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Cadmium removal from dilute aqueous solutions under galvanostatic mode in a flow-through cell

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ABSTRACT

Cadmium electrodeposition at reticulated vitreous carbon cathode from dilute aqueous solutions in a flow-through cell under galvanostatic mode is accomplished. The influence of different parameters such as background electrolyte, initial cadmium concentration, initial pH of the catholyte, and applied current density is studied. Cadmium concentration is detected by differential pulse stripping voltammetry technique using calibration curves built for each background electrolyte tested. The concentration decay is predicted based on a batch recycle reactor operating under mass transport control. The prediction of the concentration decay of cadmium ions is in good agreement with the experimental data. The current efficiency at pH 6 is influenced by the anion present in the working solution and decreases in the sequence $Cl^- > SO_4^{2-} > NO_3^-$. Also, the lowest current efficiency is observed in nitrate media at pH 6, sulfate electrolyte at pH 2, and when 3.2×10^{-4} mol L^{-1} cadmium concentration is used. Cadmium removal efficiency increases with the increase in applied current density, with the subsequent increase in the specific energy consumption. The efficiency of cadmium removal is within 81–99%, which is also influenced by the initial pH, initial cadmium concentration, and the applied current density. The current efficiency is within 12–34% with specific energy consumption between 3.9 and 11.1 kW h kg^{-1} depending on the operational parameters.

Keywords: Cadmium removal; Electrodeposition; Galvanostatic mode; Metal removal

1. Introduction

Polluted wastewater with heavy metals is the main anthropogenic source of water pollution in developing countries [1,2]. Metal contamination in the aquatic environment has attracted global attention owing to its environmental toxicity, abundance, and persistence [1,3]. Cadmium is toxic to essentially all forms of life, affecting target organs such as kidney, liver, bone, and the respiratory, cardiovascular, nervous, and

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reproductive systems [4]. Several processes have been proposed to remove heavy metals from wastewater like adsorption [5], precipitation [6], coagulation and flotation [7], electrochemical removal [8–14], among others. The electrochemical processes can provide valuable contributions to the protection of the environment through implementation of effluent treatment and production-integrated processes for the minimization of waste and toxic compounds [15]. Furthermore, the electrochemical processes have an important role to play as part of an integrated approach to the avoidance of pollution, monitoring of pollution and process efficiency, cleaner processing, and modern techniques for electrical energy storage and conversion [16]. The use of the electrochemical approach to recover/remove metal ions from wastewater can be considered as a relatively simple and clean process. Cathodic removal of metals shows several advantages in terms of costs, safety, and versatility since the metal can be recovered in its pure metallic form, or as a concentrated solution that can be recycled or allow the extraction of the pure metal in an electrowinning process [17]. Thus, the metal can be sold in the market. The electrochemical removal of heavy metal ions from wastewater is an attractive alternative process to other processes like adsorption, precipitation, and extraction since such processes do not provide the ion removed in its metallic form; also, special disposal might be necessary (such as adsorption), or contamination to the environment can be produced by organic solvents to extract the heavy metals from the wastewater, that might incur in costs and the need of special disposal of the concentrated heavy metal solutions.

Several studies dealing with cathodic cadmium removal have been published in the last decade. For instance, the selective recovery of Cd from simulated spent nickel–cadmium battery solutions was achieved with high selectivity and high current efficiency in sulfate and chloride acidic media, but nitrate acidic media was inadequate [18]. The electrochemical removal of cadmium from dilute solutions was accomplished in a rectangular spouted bed reactor by Baghban and collaborators. These authors reported that low initial concentration of cadmium, except for 270 mg L^{-1} Cd^{2+}, initial pH of the catholyte, and electrolyte concentration did not affect the overall electrochemical process [13]. Furthermore, Dutra et al. reported that cadmium removal at reticulated vitreous carbon (RVC), from acidic solutions, by electrolysis in a flow-by cell, was only feasible in deaerated electrolyte to avoid the secondary reaction of dissolved oxygen reduction [12]. Tramontina et al. [19] carried out the cathodic removal of 5 and 10 ppm of cadmium ions on RVC electrodes (30, 60, and 100 ppi) in a stirred conventional three-electrode cell under potentiostatic control. They reported that the current efficiency dropped by increasing RVC porosity in the potential range studied (−0.9 to −1.1 V vs. SCE), which was attributed to an increase in the charge consumed due to H_2 evolution and O_2 reduction reactions occurring simultaneously with cadmium reduction. Reade et al. [20] studied the removal of cadmium and cupric ions from sulfate electrolyte at pH 2 using a RVC (10 ppi) rotating cylinder electrode (with the cathode rotated at 1,500 rpm). These authors reported that the initial cadmium concentration of 56 ppm decreased to <1 ppm at current efficiencies between ~75% and below 20% (when the cadmium ion concentration had fallen to 3 ppm).

