



## Kinetic Study of Cementation Cadmium Ions Using Zinc Powder from Synthetic Sulfate Solutions

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Received: 27 06 2024; Accepted: 27 04 2025

Available: 30 04 2026

**Abstract:** Zinc production faces a significant challenge from cadmium (Cd<sup>2+</sup>). Therefore, this study investigated the cementation of cadmium from sulfate solutions with zinc powder. This work examined how pH of the solution, reaction time, and temperature affect cadmium cementation. The primary focus of the study was the impact of varying initial pH (1–5) on Cd<sup>2+</sup> removal efficiency (%) at 20 minutes. Results showed maximum cadmium cementation efficiency at a pH of 4. The cadmium cementation process was also affected by reaction times (20, 40, 60, and 80 min). Zinc powder was used to study cadmium cementation reaction kinetics in sulfate solutions at various temperatures (50, 60, 70, and 80 °C). We found that the reaction rate constant (K) rose with temperature, peaking at 80 °C. This study found the best pH, reaction time, and temperature to be 4, 80 minutes, and 80°C, respectively. When various theories were used to calculate reaction kinetics, activation energies of 13.75297 and 12.18379 were revealed. These results indicate that the Arrhenius theory best explains the reaction mechanism in this study. SEM and EDS analyses revealed a cadmium deposit. These results offer more insight into cadmium cementation.

**Keywords:** Cadmium cementation, zinc powder, synthetic sulfate solution, kinetics.

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Peer Review under the responsibility of Universidad Nacional Autónoma de México.

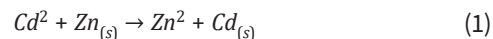
## 1. Introduction

Industrial processes, including mining, chemical industries, and metal processing, produce massive amounts of wastewater (Meng et al., 2021; Mekhelf et al., 2022). Large amounts of heavy metals in these waters threaten the environment (Hama et al., 2023). Heavy metals are stable and non-degradable. Examples of heavy metals include lead, cadmium, iron, and nickel (Yan et al., 2020; Karm et al., 2024<sub>a</sub>; Karm et al., 2024<sub>b</sub>).

Cadmium ( $\text{Cd}^{2+}$ ) is one of the most toxic environmental contaminants and poses a significant threat to human health and ecosystems due to its tendency to bioaccumulate and its relative mobility in the environment (Rao et al., 2010). In addition, cadmium can cause environmental and occupational hazards. In 2017, cadmium metal and its compounds were among the first group of metals considered toxic and carcinogenic by the International Agency for Research on Cancer (LIANG et al., 2013). In 2023, cadmium metal was valued at about \$5,500 per ton (Han et al., 2022). Therefore, extracting and separating cadmium increases financial gains and is more compatible with environmental conservation. Hydrometallurgical methods used in mineral extraction are less energy-consuming than pyrometallurgical methods (Öztürk et al., 2023). In addition, most hydrometallurgical methods use strong acids, as they help dissolve unwanted elements, enabling successful recovery operations.

Among the hydrometallurgy methods is the cementation method (Laubertová et al., 2021). The cementation method spontaneously involves electrochemical processes and is considered a heterogeneous method. It is characterized by being highly efficient, simple, inexpensive, sludge-free, capable of recovering metals with high purity, and requiring low energy. Zinc sulfide, lead, and copper ores naturally contain cadmium in low concentrations (Han et al., 2022). Cadmium ores are not abundant, but most of the cadmium is in zinc ores because of their easy isomorphous behavior (Rao et al., 2010). Zinc is considered one of the most widely used metals in the world, with applications ranging from galvanization to the production of various metal alloys (Han et al., 2022). Electrolysis in zinc sulfate solution is among the most common methods for producing zinc (Fosnacht & O'Keefe, 1983). Since zinc sulfate solutions contain metal impurities, these impurities, which have a higher chemical potential than zinc, must be purified before electrolysis begins (Han et al., 2022; Fosnacht & O'Keefe, 1983). The purity of zinc sulfate solutions is improved by reducing energy consumption and ensuring high purity (Han et al., 2022). The hot and

cold purification methods eliminate cobalt, cadmium, and nickel ions. Removal of cadmium ion ( $\text{Cd}^{2+}$ ) from acidic zinc sulfate solutions using the cementation method generally follows the following electrochemical reaction (Schroeder, 1965):



Nosier (2003) studied the cementation of cadmium for removal of cadmium from industrially contaminated water using zinc. They concluded that cadmium removal increases with temperature, agitation speed, initial cadmium concentration, and time. The activation energy was also studied. It was concluded that the spread of cadmium molecules on the surface of zinc controls cadmium production. It was confirmed by Ku et al. (2007) that, in aqueous solutions, the cementation of cadmium using zinc powder is governed by the acidity of the solutions, and that the consumption of both zinc and cadmium depends on it. The cementation remains in acidic media because of the ease of adhesion in these media, and the reactions, according to their results, follow first-order reactions.

