



# Cadmium Removal Using Bio-Electrochemical Reactor with Packed Bed Rotating Cylindrical Cathode: A Kinetics Study

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#### Abstract

The kinetics of removing cadmium from aqueous solutions was studied using a bio-electrochemical reactor with a packed bed rotating cylindrical cathode. The effect of applied voltage, initial concentration of cadmium, cathode rotation speed, and pH on the reaction rate constant (k) was studied. The results showed that the cathodic deposition occurred under the control of mass transfer for all applied voltage values used in this research. Accordingly, the relationship between logarithmic concentration gradient with time can be represented by a first-order kinetic rate equation. It was found that the rate constant (k) depends on the applied voltage, the initial cadmium concentration, the pH and the rotational speed of cathode. It was increased with increasing the applied voltage and its relationship with the applied voltage obeyed an exponential formula. The rate constant (k) was decreased with increasing the initial concentration of cadmium higher than 150ppm while at low concentrations it was increased. pH and rotational speed have different effects on the rate constant. Increasing the pH from 3 to 6 increases the rate constant; however, the rate constant became approximately constant buoyed 300 rpm.

Keywords: Cadmium; Packed bed rotating electrode; Microbial electrolysis cell; Kinetics; reaction rate constant.

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#### 1- Introduction

The contamination of the aquatic environment with heavy metals like copper, cobalt, lead, cadmium, and zinc is a critical issues that causing health risk because of the non-biodegradability and toxicity properties of these metals. They cause many diseases and disorders due to their affinity for bioaccumulation across the food chain even at very low concentrations [1,2]. Cadmium (Cd) is one of these heavy metals which has long biological halflife (more than 20 years) so they considered as highly toxic to humans. Adverse impacts of cadmium can be outlined as causing acute and chronic metabolic disturbances, such as emphysema, renal damage, testicular atrophy, and hypertension [2].

Numerous industrial operations, such as the non-ferrous metals purification and smelting, the manufacturing of battery, the electroplating industry, and the manufacture of inorganic pigments, lead to cadmium pollution in wastewater streams [3]. Hence, developing efficient approaches for cadmium removing from wastewater is a crucial duty in terms of protecting the environment and public health.

Conventional methods for removing cadmium can be physical, chemical, and biological methods [4-6]. Many of these methods are inefficient to remove Cd (if its concentration being lower than 100 mg/L) such as chemical precipitation and adsorption. Others are being expensive such as reverse osmosis, or need to large amount of chemicals such as the ion exchange method to regeneration the resin [7]. While methods like membrane processes need to high operating costs and sever from fouling [3]. Electrochemical methods such as electrodeposition and coagulation are more efficient than the traditional methods and can be efficiently remove cadmium with no consumption of organic carbon and no generation of sludge, but these methods still need to relatively high energy and high capital cost with relatively low efficiency at dilute concentration [8,9]. Hence, providing an ecologically friendly and cost-effective approach for removing cadmium while reducing sludge generation and lowering energy requirements remains a challenge.

Microbial fuel cells (MFCs) and microbial electrolysis cells (MECs) are talented methods to achieve sustainable wastewater treatment with simultaneous energy generation and value-adding products [10]. MFCs have been used to recover various metals including chromium [11], copper [12], iron [13], selenium [14], and vanadium [15] while MECs have the ability to recover heavy metals such as cobalt, lead, zinc, and cadmium [16] via applying an external voltage.

Few studies have been conducted on the removal of cadmium from wastewater using MECs [2, 10, 17-19]. However no previous work was conducted to remove cadmium using MEC with rotating cylinderical electrode.

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Removal of cadmium using rotating cylinderical electrode was successfully achieved using the traditional cathodic deposition but the energy consumption still relatively high [20].

For laboratory application, batch electrochemical reactors are comprehensively approved because they allow performance data as a function of reactant concentration or conversion to be obtained. Uniform current distribution over the electrode surfaces are assumed to be applied in the perfectly design of the electrochemical reactors. In industry, batch mode is often the most economical way to operate when production units are small [21]. Two distinct regimes could be identified in the cathodic deposition process in batch electrochemical reactors with rotating cylindrical cathode operated at galvanostatic mode that is preferred for industrial scale. The first regime is electron transport control while the second regime is mass-transport control. In the first case, decay of concentration is linearly with time while in the second case, exponential decay of the concentration with time is occurred combined with decreasing in current efficiency with time [22, 23].

Therefore, studying the kinetics of cathodic deposition is an essential process by which information about the process dynamics such as reaction rate, order of reaction, and mass transfer properties can be obtained [24].