This work is aimed at studding the cadmium electrodeposition at RVC (60 ppi) cathode from dilute aqueous solutions in a flow-through cell under galvanostatic mode. Factors affecting the electrochemical removal of cadmium such as type of background electrolyte (sulfate, nitrate, and chloride ions), initial cadmium concentration, initial pH of the catholyte, and applied current density were studied. A facile procedure for cadmium analysis was implemented using a three-electrode cell configuration in an acetate electrolyte containing the background electrolyte used during the cathodic cadmium removal. The divided parallel plate electrochemical reactor and the methodology developed in this study are useful for cathodic heavy metal removal from real effluents coming from mines and electronic wastes.

2. Theory

The reduction of cadmium ions on the cathode surface from aqueous solution is a two-electron transfer reaction (1) that is affected by hydrogen evolution and oxygen reduction as secondary reactions (2) and (3) [11]:

$$Cd^{2+} + 2e^{-} \rightarrow Cd \tag{1}$$

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

$$O_2 + 4H^{+} + 2e^{-} \rightarrow 2H_2O \quad (pH < 7) \tag{3}$$

Cadmium reduction under mass transport control can be expressed by Eq. (4) for a batch recycle system [10]:

$$\frac{C}{C_o} = \exp\left(-\frac{k_m A V_e t}{V_R}\right) \tag{4}$$

where C is the initial cadmium concentration (mol L^{-1}); C_o is the final cadmium concentration (mol L^{-1}); k_m is the mass transport coefficient (m s^{-1}); A is the cathode area (m^{-2}); V_e is the electrode volume (m^3); t is the time (s); and V_R is the volume of the reactor (m^3).

The overall current efficiency ($\emptyset$) [21] is given by Eq. (5):

$$\emptyset = \frac{\text{Charge used in reducing the cadmium}}{\text{Total charge}} \tag{5}$$

The electrical charge, q, is defined as:

$$q = nmF = \int_{t=0}^{t} i\,dt \tag{6}$$

where n is the number of electrons, m is the number of moles of cadmium (mol), F is the Faraday constant (A s mol L^{-1}), i (A) is the current, and t (s) is the time. Dividing the electrical charge (Eq. (6)) by the volume of the reactor, V (L), one obtains:

$$M = \frac{1}{nFV} q \tag{7}$$

where M is the molar concentration of cadmium (mol L^{-1}), and q (As) can be calculated by integrating the area under the curve from plotting t vs. i. The experimental slope, obtained from plotting q vs. M, is used to calculate the overall current efficiency, as shown by Eq. (8), where the theoretical slope is given by $1/nFV$.

$$\emptyset(\%) = \frac{\text{Experimental slope}}{\text{Theoretical slope}}(100) \tag{8}$$

The specific energy consumption $\left(E_s\left(\text{kW h kg}^{-1}\right)\right)$ can be calculated with Eq. (9) [22], where E_{Cell} is the cell potential and MW is the molar mass (kg mol^{-1}).

