Kinetics of Electrochemical Removal of Nickel using Bio-electrochemical Reactor with Packed Bed Rotating Cylinder Cathode

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Abstract

The kinetics of nickel removal from aqueous solutions using a bio-electrochemical reactor with a packed bed rotating cylinder cathode was investigated. The effects of applied voltage, initial nickel concentration, the rotation speed of the cathode, and pH on the reaction rate constant (k) were studied. The results showed that the cathodic deposition occurred under mass transfer control for all values of the applied voltage used in this research. Accordingly, the relationship between concentration and time can be represented by a first-order equation. The rate constant was found to be dependent on the applied voltage, initial nickel concentration, pH, and rotation speed. It was increased as the applied voltage increased and decreased as the initial concentration increased. Its relation to the applied voltage can be fitted as follows:

\[ k = k_o e^{-\beta V} \]

where \( k_o = 0.01695 \text{ min}^{-1} \) and \( \beta = 0.431 \). pH and rotation speed have two dissimilar effects on the rate constant. Increasing the pH from 3-6 leads to an increase in the rate constant, while a decrease in the rate constant beyond pH=6 has occurred. Increasing the rotation from 100 to 300 rpm results in an increase in the rate constant. However, the rate constant decreases significantly beyond a rotation speed of 300 rpm.

Keywords: Nickel, Packed bed rotating electrode, Microbial electrolysis cell, Kinetics.

1. Introduction

Heavy metals are toxic, non-biodegradable, and could be bio-accumulated in an environment, so they have become the most important environmental issue nowadays [1]. Nickel is one of these metals which poses several health and environmental threats, and its removal from wastewater is mandatory [2].

Several methods have been utilized for nickel removal, such as physiochemical methods (adsorption, coagulation, chemical precipitation, oxidation), and membrane methods (ion exchange membrane, reverse osmosis)[3]. Most of these approaches are not effective, especially when heavy metals exist at low concentrations and suffer from high operational costs [4]. On the other hand, nickel can be removed by reduction on cathode via an electrochemical process, which is nowadays an efficient method in comparison with the traditional methods due to the utilization of electrons as a clean reducing agent [5]. Recently, the adoption of cathodic deposition has increased due to the application of three-dimensional electrodes that give a high mass transfer rate and a high specific surface area [6]. In this context, different designs of these fixed bed electrodes have been used, such as reticulated vitreous carbon, felts, metal-plated
foams, and screens [7]. However, nickel removal by cathodic deposition needs high energy to reduce it at the required potential [8]. This drawback has been overcome by using microbial electrolysis cells (MECs), which efficiently remove heavy metals at a considerably lower cost [9].

Two distinct regimes could be identified in the cathodic deposition process for any electrochemical process operated in the 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, metal percent removal increases linearly with time, while in the second case, exponential decay of the concentration with time occurs, combined with a decrease in current efficiency with time [10], [11]. 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 [12].

Many studies on the kinetics of nickel electrodeposition from an aqueous solution have been published [13]-[24]. Most of these studies studied the kinetics based on charge-transfer controlled electrode kinetics [13],[14] or diffusion-kinetic control associated with a typical nucleation process [15],[24] . A few studies were performed to study the kinetics of nickel removal under mass transfer contro [16],[17]. Djaenudin et al. (2017) investigated the kinetics of nickel removal using a double chamber electrodeposition cell (DCEC). They found that nickel ion removal in DCEC followed a first-order kinetic with a removal rate constant \( k = 0.12 \text{ h}^{-1} \) (R2= 0.956). Doan et al. [17] express the nickel electrodeposition using Sherwood and Reynolds numbers.

