

THE INFLUENCE OF THE INITIAL CONCENTRATION OF HEAVY METALS AND THE ELECTRODE MATERIAL ON THE EFFICIENCY OF ELECTROCOAGULATION WATER TREATMENT

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1 Abstract (Times New Roman, 11pt, bold)

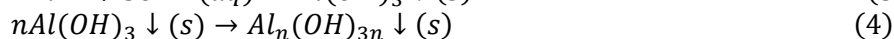
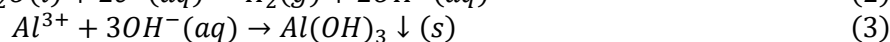
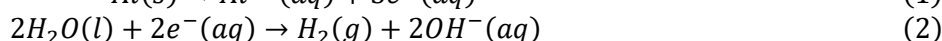
In this study, the influence of the electrode material and the initial concentration of heavy metals on the efficiency of their removal from water by the electrocoagulation process is investigated. The experiments were carried out in a batch reactor with aluminium, iron, copper and graphite electrodes at three different contaminant concentrations. For the conditions that yielded the highest efficiency, a set of experiments was conducted with perforated electrodes. The process was more successful at lower concentrations, and the material of the electrodes had the greatest influence.

Keywords: water treatment, heavy metals, electrocoagulation, kinetics, design of experiment.

2 Introduction

Pollution by heavy metals has become one of the most important environmental issues in recent years. The term "heavy metals" refers to all metals and metalloids with relatively high density such as iron (Fe), manganese (Mn), aluminium (Al), mercury (Hg), zinc (Zn), cadmium (Cd), arsenic (As), antimony (Sb), boron (B), thallium (Tl), chromium (Cr), copper (Cu), lead (Pb), and nickel (Ni) [1]. In nature, they are widely distributed as components of the earth's crust and as trace elements in living organisms. Although they occur naturally in soils and sediments, significant sources of heavy metals in the environment are human activities (agricultural, pharmaceutical, municipal, mining and metal processing waste and wastewater, fossil fuels consumption, etc.) and atmospheric sources like volcanic emissions and atmospheric dust [2]. Even though low concentrations of heavy metals are necessary for metabolism and normal growth and development of the organism, higher concentrations can have toxic and harmful effects, so the continuous release of heavy metals into the aquatic environment and increased human exposure are of concern [3], [4]. A major problem is the observed bioaccumulation in living organisms. Heavy metals enter the aquatic environment through natural or anthropogenic pathways and enter the food chain [1]. Since they are not biodegradable, they accumulate in living tissues, and pose a hazard with chronic exposure [5]. Arsenic, cadmium, chromium, lead, and mercury, for example, are considered systemic toxins because they can cause damage to several different organs, even at low levels of exposure [6]. They are also classified as carcinogens by the U.S. Environmental Protection Agency. Cadmium has been linked to "itai-itai" disease, respiratory and kidney problems, liver damage, anaemia, digestive issues, and inhibition of calcium control in the body [7]. Chromium can cause allergic dermatitis, nausea, vomiting and alopecia [8]. Excessive nickel consumption can result in anaphylaxis, red blood cell damage, chronic bronchitis, liver and kidney damage [9]. Exposure to high concentrations of cobalt can lead to hearing, vision, nerve, and thyroid problems [10]. The toxicity of heavy metals depends on the dose, route of administration, chemical form, as well as gender, age, weight, and genetics of the exposed individuals [5],[11].

To meet the growing demand for lower heavy metal levels in drinking water and wastewater, various water treatment processes are being investigated and developed. One of the promising technologies is electrocoagulation (EC). Although it is similar to chemical coagulation, EC is characterized by *in situ* generation of coagulants by passing an electric current through sacrificial electrodes. The most commonly used electrodes are electrodes made of iron and aluminium [12]. Under the influence of the electric current, the anodes dissolve, oxygen is released, and metal cations (Al^{3+} , Fe^{2+}) are formed [13],[14]. At the same time, hydrogen and hydroxide ions are released at the cathode. The hydroxide ions migrate to the anode, where they form polymeric iron and aluminium hydroxides with the metal cations, which act as coagulants. The reaction at the aluminium anode is given in Eq(1). The reaction at the cathode proceeds according to Eq(2). In the alkaline medium, aluminium hydroxide is formed as shown in Eq(3), while Eq(4) shows its polymerization.[15], [16]