$$E_s = \frac{nFE_{\text{Cell}}}{(\emptyset)\text{MW}} \tag{9}$$

3. Experimental

3.1. Chemicals

Sodium sulfate, sodium chloride, sodium nitrate, cadmium sulfate, sulfuric acid, sodium hydroxide, sodium acetate, and acetic acid were used as received without further purification (Sigma-Aldrich). All solutions were prepared with Millipore purified water (Milli-Q/RIO) except those for the simulated electrolytes (catholyte) for cadmium electrodeposition which were prepared with tap water and saturated with nitrogen (PRAXAIR, industrial grade). The anolyte was also prepared with tap water. The tap water characterization is shown in Table 1. Sodium, potassium, calcium, magnesium, total iron, and copper ions were analyzed by atomic absorption spectroscopy (NMX 2001, ASTM 2002); chlorides and alkalinity by titration (ASTM 1999). The contents of sulfates and phosphates were known by spectrophotometry (DR/4000 U) where the samples were subjected to acid digestion previous to be analyzed. The pH was measured by a calibrated pH-meter (Orion).

3.2. Cadmium analysis

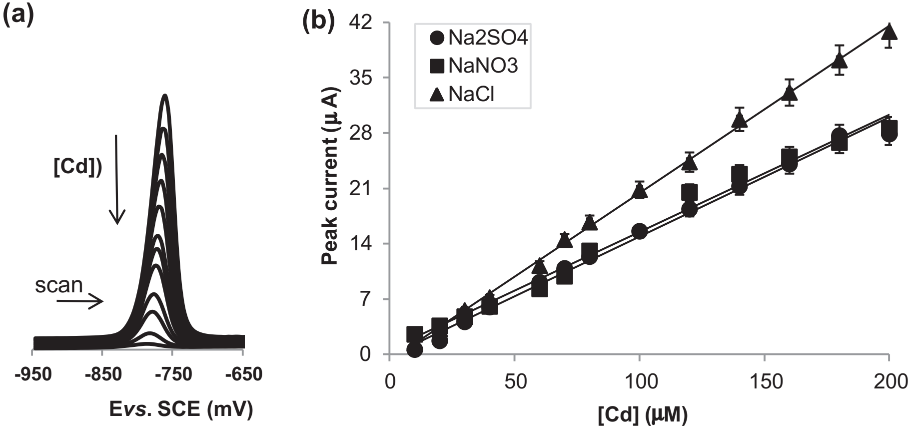
The differential pulse stripping voltammetry (DPSV) technique was used to detect Cd(II) ions at graphite electrode (3.85×10^{-3} cm^2) using a graphite auxiliary (0.28 cm^2) and SCE reference electrode connected to a potentiostat (Epsilon Model E2-BASI). A calibration curve of cadmium(II) ion concentration vs. the peak current height was built from differential pulse stripping voltammograms (figure (a) inserted in Table 2) of various concentrations of Cd(II) ions measured with graphite working electrode as shown in figure (b) inserted in Table 2. The working volume of the background electrolyte was 5 mL comprised by 0.2 M sodium acetate/acid acetic and 0.5 M of Na_2SO_4, NaCl, or $NaNO_3$ (depending on the salt used in the catholyte for cadmium removal). Thus, three calibration curves were built depending on the background electrolyte present in the catholyte reservoir (figure (b) inserted). The working parameters were as follows: accumulation time 60 s at −0.95 V, initial potential −0.95 V, end potential 0 V, pulse width 0.05 s, pulse amplitude 0.025 V, step potential 0.005 V, and pulse

Table 1
Chemical composition of tap water

Parameter	mg L^{-1}
Sodium	130 ± 3
Potassium	2.6 ± 0.1
Total iron	<0.01
Calcium	3.2 ± 0.1
Magnesium	5.5 ± 0.1
Copper	<0.02
pH (at 25˚C)	7.5 ± 0.1
Chlorides	188 ± 3
Sulfates	2.2 ± 0.05
Phosphates (as total phosphorous)	<0.001
Total alkalinity (as $CaCO_3$)	65 ± 2

Table 2
Analytical parameters for detection of cadmium

Electrolyte[a]	$-E_p$ vs. SCE (V)	Calibration curve equation	R^2
NaCl	0.800	$i_p = 0.2117[\mathrm{Cd}] - 0.7649$	0.999
Na_2SO_4	0.790	$i_p = 0.1508[\mathrm{Cd}] - 0.1922$	0.993
$NaNO_3$	0.800	$i_p = 0.1484[\mathrm{Cd}] + 0.6236$	0.987



(a) Differential pulse stripping voltammetry (DPSV)

(b) Calibration curve in different electrolytes

[a] 0.5 mol L^{-1} electrolyte + buffer (0.2 mol L^{-1} acid acetic/sodium acetate).