First-order kinetics is also documented in detail in the literature for other metals like copper. Chu et al.[19] investigated the kinetics of copper removal from aqueous solution using a packed bed cathode. They found that the removal rate of copper is under mass transfer and that the concentration of copper declines exponentially according to the following equation:

\[
C(t) = C_0 \exp \left[-\frac{zt}{v} \left(1 - \exp \left(-\frac{L}{v}\right)\right)\right]
\]

Where \( C(t) \) refers to copper concentration at any time \( t \), second, \( C_0 \) is initial copper concentration, \( mg/l \), \( a \) refers to cathodic cross-sectional area, \( cm^2 \), \( v \) is the superficial velocity (=volumetric flow rate/\( a \)), \( cm/s \), \( V \) is electrolyte volume, \( cm^3 \), \( L \) represents the characteristic length and its value was stated by the authors to be 7.3 cm [18].

In relevant research achieved by Ruotolo and Gubulin[23], the kinetic behavior of copper deposition on a fixed-bed electrochemical reactor was studied. They found that the decline of copper concentration is linear for the initial period of electrolysis and then changes to exponential for the rest. However, no rate expression has been reported.

Khattab et al.[11] investigated the kinetics of copper removal using a packed bed cathode. They discovered that the Cu(II) concentration profile with time can be represented by the rate equation:

\[
C(t) = C_0 \exp \left[-kt\right]
\]

where \( k \) refers to rate constant in \( \text{min}^{-1} \)

To our knowledge, no study has been performed to investigate the kinetics of nickel removal using a bio-electrochemical reactor with a packed bed rotating cylinder cathode, nor has a correlation of rate constant been reported based on this design of the electrochemical system.

In the present study, the kinetics of electrochemical removal of nickel from aqueous solution using a bio-electrochemical reactor with a packed bed rotating cylinder cathode were investigated based on the effect of four main parameters: the applied voltage, Ni (II) initial concentration, pH, and rotation speed.

2. Materials and Methods

The bio-electrochemical system adopted in the present research for nickel removal is composed of a rotating fixed bed bioelectrochemical reactor, an electrical motor (type, Phoenix-USA), a hotplate magnetic stirrer (IKA, Germany), a power supply (model UNI-T: UTP3315TF-L) with two Amperometers (type, Kwun Tong, Kowloon, Hong Kong). Figure 1 shows the schematic diagram of the electrochemical system. The bioelectrochemical cell is composed of a cylindrical cell body (15cm in diameter and 11 cm in length) ended above by a flange with dimensions of (15cm in diameter and a thickness of 0.5 cm). The cell and its flange were made from Perspex material. Cylindrical porous graphite with dimensions (10cm in length and 9.8 cm in diameter with 0.5 cm thickness) and having an apparent surface area of 270.176 cm2 was used as an anode. It was purchased from HP Graphite Handan Co.,Ltd. The cathode was a spiral-wound woven wire mesh rotating cylinder. It was made of continuous mesh layers of stainless steel wrapped around a central rode of the same material with dimensions of 15 cm x 0.8 cm. These wrapped mesh layers form a bed with an external diameter...
of 2 cm and a length of 4.8 cm and is bounded by two Teflon sleeves which have a 2.6 cm external diameter and a 2 cm length. The stainless steel woven screen used in the construction of the cathode has a mesh number of 30 wires per inch and its specific surface area was (38.06cm⁻¹) [19], [20]. The catholyte chamber is made from perspex material. It is composed of two main parts: the first is a supporting cylindrical perforated container having dimensions of (11 cm in length and 7 cm in diameter) perforated with 10 mm holes on its lateral surface. The distance between any two holes was (1.5 cm) and the total number of holes was 40.

This supporting container was ended on its top with a flange having the dimensions of (15 cm in diameter with a thickness of 0.5 cm) and contained four holes with a diameter of 0.4 cm for fixing the cathodic chamber with the cell body via bolts and nets. The second part is a cylindrical ion exchange membrane chamber. It was constructed from a cation exchange membrane in a cylindrical form with dimensions of (11 cm in length and 5.6 cm in diameter) joined at the bottom with a cylindrical Perspex disc with dimensions of (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 anode was inoculated with activated sludge as a source of bacteria taken from a local biological unit of Al-karkh wastewater treatment–No.2/Baghdad sewage department/Iraq and fed with the nutrient medium as a source of carbon-containing (per liter): CH₃COONa 1 g, NaH₂PO₄•H₂O 2.45 g, Na₂HPO₄ 4.58 g, KCl 0.13 g, NH₄Cl 0.31 g, adjusted to pH = 7 [21]. The cathode chamber was provided with a catholyte solution composed of nickel chloride (NiCl₂) at different concentrations based on the condition of the experiment. All chemicals used were analytical grade and purchased from Sigma Aldrich. A weight to volume ratio of 40g/160ml of catholyte as activated sludge to catholyte was chosen.

