. Bench-scale EC reactor with bipolar electrodes in parallel connections.

meter (Horiba). High-performance liquid chromatograms were recorded on an HPLC (PerkinElmer Series 200). A Spheri- 5 RP- 18 column with dimension of 220 mm \times 4.6 mm and with 5 μ m particle size and UV/vis detector with the wavelength set at 210 nm was used. The mobile phase was a mixture of methanol–water (H_3PO_4 5 Mm) 35/65 (v/v) at a flow rate of 1 ml min⁻¹.

3. Results and discussion

3.1. The effect of current density

In all electrochemical processes, current density is the most important parameter for controlling the reaction rate within the reactor [16]. It is well known that the amount of current density determines the coagulant production rate, and adjusts the rate and size of the bubble production, and hence affects the growth of flocs [12,16,17].

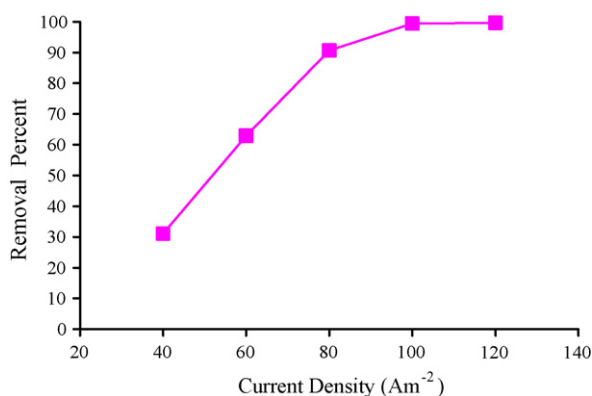


Fig. 5. Effect of current density on the removal efficiency of 4-NP with steel 310/steel 304 electrode pair. ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min).

Hence, the effect of current density on the pollutants removal was investigated. As shown in Fig. 5, an increase in current density from 40 to 120 A m⁻² yields an increase in the efficiency of 4-NP removal from 22.7 to 99.6% because when the current density increases the efficiency of ion production on the anode increases. Therefore, there is an increase in bubble generation rate, floc production and decrease of bubble size in the solution, which are beneficial for high pollutant removal efficiency by H₂ flotation [18]. For a solution with a 4-NP concentration of 20 mg/L, the optimum current density was found to be 100 A m⁻².

3.2. The effect of electrolysis time

The effect of time was studied at constant current density of 100 A m⁻² and pH 9. The 4-NP removal efficiency depends directly on the concentration of ions produced by the electrodes. When the electrolysis period increases, an increase occurs in the

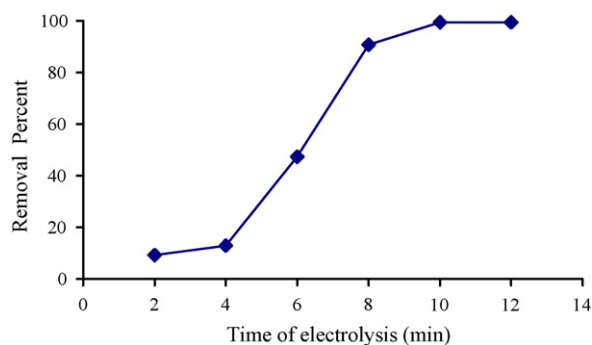


Fig. 6. Effect of the time of electrolysis on the removal efficiency of 4-NP with steel 310/steel 304 electrode pair. ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $[i] = 100$ A m⁻², $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm).

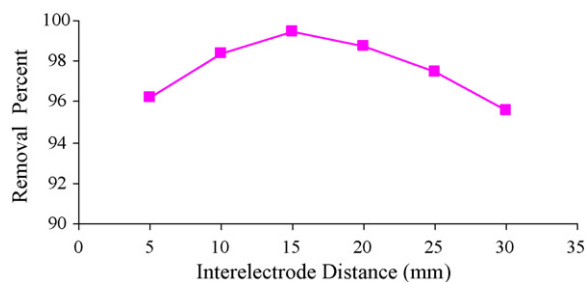


Fig. 7. Effect of the interelectrode distance on the removal efficiency of 4-NP with steel 310/steel 304 electrode pair. ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm).