Notes: DPSV (graphite working electrode (3.85×10^{-3} cm^2), graphite auxiliary (0.28 cm^2), and SCE reference electrode. Deposition time: 60 s at −0.95 V vs. SCE; initial potential: −0.95 V, end potential: 0 V; pulse width: 0.05 s; pulse amplitude: 0.025 V; step potential: 0.005 V; pulse period: 0.2 s).

period 0.2 s [23,24]. Table 2 shows the analytical parameters and the peak potential (E_p) that corresponds to the peak current height at each Cd(II) concentration in the background electrolyte used (figure (a) inserted). Peak potential gradually shifts to more positive potentials with increasing Cd(II) ion concentration. The calibration curves were linear within the concentration interval from 10 to 200 μM Cd(II) ions with high coefficients of linear correlation (R^2), as shown in the table.

3.3. Electrochemical reactor and instrumentation

Fig. 1 presents a scheme of the experimental setup of a divided parallel plate reactor in the hydraulic circuit used for cadmium removal. It was constructed from acrylic blocks (26 cm × 12 cm × 1 cm) with two electrolyte compartments. The cathode and anode were recessed into each block forming a flow channel (50 cm^2) on each one. The electrolyses were carried out at a three-dimensional RVC cathode (0.05 m × 0.05 m × 0.01 m, 60 ppi, Electrolytic Inc., NY) separated from a platinized Ti-mesh anode by a cation permeable membrane (Nafion 117). The surface area per unit volume of the RVC 60 ppi is 4,000 m^{-1} [25]; thus the area of the cathode is 0.1 m^2 (obtained by multiplying 4,000 m^{-1} times 2.5×10^{-5} m^3). The catholyte was pumped (IWAKI Co., Ltd MD-15R-115NL01) through the channel at a constant flow velocity (4.6 LPM) measuring the catholyte flow with a flow rate meter (Blue-White Industries F-1000-RB). All experiments were carried out under galvanostatic mode using a power supply (Matrix-model MPS-6005L-1). Charges were calculated by integrating the area under

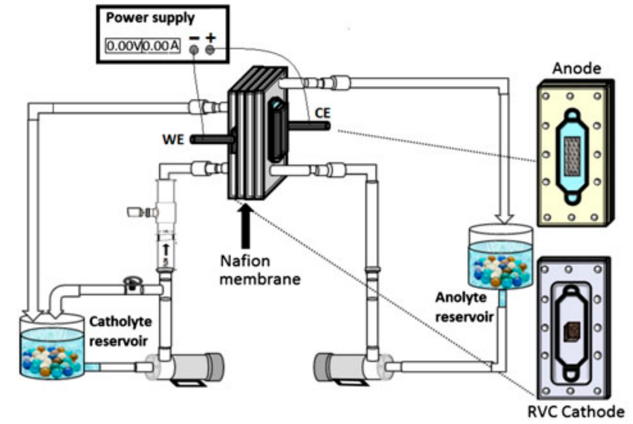


Fig. 1. Experimental setup of the electrochemical batch recycle reactor with a Ti-mesh anode and a RVC cathode.

the curve from plotting the time vs. current. The pH of the solution was determined with a portable pH-meter (Orion). Samples were withdrawn from the electrochemical reactor at timed intervals to analyze cadmium during the electrodeposition. Appropriate aliquots were added to the buffer acetate/acid acetic electrolyte (described in Section 3.2), and differential pulse stripping voltammograms were recorded for each sample under the same working conditions used to build the calibration curve. The height of the peak current were related to the concentration of Cd(II) using the respective equation of the straight line obtained from the calibration curves (reported in Table 2).