A fresh cathodic mesh screen was used in every experiment, and it was cleaned by rinsing with a nitric acid solution (1M) in an ultrasound cleaner for 10 min, then washed thoroughly with double-distilled water before using.

Experiments were conducted by the agitation of anolyte for one hour at rotation speed (300rpm) using a magnetic bar without connecting the cathode and anode to the power supply for activation of bacteria in the activated sludge, and then the required voltage was supplied to the circuit using the DC power supply by joining the positive lead to the anode while the negative lead of the power source was connected in series with a 10 resistor and the cathode. The electrochemical system was run at a temperature of 25 ± 2 °C. After
applying the required voltage, samples were taken every 10 min for the first hour, every 30 min for the second hour, and then every hour until the end of the electrolysis at 4 h. The concentration of nickel was measured by atomic absorption spectroscopy (Varian SpectrAA 200 spectrometer).

3. Results and Discussion

3.1 Effect of Applied Voltage

To determine the order of reaction kinetics of nickel deposition on the bioelectrochemical cell with a packed bed rotating cylinder cathode, plots of \( \ln(C/C_0) \) against the time at different applied voltages were constructed. Figure 2 shows the plots of \( \ln(C/C_0) \) against time at an applied voltage of 0.5V, 0.7V, 0.9V, 1.1V, and 1.3V with the inset representing the corresponding fitting lines. The effect has been investigated at a constant concentration of nickel at 150 mg/L, pH = 5, and a rotation speed of 300rpm. It was observed from the set of plots that the relation is almost linear with a coefficient of determination \( R^2 \) greater than 0.93, confirming that cathodic deposition of nickel obeys first-order reaction kinetics. Table 1 displays rate constant \( (k) \) values at several applied voltages. It was observed that by increasing the applied voltage, the rate constant increases and the relation between rate constant and applied voltage is almost fitted to an exponential form [11]. This was explained as the nickel transport rate from the solution tower to the cathode surface increases with the increase in the applied voltage, reducing the side effects of hydrogen generation at the cathode. However, by increasing the applied voltage, a mass transfer limitation occurs with a slight increase in rate constant at higher voltage [12]. Therefore, the correlation between rate constant and applied voltage could be designated by equation (3):

\[
k = k_o e^{-\beta(E)}
\]

\( \ldots (3) \)

Or in linear form as:

\[
\ln(k)=\ln(k_o) - \beta E
\]

\( \ldots (4) \)

where \( k \) refers to the rate constant in min\(^{-1}\), \( k_o \) refers to the rate constant at no current supplied, and \( E \) refers to the applied voltage in volts. \( \beta \) represents the slope of the line.

Equation 3 shows that the dependence of the rate constant on the applied voltage is similar to the Arrhenius form. The values of \( k_o \) and \( \beta \) can be calculated using the plot of \( \ln(k) \) versus applied voltage shown in Figure 3. From Figure 3, \( \beta = 0.431 \) and the value of \( k_o \) is equal to 0.01695 min\(^{-1}\) with \( R^2=0.995 \). A similar correlation was obtained by Khattab et al.[11] in their study of the kinetics of copper removal on packed bed cathode. The relation was between rate constant and current density instead of the applied voltage used in the present research. However, both current density and applied voltage represent the driving force for the electrochemical reduction of nickel.
Table 1,
Rate constant at different applied voltage.
\[ \text{C}_o=150\text{mg/L, pH=5, 300rpm} \]

| E(Volt) | \(K(\text{min}^{-1})\) | \(R^2\) |
|---------|----------------|--------|
| 0.5     | 0.02082        | 0.94838|
| 0.7     | 0.023233       | 0.93754|
| 0.9     | 0.024814       | 0.95496|
| 1.1     | 0.027100       | 0.97199|
| 1.3     | 0.0296         | 0.93859|

Fig. 3. Plot of \(\ln(K)\) against applied voltage.