The removal of pollutants from water occurs by chemical reactions and precipitation or by physical and chemical binding to colloidal metal hydroxides [17]. Thus, the process consists of dissolution of the sacrificial anode, formation of hydroxide ions and hydrogen at the cathode, electrolyte reactions at the electrode surface, adsorption of coagulants onto colloidal contaminants, and removal of the formed flocs by precipitation or flotation [18]. The flocs formed by this process are significantly larger, more stable, and contain less bound water, which greatly facilitates the separation of the resulting sludge, e.g., by filtration [18], [19]. Electrocoagulation is considered an environmentally friendly technology because it avoids the addition of chemical reagents and additives, thus preventing the formation of secondary contaminants [18],[20].

The following parameters affect the efficiency of EC in removing heavy metals from water: electrode material, solution pH, current density, treatment time, electrode potential, pollutant concentration, anion concentration, and temperature [21]. In this study, the influence of electrode material and initial pollutant concentration on the efficiency of electrocoagulation process was investigated. The experiments were statistically designed using the Design of Experiments approach (DOE). This approach can be used to determine the most important factors affecting the process and the values of these factors that allow optimization of the electrocoagulation process. These factors are independent variables, while response is a dependent variable. The main advantage of DOE is the significant reduction in the number of experiments to be performed, since the behavior of the interrelated factors can be predicted over a wide range of values. Thus, it is possible to obtain maximum information from a relatively small number of experimental data.

3 Methods

3.1 Design of experiments

Stat-Ease's Design Expert 12 software was used for experiment design and statistical analysis. The factors studied were the electrode material (Al, Fe, Cu, C) and the initial concentration of heavy metals (0.1, 1, 10 ppm), while the efficiency of the process was the response. The efficiency of the process was evaluated by measuring the change in heavy metals concentrations at the end of the process. It was calculated according to Eq(5), where C_0 is the initial concentration of the metal and C_t is the concentration of the metal measured at time t . The concentrations were measured by ICP-OES analysis.

$$\eta(\%) = \frac{C_0 - C_t}{C_0} * 100 \quad (5)$$

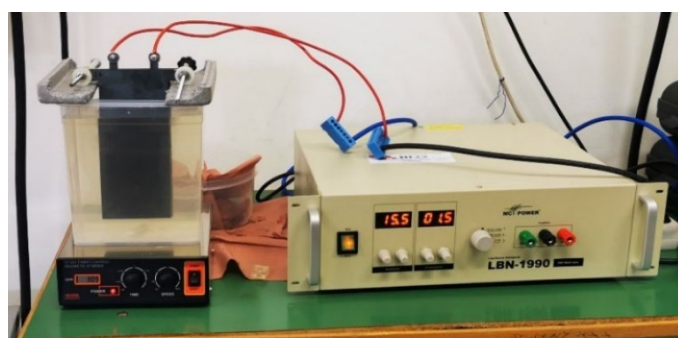
The experiments were carried out following the multi-level categoric experimental design. The obtained run matrix and responses are shown in Table 1.

Table 1. Experimental design matrix

Std	Run	Factor 1 A: material	Factor 2 B: concentration	Response 1 Mn, %	Response 2 Ni, %	Response 3 Cd, %	Response 4 Cr, %	Response 5 Co, %
6	1	Al	1	29.49	36.89	76.27	99.77	54.00
3	2	Cu	0.1	55.07	68.57	90.12	100	67.48
5	3	Fe	1	21.34	16.10	21.60	98.90	13.00
7	4	Cu	1	42.31	58.19	85.12	99.90	57.96
8	5	C	1	17.05	5.67	6.15	56.15	6.10
2	6	Al	0.1	35.80	53.20	78.52	100	68.00
1	7	Fe	0.1	41.02	57.93	89.63	99.60	64.71
9	8	Fe	10	6.89	8.95	13.09	97.80	7.76
10	9	Al	10	9.48	28.18	31.06	97.9	33.17
12	10	C	10	7.76	9.19	6.77	57.28	7.00
4	11	C	0.1	16.67	2.38	4.90	55.37	2.68
11	12	Cu	10	32.56	44.89	59.79	99.30	45.28