In the case of using a batch electrochemical reactor with rotating cylindrical cathode operated under galvanostatic conditions (mass-transport control) for metal removal from waste solutions, the concentration profile of heavy metal with time can be represented by the following first order rate equation[21,24-27]:

$$C_{(t)} = C_o \exp[-kt] \tag{1}$$

Where: k refers to rate constant in min<sup>-1</sup>

To our knowledge, no study has been performed to investigate the kinetics of Cd removal using a bioelectrochemical reactor with a packed bed rotating cylindrical cathode, and no rate-steady correlation has been reported based on this design of the electrochemical system. In this study, the kinetics of the electrochemical removal of Cd from an aqueous solution was investigated using a bio-electrochemical reactor with a packed bed rotating cylindrical cathode based on the effect of four main variables: applied voltage, initial concentration of Cd(II), pH, and rotation speed of cathode.

#### 2- Materials and Methods

The electrochemical system adopted in the present work for cadmium removal consists of a rotating fixed bed bioelectrochemical reactor, a hot plate magnetic stirrer (IKA, Germany),an electric motor (type, Phoenix-USA), a power source (UNI-T model: UTP3315TF-L). ), and two Avometers (Type, Kwun Tong, Kowloon, HK) for current and voltage measurements. Fig. 1 shows a schematic diagram of the electrochemical system. The bioelectrochemical cell consisted of a cylindrical cell body (15 cm in diameter and 11 cm in length) terminated at the top with a flange of dimensions (15 cm in diameter and 0.5 cm in thickness), the cell and its flanges made of Perspex. Porous Graphite cylinder having dimensions (10 cm in length x 9.8 cm in diameter with thickness of 0.5 cm) was used as an anode. It has an apparent surface area (270.176 cm<sup>2</sup>) and purchased from (HP Graphite Handan Co., Ltd.).

The cathode was a rotating cylinder of spiral-wrapped woven wire mesh, consisting of continues stainless steel mesh layers wrapped around a central rod with dimensions (15 cm x 0.8 cm) to make packed bed with dimensions (2 cm outside diameter and 4.8 cm long) made of the same material and surrounded by two Teflon sleeves with an outer diameter of 2.6 cm and 2 cm long. The stainless steel woven screen used to create the cathode has a mesh of 30 wires per inch and has a specific surface area of 38.06 cm<sup>-1</sup> [28, 29].

The catholyte chamber is made of Perspex. It consists of two main parts: the first is a perforated supporting cylindrical vessel of dimensions (11 cm in length and 7 cm in diameter) perforated with holes 1 cm on its side surface. The distance between any two holes was (1.5 cm) and the total number of holes was (40).

This supporting container is ended from the top with a flange of dimensions (15 cm in diameter and 0.5 cm in thickness) containing four holes (0.4 cm in diameter) for fixing the cathodic chamber to the cell body by means of screws and nets. The second part is a cylindrical ion exchange membrane chamber. It was constructed of a cylindrical cation exchange membrane with dimensions (11 cm in length and 5.6 cm in diameter) fixed at the bottom with a cylindrical perspex disc having dimensions (5.5 cm in diameter and 0.5 cm in thickness). The cation exchange membrane was (IONIC-64LMR.)



Fig. 1. schematic diagram of the electrochemical system

The anodic electrode was inoculated with activated sludge as a source of bacteria taken from a local biological wastewater treatment unit in Karkh No. 2 / Baghdad Sewage Department / Iraq and fed with the nutrient medium as a carbon-containing source (per liter). ): CH<sub>3</sub>COONa 1 g, NaH<sub>2</sub>PO<sub>4</sub> • H<sub>2</sub>O 2.45 g, Na<sub>2</sub>HPO<sub>4</sub> 4.58 g, KCl 0.13 g, NHC<sub>4</sub>Cl 0.31 g, pH = 7 [30].

The cathode chamber was supplied with a cathodic solution composed of cadmium chloride (CdCl<sub>2</sub>) at different concentrations based on the conditions of the experiment. All chemicals used were analytical grade (CH<sub>3</sub>COONa: purity (98.5%) LTD, India, NaH<sub>2</sub>PO<sub>4</sub> • H<sub>2</sub>O: purity (>97%), United Kingdom, Na<sub>2</sub>HPO<sub>4</sub>: purity (98%), United Kingdom, KCl: purity (>99%) LTD, India, NHC<sub>4</sub>Cl: purity (99%), India, CdCl<sub>2</sub>: purity (99.99%) BDH, England). A weight-to-volume ratio of 40 g/160 mL catholyte was chosen as activated sludge to catholyte ratio.