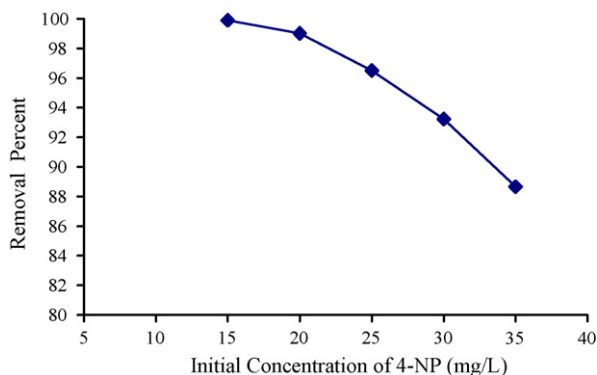


Fig. 8. Effect of the initial concentration of the 4-NP on the removal efficiency of 4-NP with steel 310/steel 304 electrode pair. ($C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², $d = 1.5$ cm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm).

concentration of ions and their hydroxide flocs. Fig. 6 shows the relationship between the 4-NP removal efficiency and the electrolysis time. According to the results for a solution having a 4-NP concentration of 20 mg/L, and a treatment unit with current density of approximately 100 A m⁻², 10 min of operating time is sufficient for nearly complete removal efficiency of 99.4%.

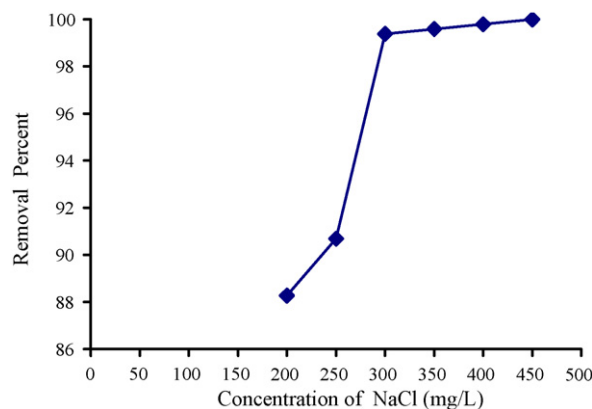


Fig. 9. Effect of NaCl on the removal efficiency of 4-NP with steel 310/steel 304 electrode pair. ($C_{0[4-NP]} = 20$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min).

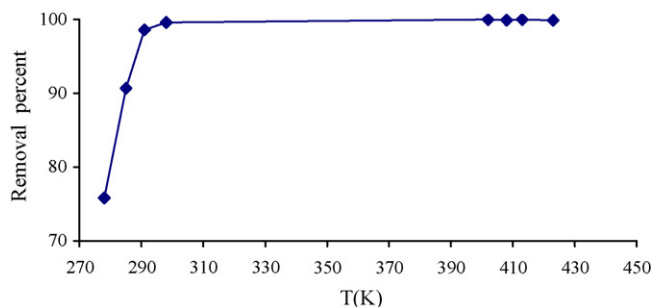


Fig. 10. Effect of the temperature on the removal efficiency of 4-NP with steel 310/steel 304 pair. ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², $d = 1.5$ cm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm).

3.3. The effect of the interelectrode distance

When the interelectrode distance increases, the electrical current decreases. To achieve a certain current density, the voltage must be increased. As shown in Fig. 7, with decreasing distance from 50 to 15 mm, the removal efficiency of 4-NP increases. This change probably occurs because the electrostatic effects depend on the interelectrode distance, so when this distance increases, the movement of produced ions would be slower and they would have more opportunity to aggregate and produce flocs. But with decreasing distance more than 15 mm, less interaction of ions with hydroxyl polymers is expected. In the other words, decreasing of local concentration and electrostatic attraction are the reasons for decreasing the removal of 4-NP.