3.2 Experimental setup and procedure

Experiments were carried out in a 3-litre plexiglass batch reactor with two plate electrodes connected to the laboratory power supply. Mixing was achieved using a magnetic stirrer (set at 300 rpm) placed under the reactor. The experimental setup is shown in Figure 1. Four different electrode pairs (Figure 2) were used: Al/Al, Fe/Fe, Cu/Cu, and C/C. The active surface area or total immersed surface area of each electrode was 119 cm² and the distance between the electrodes was 1 cm. The experiments were performed with synthetic solutions containing Cd, Cr, Co, Mn, and Ni. Three different concentrations were tested: 0.1, 1, and 10 ppm. All other parameters were kept constant. A current density of 15 mA cm⁻² was used and all experiments were conducted at a room temperature of 25 °C. The initial pH of the solution varied between 6.7 and 7.1. The anion concentration was not measured but is expected to be similar for experiments performed with the same concentration. Furthermore, no additional electrolyte was added. The duration of each experiment was 2 minutes, with samples taken every 15 seconds with a syringe.

**Figure 1.** Experimental set-up

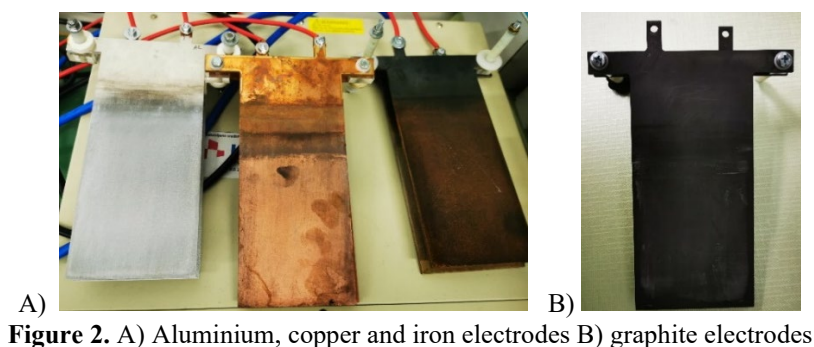


Figure 2. A) Aluminium, copper and iron electrodes B) graphite electrodes

A set of experiments was performed under the same conditions with perforated Al, Cu and Fe electrodes of the same dimensions as those originally used. The perforated electrodes are shown in Figure 3. The efficiencies of these experiments were compared with those obtained with non-perforated electrodes.



Figure 3. Perforated electrodes

4 Results and discussion

4.1 The results of statistical analysis

The three-level categorical design resulted in suitable factorial models. Using ANOVA, the quantitative significance of the models and each factor was calculated. A quantitative measure of model significance is the p-value or level of significance. The statistical significance level, P, indicates how likely it is that the difference between the experimentally tested value of removal efficiency and the value predicted by the model is due to chance. The P-value describes the probability that an error occurred in the prediction of the result (predicted removal efficiency). If the P-value is below the permitted level of significance, which is usually 5% ($P < 0.05$), the probability of an error in the prediction is less than 5%, i.e., there is a statistically significant difference. The models obtained for the removal nickel, cadmium, chromium, and cobalt indicated that the only significant factor is the electrode material with p-values ranging from 0.0001 to 0.0234. For manganese, both electrode material and initial concentration had a similar effect, with p-values of 0.0049 and 0.0045, respectively. All models showed reasonable agreement between the predicted and adjusted R^2 with a difference of less than 0.2. Table 2 shows the results of the experiments compared to the predicted values. Graphs of the predicted versus the actual values and normal plots of the residuals confirm the good fit of the model, as seen in Figure 4 in the case of manganese removal. Figure 5 shows the best solution for process optimization, i.e., the combination of factors that leads to maximum efficiency.

Table 2. The comparison between predicted (*) and observed efficiencies.

Material	C	Cd	Cd*	Co	Co*	Cr	Cr*	Ni	Ni*	Mn	Mn*
Fe/Fe	0.1	89.63	60.31	64.71	43.61	99.60	99.01	57.93	40.67	41.02	33.93
Fe/Fe	1	21.60	41.81	13.00	25.66	98.90	98.95	16.10	24.36	21.34	24.34
Fe/Fe	10	13.09	22.20	7.76	16.20	97.80	98.34	8.95	17.95	6.88	10.96