A new cathodic mesh screen was used in each experiment and was cleaned by rinsing with a nitric acid solution (1 M) in an ultrasonic cleaner for 10 min and then thoroughly washed with double-distilled water before use. Experiments were carried out by stirring the anolyte for one hour at a rotational speed (300 rpm) using a magnetic bar without connecting the cathode and anode to the power source for activating the bacteria in the activated sludge, then the required voltage was provided to the circuit using DC power supply by connecting the positive wire to the positive electrode while the negative to the cathode in series with a resistor 10  $\Omega$ . The experiments have been done at room temperature. After applying the required voltage, samples were taken every 10 minutes for the first hour, every 30 minutes for the second hour, and then every hour until the end of electrolysis at 4 hours. Cadmium concentration was measured by an atomic absorption spectrometer (Varian SpectrAA 200 Spectrometer).

#### 3- Results and Discussion

#### 3.1. Effect of Applied Voltage

To determine the order of the reaction kinetics of cadmium deposition on the bioelectrochemical cell with a packed bed rotating cathode, plots of concentration profile with time at different applied voltages were generated. Figure 2 shows the concentration profile with time at applied voltages of 0.6 V, 0.9 V, 1.2 V, 1.5 V and 1.8 V with the inset representing plots of ln (C/Co) versus time. The effect was studied with a constant concentration of cadmium at 150 mg/L, pH = 5, and a rotational speed of 300 rpm with electrolysis time of 4h(which have been determine based on a preliminary experiment using the mentioned conditions and applied voltage of 0.6 in which cadmium removal efficiency higher than95% was observed). It is observed that the relationship between ln (C/Co) and time is almost linear with the coefficient of determination ( $\mathbb{R}^2$ ) greater than 0.976 confirming that the cathodic precipitation of Cd is subject to a first-order reaction kinetic.

Table 1 displays values of the rate constant (k) at several applied voltages. It has been observed that by increasing the applied voltage, the rate constant increases and the relationship between the rate constant and the applied voltage is almost fitting to an exponential form [23]. This was interpreted as, the transfer rate of Cd from the solution near the cathode surface increases with the increase in the applied voltage while decreasing the side effect of hydrogen generation at the cathode. However, by increasing the applied voltage, a mass transfer limit occurs with a slight increase in rate constant at higher voltage [24].



Fig. 2. Plots concentration profile with time at different applied voltages. Inset: Inset: ln(C/Co) vs. Time.. Co=150mg/L, pH=5, 300rpm

Table 1. Rate constant at different applied voltage, Co=150mg/L, pH=5, 300rpm

E(Volt)	K(min <sup>-1</sup> )	$\mathbf{R}^2$
0.6	0.016930	0.985111
0.9	0.018028	0.983117
1.2	0.019286	0.977901
1.5	0.021708	0.976634
1.8	0.024226	0.976065

The relationship between the rate constant and the applied voltage can be determined by equation (2):

$$k = k_0 e^{-\beta(E)} \tag{2}$$

Or in linear form as:

$$\ln(k) = \ln(k_0) - \beta E \tag{3}$$

Where: k refers to the rate constant in min<sup>-1</sup>,  $k_o$  refers to the rate constant at no current supplied, and E refers to the applied voltage in Volt. $\beta$  represents the slope of the line.

Equation 2 shows that the dependence of the rate constant on the applied voltage is similar to the Arrhenius formula. Values of  $k_0$  and  $\beta$  can be determined from the plot of ln(k) versus applied voltage as shown in Figure 3. From Fig. **3**,- $\beta$ =0.282475and the value of  $k_0$  is equal 0.01408 min<sup>-1</sup> with R<sup>2</sup>=0.98784. A similar correlation was obtained by Khattab et al.[24] in their studying the kinetics of copper removal on packed bed cathode,and their relation was between the rate constant and current density instead of the applied voltage that used in the present research. However, both current density and applied voltage represent the driving force for the electrochemical reduction of heavy metals like the cadmium.



## 3.2. Effect of the Initial Concentration of Cadmium

The effect of the initial concentration of Cd ions on the rate constant was also considered. Fig. **4** shows the plots of concentration profile with time at different initial Cd concentration of 50, 100, 150, 200 and 250 mg/L with the inset representing plots of ln (C/Co) versus time. The effect was verified at a constant applied voltage of 1.5 V, pH = 5, and 300 rpm.