4. Results and discussion

4.1. *Influence of background electrolyte on cadmium electrodeposition*

Fig. 2 shows the polarization data, recorded manually, for the reduction of 1.78×10^{-3} mol L^{-1} cadmium ion in three catholytes such as sulfate, chloride, and nitrate (0.5 mol L^{-1}, pH 6) at 276 L h^{-1}. Limiting current plateaus are observed for cadmium reduction in the sulfate background electrolyte. The limiting current plateau extends into the potential range of −1.2 to −1.4 V. Beyond this potential range, the hydrogen evolution interferes negatively on cadmium deposition. The polarization data for the blank (● symbols) and Na_2SO_4 (○ symbols) are for the absence and presence of cadmium in the sulfate electrolyte using a RVC cathode without previous use for cadmium electrodeposition; the rest of the polarization data corresponds to an RVC used after several electrolyses for cadmium removal. A well-defined plateau is observed when cadmium electrodeposition happens on cadmium, in the sulfate catholyte, with higher limiting current than that at RVC. Thus, the applied current density used for cadmium electrodeposition in the sulfate catholyte was within 3.5–11.3 Am^{-2}.

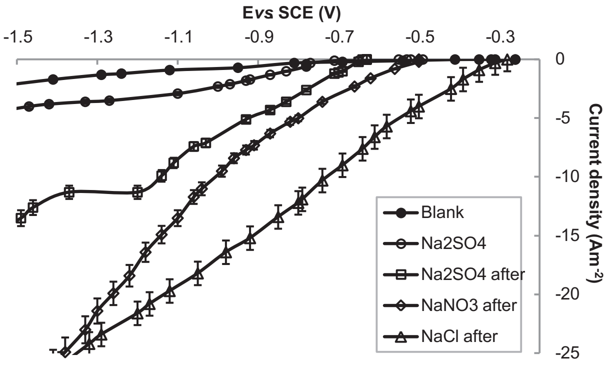


Fig. 2. Polarization curves for the reduction of 1.78×10^{-3} mol L^{-1} Cd(II) in three background electrolytes (0.5 mol L^{-1}): (a) Na_2SO_4, (b) NaCl, and (c) $NaNO_3$ at 280 L h^{-1}. The symbols joined by the solid lines are experimental data taken manually.

The electrochemical removal of cadmium in the flow-through cell was accomplished under galvanostatic mode. The decrease in cadmium concentration at the RVC cathode in three background electrolytes (Na_2SO_4, NaCl, and $NaNO_3$) was evaluated as a function of electrolysis time. Cadmium concentration was detected by the differential pulse stripping voltammograms (technique implemented in this study, as reported in Section 3.2) using the calibration curves built for each background electrolyte (see Table 2). A constant current density of 3.5 Am^{-2} was applied to the electrochemical cell with a power supply to electrodeposit cadmium ions from an initial concentration of 2×10^{-3} mol L^{-1} Cd(II) in solution at pH 6, as shown in Fig. 3. Fig. 3(a) shows the normalized cadmium concentration decrease as a function of three background electrolytes during the electrolysis time. The three concentration profiles follow an exponential decay as expressed by Eq. (4) for a batch recycle system [10]; the inset of Fig. 3(a) shows that $\ln(C_t/C_o)$ vs. t is linear.

The electrodeposition rate of cadmium is slightly lower in the sulfate catholyte than that obtained in chloride and nitrate media at the same initial pH. Cadmium removal was accomplished with current efficiencies ($\emptyset$) that vary between 17 and 34% depending on the background electrolyte, as depicted in Table 3. The low current efficiency may be attributed to the co-reduction of dissolved oxygen (not removed completely) and hydrogen ions that have unfavorable influence at low metal concentration removal [11,12]. The cadmium removal in chloride media was 99% ($\emptyset = 34\%$), 95% in sulfate media ($\emptyset = 29\%$), and 95% in nitrate media ($\emptyset = 17\%$), with a specific energy consumption between 3.9 and 7.8 kW h kg^{-1} at 2 h of electrolysis. Cadmium electrodeposition in nitrate media was with the lowest current efficiency.