Al/Al	0.1	78.52	80.82	68.00	66.84	100.00	99.47	53.20	52.43	35.80	35.77
Al/Al	1	76.27	62.32	54.00	48.89	99.77	99.41	36.89	36.13	29.49	26.19
Al/Al	10	31.06	42.71	33.17	39.43	97.90	98.79	28.18	29.71	9.48	12.81
Cu/Cu	0.1	90.12	97.22	67.48	72.03	100.00	99.98	68.57	70.23	55.07	54.17
Cu/Cu	1	85.12	78.71	57.96	54.08	99.90	99.92	58.19	53.92	42.31	44.58
Cu/Cu	10	59.79	59.10	45.28	44.62	99.30	99.31	44.89	47.51	32.56	31.20
C/C	0.1	4.90	24.82	2.68	20.38	55.37	56.51	2.38	18.76	16.67	24.68
C/C	1	6.16	6.31	6.10	2.43	56.16	56.45	5.67	2.45	17.05	15.09
C/C	10	6.77	-13.30	7.01	-7.03	57.28	55.84	9.19	-3.96	7.76	1.71

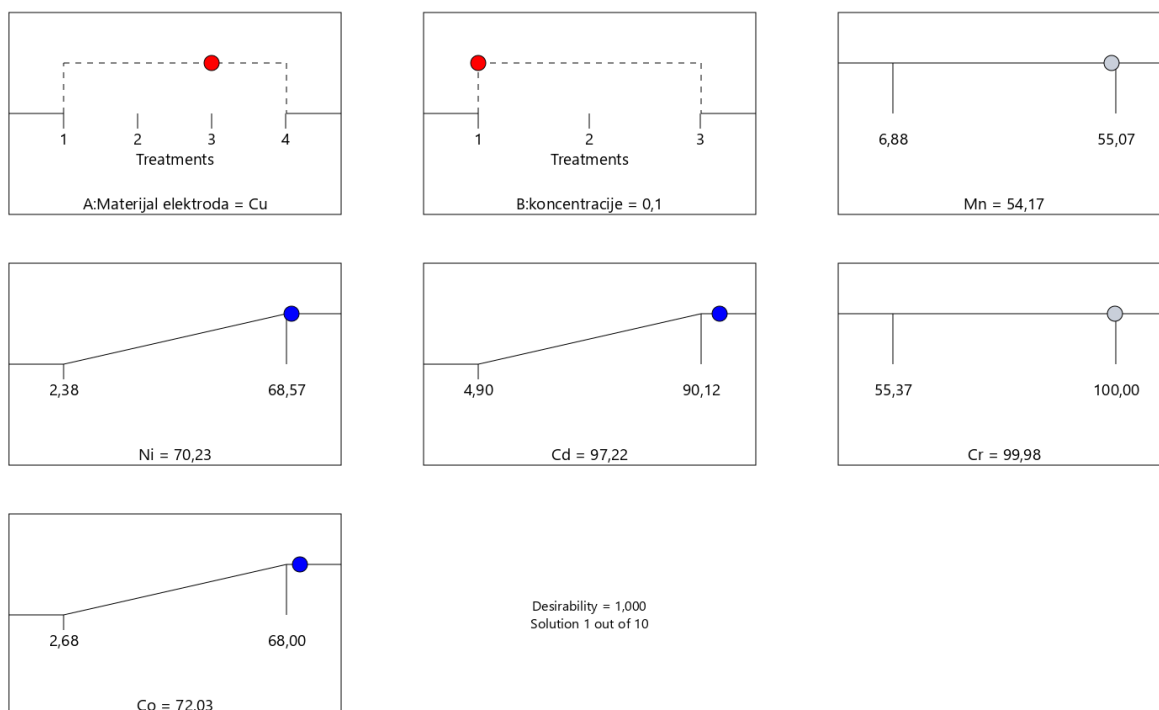


Figure 5. Ramp graphs presenting the results of process optimization

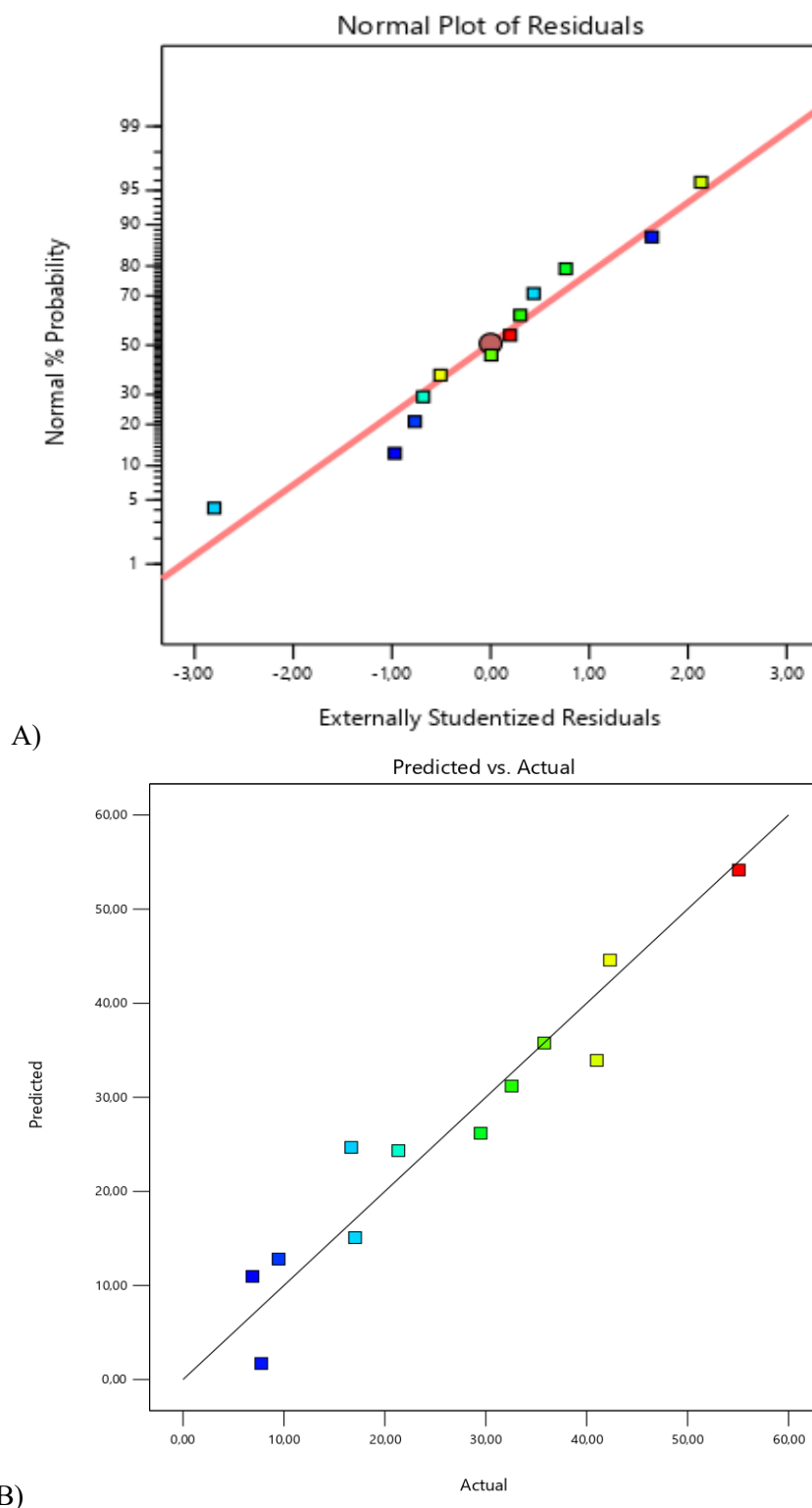


Figure 6. Normal plot of residuals (A) and predicted vs. actual graph (B) for manganese model

4.2 The effect of electrode material

The electrode material determines which electrochemical reactions occur in the EC system and the material selection depends on the pollutants to be removed and the chemical properties of the electrolyte [21]. In this study, three metallic electrode materials were tested: aluminium, iron and copper. These

elements differ in many chemical and physical properties such as ion size, ion charge, oxidation potential, and polarity of the ion-OH bond [22]. The size and the structure of the resulting metal hydroxide compound also differ, resulting in different adsorption capabilities of each hydroxide [23]. Graphite electrodes have also been tested. Although they are insoluble anodes, these electrodes show high oxygen evolution over-potential [22].

As expected, and confirmed by the model, the electrode material is the most influential factor in an EC process. All metal electrodes were found to be efficient, while graphite electrodes proved to be unsuitable for heavy metal removal and partially removing only chromium ions. It is also very important to note, that the composition of the electrodes and possible impurities can greatly affect the process. For example, manganese is a common impurity in metal electrodes and can be leached out of from the electrodes during the EC process resulting in diminished overall efficiency. In general, attention should be paid to the concentration of the electrode material in the outlet stream. For example, although copper electrodes resulted in the highest efficiency, due to the electrode potential of copper, a significant amount of copper was present in the solution after the EC treatment. Figure 7 shows how the concentration of the different electrode materials changed through the processes under the same experimental conditions.