Fig. 4. Plots concentration profile with time at different initial concentration Inset: ln(C/Co) vs. Time. E=1.5V,pH=5,300rpm

From Fig. 4, it can be seen that the relationship between  $\ln(C/Co)$  and time is almost linear with a coefficient of determination  $(R^2)$  greater than 0.96. The rate constant values (k) at several initial cadmium concentrations are tabulated in Table 2. It was observed that the rate constant decreased with increasing initial concentration above 150ppm as shown in Fig. 5. The relation could be represented by a second order polynomial formula (k = 0.0167094 + 8.208542857E-005 [Cd] - 3.531714286E-007  $[Cd]^2$ ) with R<sup>2</sup> (0.9646). A similar trend was observed by Khattab et al. [24] In their study the kinetics of copper removal on a packed bed cathode. At higher concentrations, the system may come under mixed control and with increasing concentration, the system only became under control of electron transfer. However, such a phenomenon was not observed as confirmed by the high value of R<sup>2</sup>. A mixed control process occurred in previous studies [31, 32]

Table 2. Rate constant at different initial concentration E=1.5V,pH=5,300rpm

Conc.(mg/L)	K(min <sup>-1</sup> )	$\mathbf{R}^2$
50	0.020157	0.978781
100	0.020723	0.966769
150	0.021708	0.976634
200	0.018820	0.985742
250	0.015142	0.979934



Fig. 5. Effect of concentration on the rate constant (k)

#### 3.3. Effect of pH

To demonstrate the effect of pH on the rate constant, plots of concentration profile with time were generated at different pH values as shown in Fig. **6** with the inset plots of  $\ln(C/Co)$  versus time. The effect was studied at a constant voltage of 1.5 V, cadmium concentration of 150 mg/L, and rotation speed of 300 rpm.

From this set of plots, it can be seen that the relationship between ln (C/Co) and time is almost linear with the coefficient of determination ( $\mathbb{R}^2$ ) greater than 0.96 for pH value 5-7 but at lower pH  $\mathbb{R}^2$  became less than 0.94, hence a deviation from linearity was happened due to effect of side reaction (hydrogen generation). The rate constant values (k) at several initial pH values are also tabulated in Table 3. It was observed that the rate constant increased with increasing pH to a value of 6 and then started to a slightly decrease as the pH became more alkaline as shown in Fig. 7.

The relation between pH and k could be represented by second order polynomial formula а (k 0.0008373428571 0.007224785714 pH 0.0005799285714 pH<sup>2</sup>) with R<sup>2</sup> (0.9578). Explanation of this behavior depends on the effect of hydrogen evolution where a side reaction is occurred on the cathode and its rate increases with decreasing pH which leads to a decrease in the transfer of cadmium towards the cathode and then a decrease in the value of the rate constant. At a pH higher than 6, cadmium ions could be precipitated as hydroxides instead of a metal and then the cadmium deposition on cathode rate decreases [33]. Therefore, pH = 6 is taken into account when further study of the effect of rotational speed.



Fig. 6. Plots concentration profile with time at different at initial pH. Inset: Inset: ln(C/Co) vs. Time.. E=1.5V,Co=150mg/L,300rpm

Table 3. Rate constant at different initial pH, E=1.5V, Co=150mg/L, 300rpm

pН	K(min <sup>-1</sup> )	$R^2$
7	0.022872	0.993228
6	0.023805	0.961809
5	0.021708	0.976634
4	0.020968	0.933699
3	0.017163	0.943200



Fig. 7. Effect of pH on the rate constant (k)

### 3.4. Effect of Rotation Speed

The effect of cathode rotation speed on the rate constant was shown in Fig. 8 as plots of concentration profile with time at different rotation speeds with the inset representing the plots of  $\ln(C/Co)$  against time. The effect was verified at a constant voltage of 1.5 V, an initial cadmium concentration of 150 mg/L, and pH = 6. From this set of plots, it can be seen that the relationship between  $\ln(C/Co)$  and time is approximately linear with a coefficient of determination (R<sup>2</sup>) greater than 0.96 except at rotation speed of 500rpm. Rate constant values (k) at multiple rotational speeds are tabulated in Table **4** and Fig. **9**.

The relation could be represented by a second order polynomial formula (k = 0.00931242 + 6.615287143E-005 (rotation speed) - 7.190928571E-008 (rotation speed)<sup>2</sup> with R<sup>2</sup> (0.9608). It can be seen that increasing the rotational speed leads to increase the rate constant up to 300 rpm and beyond this value of the rotational speed, the rate constant starts to decline. This behavior can be explained as increasing the rotation speed leads to a decrease in the boundary layer thickness on the cathode surface and thus more cadmium ions can be deposited [20].