Fig. 3(b) illustrates the pH variation during cadmium electrolysis. The pH in the sulfate catholyte tends to decrease to pH 5 and to pH 4 after 10 and 90 min of electrolysis, respectively. This may be attributed to the acid/base equilibrium of the HSO_4^-/SO_4^{2-} ($pK_a = 1.92$ [26]) that tends to decrease the pH of the solution. Regarding chloride and nitrate media, the pH starts to increase after 20 min for nitrate and after 40 min for chloride background electrolytes. The final pH of the catholyte is basic (pH 12 for chloride and 10 for nitrate electrolytes, respectively). This may be

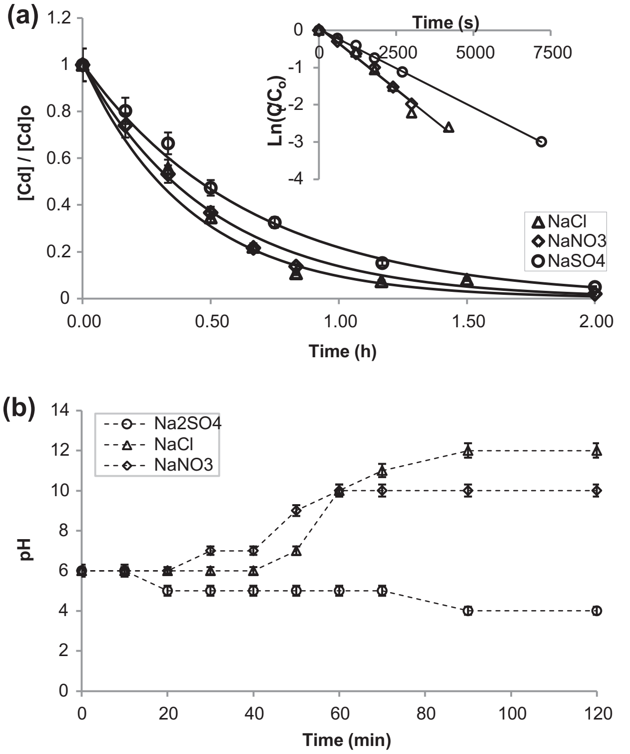


Fig. 3. (a) Influence of electrolyte on cadmium removal at an initial pH 6 and (b) variation of the solution pH during the electrolysis.

attributed to hydrogen ions consumption because of hydrogen evolution which leads to an increase in the pH near the electrode surface; consequently, the pH of the solution increases resulting eventually in cadmium hydroxide precipitation [27]. This phenomenon may explain the faster cadmium removal observed in chloride and nitrate solutions, starting at ~40 min (Fig. 3(a)) than that in the sulfate catholyte; also, the normalized concentration data slightly deviate from the prediction (solid lines) for an exponential decay due to the possible formation of cadmium hydroxide.

4.2. Influence of initial cadmium concentration

The influence of initial cadmium concentration on the electrochemical deposition of cadmium in sulfate background electrolyte at initial pH 6 is illustrated in Fig. 4. Constant current densities of 3.5, 1.75, and 0.875 Am^{-2} were applied to the electrochemical reactor to remove initial concentrations of cadmium of 2.0×10^{-3}, 1.35×10^{-3}, and 3.2×10^{-4} mol L^{-1}, respectively. The concentration decay for an initial concentration of 1.35×10^{-3} mol L^{-1} was not exponential in the first 40 min of electrolysis. This may be associated with incomplete removal of oxygen from the solution. The co-reduction of dissolved oxygen and hydrogen ions has unfavorable influence at low metal concentration removal [11,12]. Table 3 shows that the current efficiency decreases as the initial cadmium concentration decreases, in agreement with Dutra et al. who studied cadmium removal in a flow-by cell [12] and also with Baghban et al. who studied the electrochemical removal of cadmium in a rectangular spouted bed reactor [13]. Cadmium removal efficiency slightly decreases with the decrease in the initial cadmium concentration. 81–97% was accomplished at 2 h of electrolysis, as shown in Table 3. The specific energy consumption also increases by decreasing the initial cadmium ions concentration.