3.2 Effect of the initial concentration of Nickel

The impact of the initial concentration of Ni (II) on the rate constant was also considered. Figure 4 shows the plots of \(\ln(C/C_o)\) against time at nickel initial concentrations of 50, 100, 150, 200, and 250 mg/L with the inset representing the corresponding fitting lines. The effect has been investigated at a constant applied voltage of 1.1V, pH=5, and 300rpm. From this set of plots, it can be seen that the relationship is almost linear with a coefficient of determination \(R^2\) greater than 0.94. Rate constant \(k\) values at several nickel initial concentrations are tabulated in Table 2. It was observed that the rate constant decreased as the initial concentration increased, as shown in figure 5. A similar trend was observed by Khattab et al.[11] in their study of the kinetics of copper removal on a packed bed cathode. At higher concentrations, the system may become under mixed control and, with increasing concentration, the system may become solely under electron transfer control. However, no such phenomenon was observed, as confirmed by the high value of \(R^2\). Mixed control operations have happened in previous studies [18], [23].
Fig. 4. Plots of $\ln(C/C_0)$ against time at different initial concentration. Inset: fitting curves. $E=1.1V$, $pH=5$, $300rpm$.

Table 9,
Rate constant at different initial concentration.
$E=1.1V$, $pH=5$, $300rpm$

| Conc.(mg/L) | K(min$^{-1}$) | R2       |
|------------|--------------|----------|
| 50         | 0.027827     | 0.969077 |
| 100        | 0.026573     | 0.981468 |
| 150        | 0.027100     | 0.971993 |
| 200        | 0.019241     | 0.941139 |
| 250        | 0.014824     | 0.972635 |

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

To demonstrate the impact of pH on the rate constant, plots of ln(C/Co) against time at different values of pH were constructed as shown in Figure 6, with the inset representing the corresponding fitting lines. The effect has been investigated at a constant applied voltage of 1.1V, a nickel concentration of 100mg/L, and 300rpm. From this set of plots, it can be seen that the relationship between ln(C/Co) and time is almost linear with a coefficient of determination (R²) greater than 0.95. Rate constant (k) values at several initial pH values are tabulated in Table 3. It was observed that the rate constant increased with increasing pH up to a value of 6, then started to decrease as pH became more alkaline as shown in figure 7. The interpretation of this behavior depends on the effect of hydrogen evolution as a side reaction occurred on the cathode where its rate increases as pH decreases, leading to a lower transfer of nickel towards the cathode. Hence the low value of the rate constant was observed. At pH higher than 6, nickel ions may be precipitated as hydroxides rather than as metal, hence the rate of nickel deposition decreases [22]. Therefore, pH = 6 is considered in further investigation of the effect of rotation speed.

![Figure 6. Plots of ln(C/Co) against time at different initial pH. Inset: fitting curves. E=1.1V, Co=100mg/L, 300rpm](image)

| pH | k(min⁻¹) | R²  |
|----|----------|-----|
| 7  | 0.018339 | 0.974491 |
| 6  | 0.027829 | 0.969513 |
| 5  | 0.026573 | 0.981468 |
| 4  | 0.026069 | 0.972654 |
| 3  | 0.016042 | 0.989982 |

Table 3, Rate constant at different initial pH. E=1.1V, Co=100mg/L, 300rpm
3.4 Effect of Rotation Speed