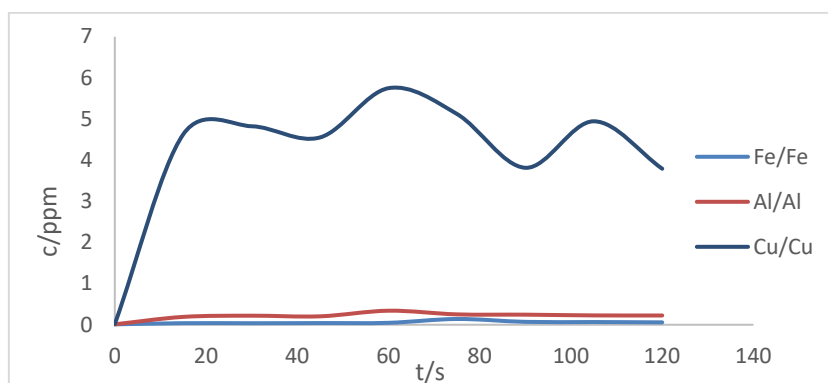


Figure 7. The change in concentration of Fe, Al and Cu during the process with corresponding electrodes

To determine the effect of electrode geometry on the process, the experiment that yielded the highest efficiencies was repeated with perforated electrodes. Perforations reduce the active surface area of the electrode and, consequently, result in lower current density. However, perforations allow better mixing of the solution and could also reduce the effect of electrode passivation [24]. In this case, there was little difference between the overall efficiencies of perforated and non-perforated electrodes. In the experiment with Cu electrodes at 1 ppm, perforated electrodes resulted in 92.6%, 64.3%, 99.7%, 68%, and 51.9% removal of Cd, Co, Cr, Ni, and Mn, respectively. Figure 8 compares these two processes.

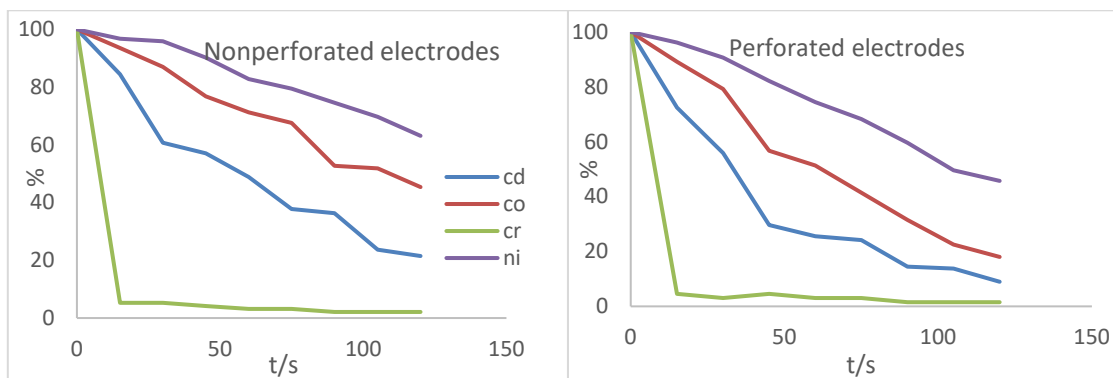


Figure 8. The comparison of the removal rate for the process with perforated and nonperforated electrodes.

4.3 The effect of initial concentration of contaminants

There are several studies indicating that the removal efficiency of heavy metals from water by the EC process is higher at lower initial concentrations of the contaminating metals [25], [26],[27]. The results of our study are consistent with these findings. When the initial concentration was increased, the removal efficiency for nickel, cadmium, manganese, and cobalt ions decreased. This could be explained by Faraday's law, according to which a constant amount of metal dissolves from the anode and passes into solution for the same current density and electrolysis time [25]. This means that regardless of the initial concentration of heavy metals, the same amount of metal hydroxides was formed in the solution, and in the case of higher concentrations, that amount was not sufficient to absorb all heavy metal ions. In addition, lower efficiencies at high initial concentrations of the metals may be associated with saturation of the electrode due to the formation of an oxide layer on the electrode surface. The only exception was chromium. In this case, the initial concentration had very little effect on the removal rate, and in almost all cases, with the exception of the graphite electrodes, an efficiency greater than 99% was achieved. This can be attributed to the fact that chromium is removed quickly and almost all of it is removed in the first 30 seconds. The effects on chromium removal for graphite electrodes are shown in Fig 9.