Table 3
Cadmium electrodeposition under galvanostatic mode using different conditions

Electrolyte (0.5 mol L^{-1})	Cd^{2+} initial concentration (ppm)	Initial pH	Cell voltage (V)	Current density (A m^{-2})	Current efficiency (%)	Cadmium removal (%)	Specific energy consumption (kW h kg^{-1})
NaCl	225	6	2.78	3.5	34	99	3.9
Na_2SO_4	225	6	2.78	3.5	29	95	4.5
NaNO_3	225	6	2.78	3.5	17	95	7.8
Na_2SO_4	225	2	2.78	3.5	17	86	7.8
Na_2SO_4	225	4	2.78	3.5	22	95	6.0
Na_2SO_4	225	6	2.78	3.5	30	99	4.4
Na_2SO_4	220	6	2.78	3.5	30	97	4.4
Na_2SO_4	152	6	2.78	3.5	32	95	4.1
Na_2SO_4	35	6	2.78	3.5	12	81	11.1
Na_2SO_4	225	6	2.78	3.5	29	85	4.5
Na_2SO_4	225	7	2.85	5.5	24	99	5.7
Na_2SO_4	225	7	3.70	11.3	22	99	8.0

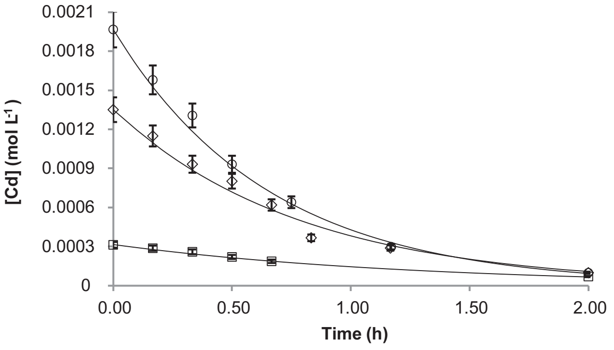


Fig. 4. Influence of initial cadmium removal in sodium sulfate electrolyte at an initial pH 6.

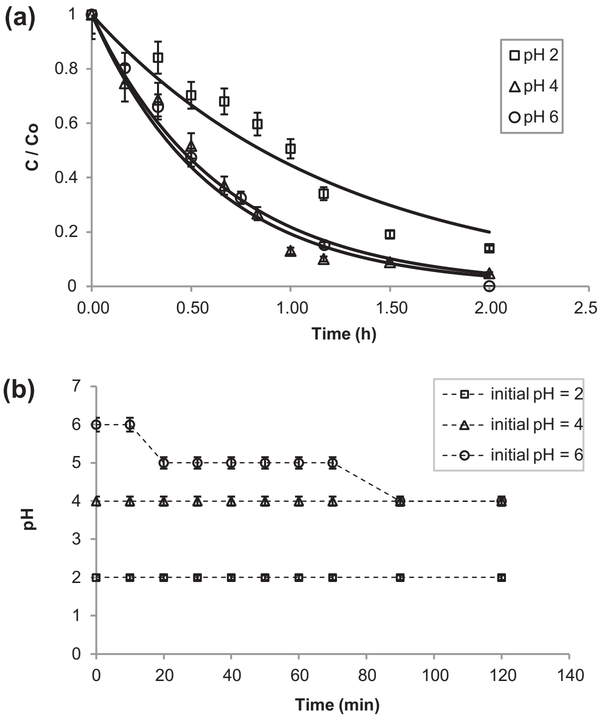


Fig. 5. (a) Influence of initial pH on cadmium removal in sodium sulfate electrolyte and (b) variation of the solution pH during the electrolysis.