3.4. The effect of initial concentration of 4-NP

As shown in Fig. 8, the removal efficiency decreases from 99.9 to 88.7% by increasing the concentration of 4-NP from 15 to 35 mg/L. One of the most important pathways of 4-NP removal by EC process is adsorption onto metallic hydroxide flocs and the adsorption capacity of flocs is limited at higher concentrations [12].

According to the results, above the concentration of 20 mg/L the adsorption capacity of flocs becomes exhausted. On the other hand due to the presence of intermediate products formed during

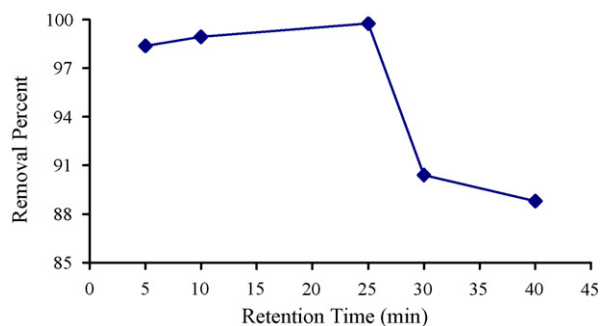


Fig. 11. Effect of the retention time on the removal efficiency of 4-NP with steel 310/steel 304 electrode pair. ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, $\kappa = 0.706$ mS/cm).

Table 1

The effect of the electrode material on the removal efficiency of 4-NP

Anode	Steel 310	Al	Steel 310	Al	Graphite	Graphite	Fe	Fe	Graphite	Steel 310	Fe	Graphite
Cathode	Steel 304	Al	Al	Steel 304	Fe	Steel 304	Steel 304	Al	Al	Graphite	Graphite	Graphite
R%	99.4	16.2	95.4	12.1	56	59.5	98	98.8	54.5	97.8	97.4	76

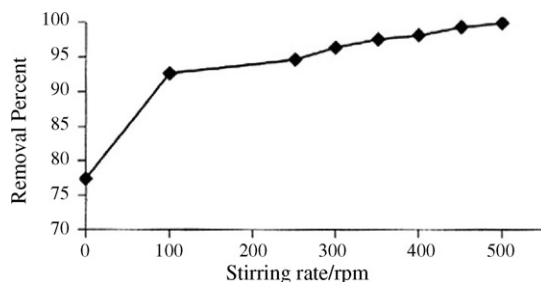
pH 9, $\kappa = 0.706$ mS/cm.

Fig. 12. Effect of the stirring rate on the removal efficiency of 4-NP with steel 310/steel 304 electrode pair. ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm).

the electrolysis at higher concentrations, which could compete with 4-NP and water for the active sites on the electrode [12].

3.5. The effect of the concentration of NaCl

When the concentration of NaCl in solution increases, solution conductivity and current density would be raised. Consequently, with respect to [19]:

$$V = E_C - E_A - |\varepsilon_A| - |\varepsilon_C| - IR_{cell} - IR_{circuit}$$

The necessary voltage to access to a certain current density will be diminished, so the consumed electrical energy is decreased. Similar kind of effect of increase in removal efficiency with increase in conductivity was also reported by Lin and Peng [20,21], and Daneshvar et al. [19]. For lower concentration, there is decrease of that rate, probably because there are not enough ions to conduct the current. Thus, it may be concluded that increasing the electrical resistance of the solution would decrease the efficiency of the process. The effect of NaCl concentration on the removal efficiency is shown in Fig. 9. It

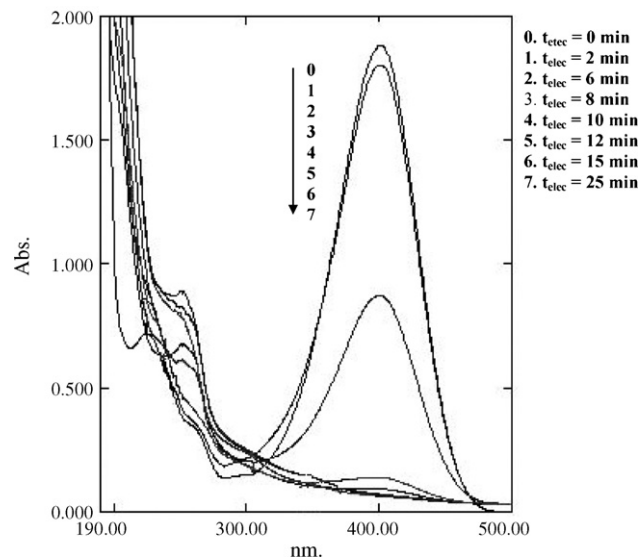


Fig. 14. Spectral changes of 4-NP during electrolysis with steel 310/steel 304 electrode pair, ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm).