In general, for higher concentrations, a longer time is required for removal, and the electrocoagulation process is more effective at the beginning when the concentration is higher than at the end when the concentration is low [28]. For example, in the study by Dermentzis et al. [28], 99% of Ni was removed from 250 mL of a 100 ppm solution in 10 min using aluminium electrodes and a current density of 30 mA cm⁻². In our study, using a much larger volume of solution (3 L) and a lower current density (15 mA cm⁻²), the Ni concentration decreased by 28% from the initial concentration of 10 ppm in only 2 minutes.

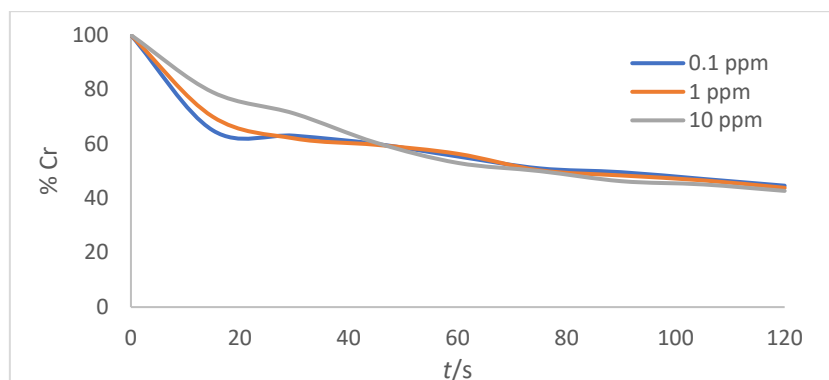


Figure 9. Removal of chrome using graphite electrodes

4.4 The kinetics of heavy metals removal

A kinetic study was carried out to describe the removal process of Ni, Co, Mn and Cd under optimal operating conditions that resulted in the highest efficiency (Cu, 0.1 ppm). There are several kinetic models used to describe the kinetics of the EC process for the removal of heavy metals. These models include first order, second order, pseudo-first order, pseudo-second order, and Elovich models[22]. For an EC batch process, the mass conservation of heavy metal ions follows the Eq (6), where the r_D is the rate of metal ion removal, C is the concentration of the metal ion, and t is the time (min).

$$-\frac{dC}{dt} = -r_D \quad (6)$$

For the zeroth order, the removal rate is defined in the Eq (7), where k is the zeroth rate constant.

$$r_D = -k \quad (7)$$

For the first-order kinetics the model is described by Eq (8), where k_1 is the first order rate constant. The integrated equation for the initial conditions of $C(0)=C_0$, at $t=0$ is given in Eq (10).

$$r_D = -k_1 C \quad (9)$$

$$C(t) = C_0 e^{-k_1 t} \quad (10)$$

The second order model is described by the Eq(11), where k_2 is the second order rate constant. Upon integration with the same initial conditions as above, the time dependant concentration is obtained in Eq (12).

$$r_D = -k_1 C^2 \tag{11}$$

$$\frac{1}{c(t)} = \frac{1}{c_0} + k_2 t \tag{12}$$

To determine the appropriate reaction order, the concentrations of Ni, Co, Mn and Cd were plotted against time and modeled as zero, first and second order reaction kinetics. The most appropriate model was selected based on their R² values. The closer the R² value is to 1, the better the model fit. The kinetic parameters of zero-, first- and second-order models together with the R² values are given in Table 3. Although it can not be used to describe our process, zero order kinetics was tested because of studies such as one by Garcia-Carrillo et al. [29] in which it is used to describe silver and gold removal by EC. A graphical comparison of the kinetic models is shown in Figures 10-12. As it can be seen, cobalt, nickel, cadmium and manganese removal follows the first-order kinetic. None of the models can appropriately describe chromium removal. These results vary from studies in which heavy metal removal by EC process is described by either second-order or pseudo-first order kinetics [23],[30]. However, these differences can be due to electrode type and the current density that was used.