The impact of cathode rotation speed on the rate constant was shown in Figure 8 as plots of $\ln(C/C_0)$ against time at different rotation speeds, with the inset representing the corresponding fitting lines. The effect has been investigated at a constant applied voltage of 1.1V, an initial nickel concentration of 100mg/L, and pH=6. From this set of plots, it can be seen that the relationship is almost linear with a coefficient of determination ($R^2$) greater than 0.95. Rate constant ($k$) values at several rotation speeds are tabulated in Table 2 in Figure 9. It can be seen that increasing rotation speed results in an increasing rate constant up to 300rpm, and beyond the rotation speed value of 300 rpm, the rate constant starts to decline. This behavior can be interpreted as increasing the rotation speed results in decreasing the thickness of the boundary layer on the cathode surface; hence more nickel ions could be deposited [7].
Table 11,
Rate constant at different rotation speed.
E=1.1V, Co=100mg/L, pH=6

| Rotation speed (rpm) | K(min⁻¹)  | R²        |
|----------------------|-----------|-----------|
| 100                  | 0.017811  | 0.968299  |
| 200                  | 0.020271  | 0.960155  |
| 300                  | 0.027829  | 0.969513  |
| 400                  | 0.024358  | 0.974501  |
| 500                  | 0.021013  | 0.928637  |

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

4. Conclusions

The cathodic deposition of nickel using a biocatalytic reactor with a packed bed rotating cylinder cathode was found to follow first-order kinetics. Results show that the rate constant is affected by the applied voltage, initial concentration of nickel, pH, and rotation speed. An exponential correlation between rate constant and applied voltage similar to the Arrhenius form was observed. Increasing the applied voltage leads to enhancement of the rate constant, while concentration has the opposite effect. pH and rotation speed have two dissimilar effects on the rate constant. Increasing pH from 3-6 leads to an increase in the rate constant, while beyond pH 6, the rate constant was found to be declining. The rate constant increases as the rotation speed increases up to 300rpm, then the rate constant declines beyond this value. From a scale-up point of view, the results of the present work are significant in selecting the appropriate values of the applied voltage and rotation speed to get the same hydrodynamic similarity in the industrial application based on their optimum values, namely 1.1 volts and 300rpm.

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دراسة حركية التفاعل في الازالة الكهروكيميائية للكيبل باستخدام مفاعل كهروكيميائي احيائي ذات القطب الكاثودي الدوار ذو الحشوة الثابت

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الخلاصة

تم دراسة حركة التفاعل لإزالة البكتريت باستخدام مفاعل كهروكيميائي أحيائي ذات قطب كاثودي دوار مؤلف من حشوة ثابتة. حيث تم دراسة تأثير كل من الجهد الكهربائي المسلط، التركيز البصري للبكتريت، سرعة الدورة الكهربائي والدورة الحساسية على ثبات معدل التفاعل (k). أظهرت النتائج ان الترسب البكتريي ينخفض تحت تأثير انتقال الكثافة لكل جهد الكهربائي المسلط وبناء على ذلك يمكن تمثيل العلاقة بين ثبات التركيز للبكتريت مع الزمن بعادلة ثابتة من المعرفة الأولية. وجد ان ثبات معدل التفاعل يعتمد على الجهد الكهربائي المسلط، التركيز البصري للبكتريت، الدورة الحساسية وسرعة الدورة. حيث انه يزداد مع زيادة قيمة الجهد الكهربائي المسطح وقابلية مع زيادة تركيز البكتريت. بالإضافة إلى مثال علاقته مع الجهد الكهربائي المسطح والدورة.

تعتبر العملية الأساسية المحتملة لعملية الترسب الكهربائي بالصعوبة التالية:

\[ k = k_0 e^{-\beta U} \]

حيث أن (k) ثابت قيمة تساوي (0.16) و (β) قيمة تساوي (0.43).

أظهرت التأثير كل من الدورة الحساسية وسرعة الدورة على ثبات معدل التفاعل غير متوازي حيث أن زيادة الدورة الحساسية من (6) إلى (3) يؤدي إلى زيادة ثابت معدل التفاعل بينما عند زيادة الدورة الحساسية إلى قيمة أعلى من (6) يتوقف ثابت معدل التفاعل. زيادة سرعة الدورة من (300 rpm) إلى (100 rpm) يؤدي إلى زيادة ثابت معدل التفاعل وهذا ينخفض تدريجيا بعد زيادة سرعة الدورة الحساسية إلى قيمة أعلى من (300 rpm).