can be seen that there is an increase in the removal efficiency up to 100%.

3.6. The effect of temperature

The electrochemical reaction rate like any other chemical reaction rates increases when temperature of solution increases. As shown in Fig. 10, when the temperature increases, the efficiency of 4-NP removal increases slightly. The reason could be due to increase in mobility and collision of ions with hydroxide polymer.

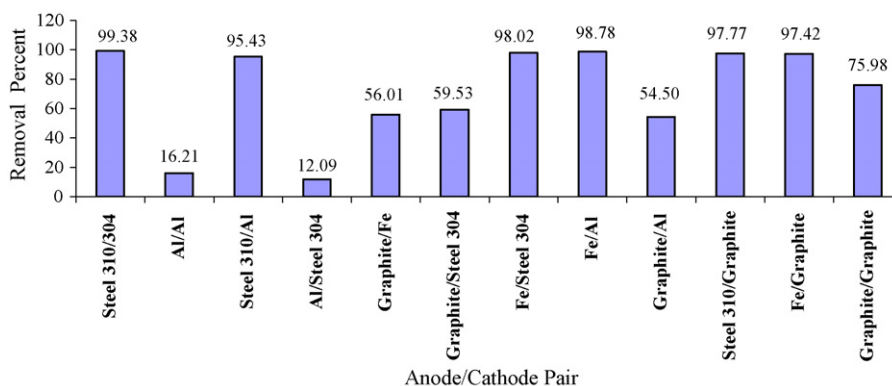


Fig. 13. Effect of the electrode material on the removal efficiency of 4-NP ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $t_{elec.} = 10$ min, $[i] = 100$ A m⁻², $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm).