Table 3. Predicted rate constants and corresponding R² values for kinetic models

Metal	k_0 (ppm min ⁻¹)	R ²	k_1 (min ⁻¹)	R ²	k_2 (ppm ⁻¹ min ⁻¹)	R ²
Cd	0.0013	0.849	0.0189	0.973	0.3818	0.901
Co	0.0007	0.851	0.0084	0.982	0.1124	0.885
Cr	0.0006	0.330	0.0271	0.592	7.0779	0.851
Ni	0.0008	0.924	0.0086	0.972	0.1022	0.932
Mn	0.0006	0.937	0.0060	0.972	0.0615	0.889

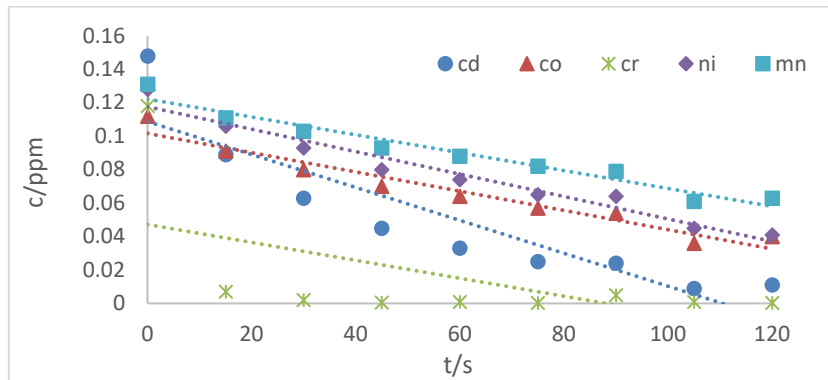


Figure 10. Zero-order kinetics model

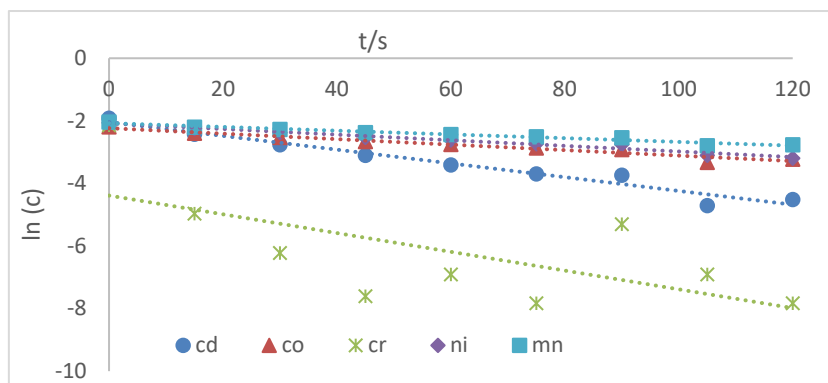


Figure 11. First-order kinetics

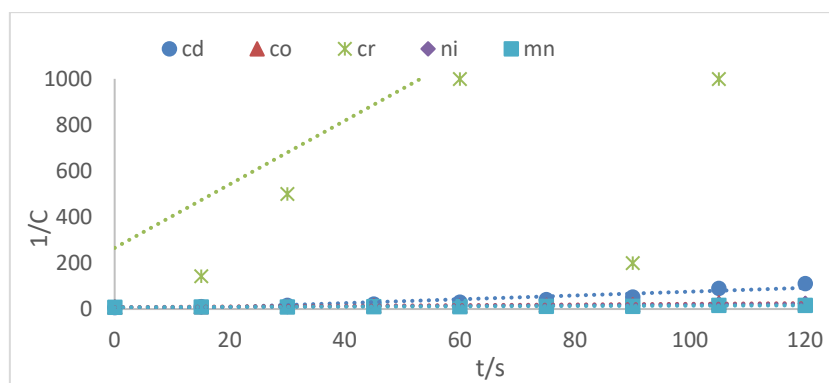


Figure 12. Second order kinetics

5 Conclusion

The most influential factor in the EC process for heavy metal removal is the material from which the electrode is made. The efficiency of the EC process increases as the initial concentration of the metal decreases. To achieve the same removal efficiency for higher initial concentrations of pollutants, a longer treatment time is required. Perforated and non-perforated electrodes showed similar behavior and overall efficiencies. In most cases, the kinetics of heavy metal removal followed first-order kinetics. While the EC process is effective in reducing heavy metals in water, further research and optimization of the process is needed, especially for highly contaminated waters.

Acknowledgements

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