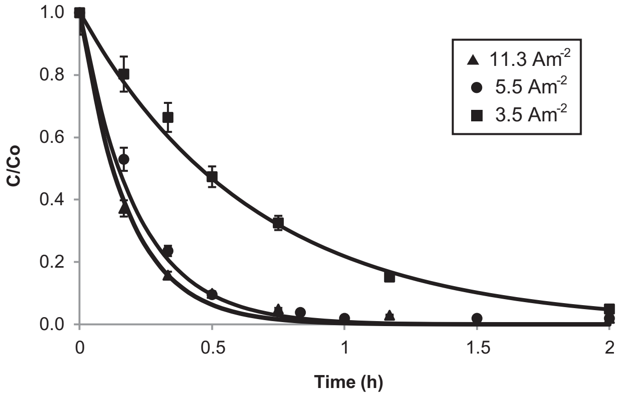


Fig. 6. Influence of applied current on 200 ppm cadmium removal in sodium sulfate electrolyte.

4.3. Influence of initial pH of the electrolyte

The influence of initial pH of sodium sulfate catholyte on cadmium electrodeposition is shown in Fig. 5. This figure also shows the pH variation during the electrolysis for cadmium removal. Constant initial pH of 2, 4, and 6 were tested for the electrochemical removal of cadmium at initial concentration of 2.0×10^{-3} mol L^{-1} with applied current density of 3.5 Am^{-2} (Fig. 5(a)). The rate of the cadmium electrodeposition for initial pH 2 is slower than that at pH 4 and 6 which show similar rate for the removal of cadmium. Cadmium removal efficiency increases with increasing the initial pH of the catholyte. 86% at pH 2, 95% at pH 4, and 99% at pH 6 were acquired at 2 h of electrolysis, as shown in Table 3, with the subsequent decrease in the specific energy consumption. Segundo et al. [28] reported a removal efficiency of 93.91 and 81.24% for cadmium at pH 2.5 and 1.5, respectively, in NaCl electrolyte using Chemelec electrochemical reactor. Their results showed that cadmium removal efficiency decreases with the decrease in initial pH of the electrolyte.

The variation of the pH during the electrolyses, shown in Fig. 5(b), indicates that the initial pH 6 decreases to pH 4 with reaction time due to the HSO_4^-/SO_4^{2-} acid/base equilibrium. The current efficiency at pH 2 is lower (17%) than that at pH 4 (22%) and 6 (30%) which may be attributed to the higher hydrogen ion concentration at pH less than 4 that is electrochemically reduced to hydrogen gas. Cadmium deposition was similar at pH 4 and 6 (~98%) but higher than that at pH 2 (86%).

4.4. Influence of the applied current density

The influence of the applied current density on cadmium electrodeposition in sulfate media is illustrated in Fig. 6. The rate of the electrochemical removal of cadmium is increased by increasing the current density. However, the current efficiency slightly decreases with increasing the current density, whereas the specific energy consumption increases, as reported in Table 3. The current efficiency for cadmium electrodeposition was in agreement with previous reports [12,13].

5. Conclusions

This study shows that the electrodeposition of cadmium at RVC cathode, from dilute aqueous solutions, can be accomplished in a flow-through cell under galvanostatic mode. Factors such as initial pH of the solution, background electrolyte, initial cadmium concentration, and applied current density influence the electrochemical removal of cadmium. The current efficiency is influenced by the pH, background electrolyte, and applied current density. The current efficiency at pH 6 is influenced by the anion present in the working solution and decreases in the sequence $Cl^- > SO_4^{2-} > NO_3^-$. Also, the lowest current efficiency is observed in nitrate media, sulfate electrolyte at pH 2, and when 3.2×10^{-4} mol L^{-1} cadmium concentration is used. Cadmium removal efficiency increases with the increase in the applied current density and with the subsequent increase in the specific energy consumption. The efficiency of cadmium removal is within 81–99%, which is also influenced by the initial pH, initial cadmium concentration, and the applied current density. The current efficiency is within 12–34% with specific energy consumption between 3.9 and 11.1 kW h kg^{-1} depending on the operational parameters.

The divided parallel plate electrochemical reactor and the methodology developed in this study will be used to carry out the cathodic cadmium removal (including heavy metal) from real effluents coming from mines and electronic wastes.

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