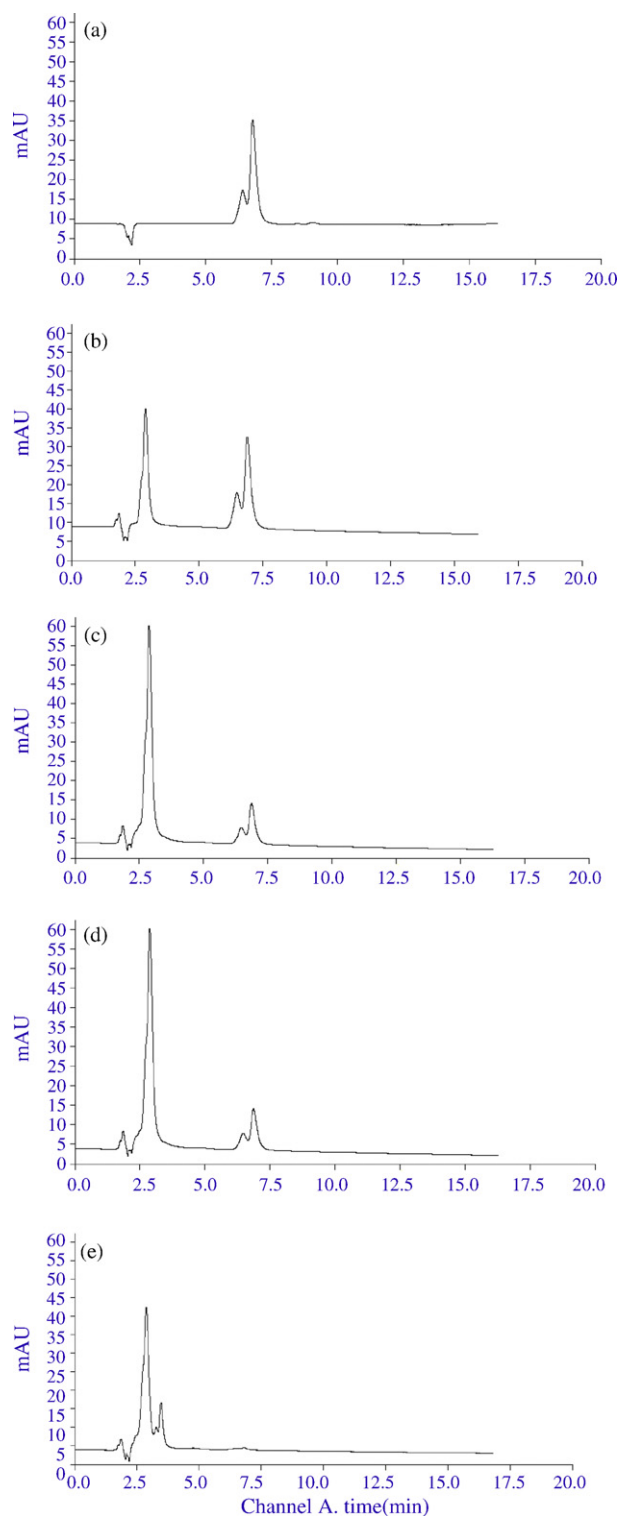


Fig. 15. The HPLC Chromatograms of 4-NP removal and formation of intermediates with steel 310/steel 304 electrode pair ($C_{0[4-NP]} = 20$ mg/L, $C_{NaCl} = 300$ mg/L, $[i] = 100$ A m⁻², $d = 15$ mm, pH 9, $\lambda_{max} = 400$ nm, retention time = 25 min, $\kappa = 0.706$ mS/cm). a; (0 min), b; (2 min), c; (6 min), d; (8 min), e; (10 min).

Table 2

The effect of material and different electrode connections in presence of one and two sacrificial electrodes on the removal efficiency of 4-NP

	Electrode connections	Electrodes material	Total electrodes	R%
1	Series	Monopolar electrodes Sacrificial electrode: Al Anode: steel 310 Cathode: steel 304	4	100
2	Series	Monopolar electrodes Sacrificial electrode: Fe Anode: Fe Cathode: steel 304	4	100
3	Series	Monopolar electrodes Sacrificial electrode: Al Anode: Al Cathode: Al	4	15.4
4	Series	Monopolar electrodes Sacrificial electrode: Fe Anode: Al Cathode: Fe	4	10.8
5	Series	Monopolar electrodes Sacrificial electrode: Al Anode: Al Cathode: Fe	4	16.3
6	Series	Monopolar electrodes Sacrificial electrode: steel 310 Anode: Fe Cathode: graphite	4	98.9
7	Parallel	Monopolar electrodes Sacrificial electrode: Fe Anode: steel 310 Cathode: steel 304	4	100
8	Parallel	Bipolar electrodes Sacrificial electrode: Fe Anode: Fe Cathode: steel 304	4	100
9	Parallel	Bipolar electrodes Sacrificial electrode: Al Anode: Fe Cathode: steel 304	4	96.1
10	Parallel	Bipolar electrodes Sacrificial electrode: Fe Anode: Al Cathode: Fe	4	23.7
11	Parallel	Monopolar electrodes Sacrificial electrode: steel 310 Anode: steel 310 Cathode: graphite	4	99.2
12	Parallel	Monopolar electrodes Sacrificial electrode: steel 310 Anode: steel 310 Cathode: steel 304	3	99.6
13	Parallel	Monopolar electrodes Sacrificial electrode: steel 310 Anode: steel 310 Cathode: graphite	3	97.6
14	Parallel	Monopolar electrodes Sacrificial electrode: steel 310 Anode: steel 310 Cathode: Fe	3	99.2
15	Parallel	Monopolar electrodes sacrificial electrode: steel 310 Anode: graphite Cathode: steel 304	3	74.4

pH 9, $\kappa = 0.706$ mS/cm.