Fig. 8. Plots concentration profile with time at different rotation speed Inset: Inset: ln(C/Co) vs. Time E=1.5V,Co=150mg/L, pH=6

Table	4. IN	an	constant	ai	uniterent	Totation	specu
E=1.5	V, Co=	=150	mg/L, pH	=6			
Rotat	tion sp	eed	K(m <sup>2</sup>	in <sup>-1</sup> )		$\mathbf{R}^2$	

different rotation

Poto constant at

Table 4

rpm	$K(\min^{-1})$	$\mathbf{R}^2$	
100	0.015348	0.990402	
200	0.019015	0.985186	
300	0.023805	0.961809	
400	0.0234283	0.98932	
500	0.0246450	0.906346	



Fig. 9. Effect of rotation speed on the rate constant (k)

It is important to note that the final concentration of cadmium under different effect of parameters were in the range of 3.5 ppm to 0.5 ppm which is higher than the standard limit for drinking water according to Iraqi standards (0.003mg/l) [34]. However, the treated wastewater could be used for agricultural applications.

#### 4- Conclusions

It was found that the cathodic deposition of cadmium using a bioelectrochemical reactor with a packed bed rotating cylindrical cathode follows first-order kinetic properties. The results showed that the rate constant is affected by the applied voltage, the initial concentration of cadmium, pH and the rotational speed. An exponential correlation was observed between the rate constant and the applied voltage similar to the Arrhenius formula. Increasing the applied voltage enhances the rate constant while the concentration has the opposite effect. pH and rotational speed have different effects on the rate constant. Increasing pH from 3 to 6 results in increase the rate constant while after pH 6, a decrease in the rate constant was found. The rate constant increases with increasing rotational speed up to 300 rpm and then the rate constant decreases slightly beyond this value. From a scale up point of view, the results of the present work are important in choosing the appropriate values of the applied voltage and rotational speed to obtain the same hydrodynamic similarity at the industrial applications based on their optimum values of 1.1 volts and 300 rpm.

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# ازالة الكادميوم باستخدام مفاعل كهروكيميائي–احيائي ذات القطب الكاثودي الدوار ذو الحشوه الثابته: دراسة حركية التفاعل

زهراء على كاظم و على حسين عبار

قسم الهندسه الكيميائيه الاحيائيه/كلية الهندسه الخوارزمي/ جامعة بغداد

الخلاصة

تم دراست حركية التفاعل لازالة الكادميوم من المحاليل المائيه باستخدام مفاعل كهروكيميائي-احيائي ذات قطب كاثودي دوار مؤلف من حشوه ثابته. حيث تم دراسة تاثير كل من الجهد الكهربائي المسلط ,التركيز البدائي للكادميوم ,سرعة دوران الكاثود والداله الحامضيه على ثابت معدل التفاعل (k) . اظهرت النتائج ان الترسيب الكاثودي للكادميوم يحدث تحت تاثير انتقال الكتله لكل قيم الجهود الكهربائيه المسلطه وبناء على ذلك يمكن تمثيل العلاقة بين تغيير التركيز اللوغاريتمى مع الزمن بمعادلة تفاعل من المرتبه الاولى .

وجد ان ثابت معدل التفاعل يعتمد على الجهد الكهربائي المسلط , التركيز البدائي للكادميوم ,الداله الحامضيه وسرعة الدوران . حيث انه تمت زيادته مع زيادة الجهد المسلط وامتثلت علاقته بالجهد المسلط معادلة أسية.

ينخفض المعدل الثابت (k) مع زيادة التركيز الأولي للكادميوم أعلى من 150 ppm بينما تم زيادته عند التراكيز المنخفضة. الأس الهيدروجيني وسرعة الدوران لهما تأثيرات مختلفة على ثابت المعدل. تؤدي زيادة الأس الهيدروجيني من 3 إلى 6 إلى زيادة ثابت المعدل بينما يحدث انخفاض طفيف في ثابت المعدل عند الرقم الهيدروجيني = 7. زيادة سرعة التدوير من (100 rpm) الى (500 rpm) يؤدي الى زيادة ثابت معدل التفاعل ومع ذلك ، أصبح ثابت المعدل ثابتًا تقريبًا عند(300 rpm) .

الكلمات الدالة: الكادميوم, خلية التحليل الكهربائي الميكروبية, الخواص الحركية, ثابت معدل التفاعل