3.7. The effect of retention time

At the end of the electrocoagulation process, the sample was poured into a 250 ml graduated cylinder for precipitation of flocs. When retention time is increased (up to 25 min), the removal efficiency of 4-NP increases.

After 25 min adsorbed contaminated particles would be released into the solution due to saturation and consequently the removal efficiency is decreased. The ease of desorption of the adsorbed contaminated particles can be the reason for the physical adsorption mechanism. This result is shown in Fig. 11.

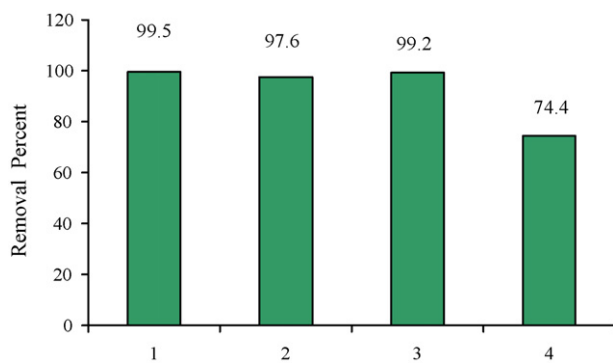


Fig. 16. The effect of material and different electrode connections in presence one sacrificial electrode on the removal efficiency of 4-NP ($C_{0[4-NP]} = 20 \text{ mg/L}$, $C_{NaCl} = 300 \text{ mg/L}$, $t_{elec.} = 10 \text{ min}$, $[i] = 100 \text{ A m}^{-2}$, $d = 15 \text{ mm}$, pH 9, $\lambda_{max} = 400 \text{ nm}$, retention time = 25 min, $\kappa = 0.706 \text{ mS/cm}$). For the types of electrodes refer to specified number in Table 2.

3.8. The effect of stirring rate

As shown in Fig. 12, the removal percent of 4-NP increased significantly with increasing the stirring rate. With increasing stirring rate, flocs are formed and attached together, and precipitation becomes easier.

3.9. The effect of the electrode material

Electrode assembly is the heart of the present investigation [22]. Therefore the selection of materials is very important. Under exactly the same conditions, the electrolysis of 4-NP solution was performed with different electrodes. The results are presented in Table 1 and Fig. 13.

It was observed that steel 310 and Fe are effective, compared to graphite and Al. It could be concluded that the soluble electrodes are particularly effective in removing 4-NP. The contaminated particle from solution using Al electrodes is mainly removed by electrocoagulation, while the 4-NP removal by Fe and steel 310 electrodes is due to the collective effect of electrocoagulation and electrooxidation and also due to the less solubility of Al it was not as effective. Another possible reason for this behavior could be the adsorption capacity of hydrous aluminum oxide for contaminants which is much lower than hydrous ferric oxides. The removal of 4-NP solu-

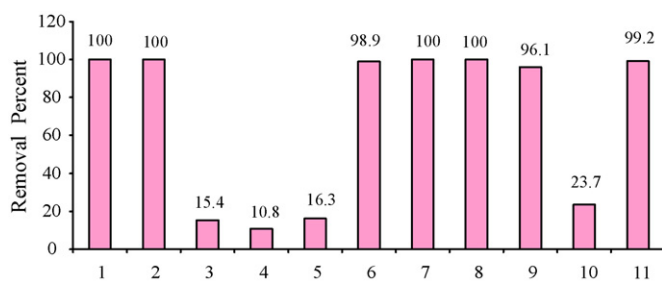


Fig. 17. The effect of material and different electrode connections in presence two sacrificial electrode on the removal efficiency of 4-NP ($C_{0[4-NP]} = 20 \text{ mg/L}$, $C_{NaCl} = 300 \text{ mg/L}$, $t_{elec.} = 10 \text{ min}$, $[i] = 100 \text{ A m}^{-2}$, $d = 15 \text{ mm}$, pH 9, $\lambda_{max} = 400 \text{ nm}$, retention time = 25 min, $\kappa = 0.706 \text{ mS/cm}$). For the types of electrodes refer to specified number in Table 2.

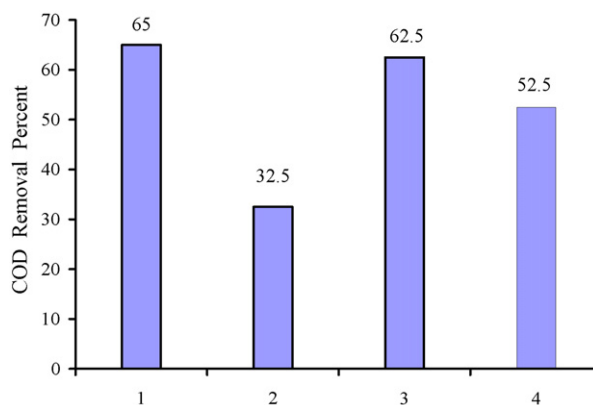


Fig. 18. Effect of cathode material and different electrode connections on the COD reduction ($C_{0[4-NP]} = 20 \text{ mg/L}$, $C_{NaCl} = 300 \text{ mg/L}$, $t_{elec.} = 10 \text{ min}$, $[i] = 100 \text{ A m}^{-2}$, $d = 15 \text{ mm}$, pH 9, $\lambda_{max} = 400 \text{ nm}$, retention time = 25 min, $\kappa = 0.706 \text{ mS/cm}$). For the types of electrodes refer to specified number in Table 2.

tion as a function of time for electrocoagulation treatments with steel 310/steel 304 as electrodes pairs was followed with UV/vis spectrophotometer, and shown in Fig. 14. Also, the changes of removal of 4-NP solution for electrocoagulation treatment with steel 310/steel 304 (anode/cathode) was recorded on an HPLC, shown in Fig. 15.

The 4-NP removal process may be due to the interaction of the 4-NP molecules with iron hydroxides which can remove the 4-NP from solution either by surface complexation or electrostatic attraction.

3.10. The effect of different electrode connections

As can be seen from Table 2 and Figs. 16 and 17, the best results could be obtained when steel 310 and Fe are used as anode while employing Al and graphite as anode would not be satisfactory. This result could also be due to the differences in the solubility of the above mentioned electrodes and also because of adsorption capacity of formed hydroxides. A comparison of the findings shows that the types of sacrificial electrodes were not very significant in our study and the types of connections have been reported in Table 2.

3.11. COD removal

Mineralization of 4-NP solution in this process was monitored by COD reduction. COD values have been related to the total concentration of organics in the solution. Results show that 65% COD reduction of 4-NP with initial concentration of 20 mg/L after 10 min of electrolysis with 300 mg/L NaCl and current density of 100 A/m². The results are shown in Fig. 18.

3.12. Removal of 4-NP in real wastewater by electrocoagulation process

Removal of 4-NP by electrocoagulation process in real wastewater was studied with a sample collected from Tabriz petrochemical plant containing 4-NP and other phenolic

compounds using steel 310 as anode and steel 304 as cathode. Results show that 52% removal of 20 mg/L of 4-NP at pH 9 and current density being 100 A/m², interelectrode distance of 15 mm and stirring rate of 400 rpm could be achieved after 10 min and increasing reaction time to 12 min, the removal percent of 4-NP only increases to 56%.

4. Conclusions

The removal of 4-NP solution by means of electrocoagulation was affected by the current density, time of electrolysis, interelectrode distance and stirring rate. The results showed that for 20 mg/L 4-NP solution with COD of 40 mg O₂/L and NaCl concentration of 300 mg/L, 4-NP and COD elimination of up to 99% and 65% were obtained respectively, when the pH was about 9, time of electrolysis was approximately 10 min, current density was 100 A m⁻² at temperature of 298 K, interelectrode distance of 15 mm and stirring rate of 400 rpm.

The experimental results showed that more soluble electrodes (Fe and steel 310) as anodes would appear to maintain a better removal. Also, the findings show that the types of sacrificial electrodes were not very significant in removal of 4-NP. Because of relatively short reaction times and also easy handling, electrocoagulation process can be utilized as an attractive process for removal of 4-NP in the real wastewater.

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