First-order mechanisms have explained cementation reactions (Aurousseau et al., 2004). Regarding the reaction mechanism and kinetics of the reaction, there is still some uncertainty. Differences in first-order motor mechanisms have been documented by researchers (Guerra & Dresinger, 1999; Hiskey & Lee, 2003).

Sedzimir (2000) mainly criticized the passivation model and the empirical rate equation developed by Guerra and Dresinger (1999). Nan et al. (2019) used an ultrasonic field with an existing physical field to cement cadmium to solve the problems of floating cadmium sponge formation and concluded that the ultrasonic field for the cadmium cementation process reduces the formation of cadmium sponges. The use of acoustic fields addresses the researcher's objective, but it is not a better option for the cadmium deposition process due to the high cost of ultrasonic equipment. In addition, using ultrasonic fields in conjunction with an existing physical field leads to the formation of large numbers of floating cadmium sponges on the surfaces of the anode and cathode. Thus, an electrical short circuit occurs during cadmium deposition. In light of the above studies, most researchers focused on ways to improve cadmium cementation, but it was shown that there are problems, either operational or due to high cost and high energy consumption. In this study, zinc powder was used as both the anode and cathode, and a zinc sulfate solution served as the electrolyte. The reaction rate and activation energy were calculated at

different temperatures using mathematical modeling to describe cadmium cementation. Thus, this mathematical model prevents high operational costs. In this work, the effect of pH on the removal efficiency of cadmium ions ( $\text{Cd}^{2+}$ ) at pH (1, 2, 3, 4, and 5) was investigated. After the optimal selection of the pH for cadmium ion ( $\text{Cd}^{2+}$ ) cementation, the cementation process of cadmium ions ( $\text{Cd}^{2+}$ ) in a synthetic zinc sulfate solution was studied at different times and temperatures. Also, the cementation kinetics of cadmium and the activation energy of cadmium cementation in zinc sulfate solution were calculated using collision theory, Arrhenius theory, and transition-state theory.

## 2. Materials and Methods

A 2-liter glass reactor was used in all experiments, and an electric stirrer at 300 rpm was used. Zinc powder with 99.5% purity and a particle size of 75  $\mu\text{m}$  was used in this work as a sacrificial metal for the cementation of cadmium ions ( $\text{Cd}^{2+}$ ). A concentration of zinc sulfate solution 80 g/l was prepared by dissolving the analytical grade of the zinc sulfate powder (Merck) in distilled water. A solution containing 500 mg/ml  $\text{Cd}^{2+}$  was prepared by dissolving cadmium sulfate ( $\text{CdSO}_4 \cdot n\text{H}_2\text{O}$ , Wako Pure Chemical Industries, Ltd., Japan) in deionized water. The cadmium removal efficiency (RE %) was calculated using Equation 2 (Karm et al., 2024<sub>a</sub>) at different initial pH values (1, 2, 3, 4, and 5) for 20 min. Sulfuric acid ( $\text{H}_2\text{SO}_4$ ), and sodium hydroxide (NaOH) were used to adjust the acidity of the solution.

$$\text{RE}\% = \frac{(C_0 - C_f)}{C_0} \quad (2)$$

Where  $C_0$  represents the initial concentrations of  $\text{Cd}^{2+}$  and  $C_f$  represents the final concentrations of  $\text{Cd}^{2+}$ .

Also, to investigate the cementation of cadmium ions from sulfate solutions at various temperatures (50, 60, 70, and 80 °C) using a heater motor (Heidolph MR 3001 K) and shaking the solution at other times (20, 40, 60, and 80 min). After the reaction times had elapsed, the solution was filtered using a vacuum filtration pump. After the reaction times had passed, they filtered the solution using a vacuum filtration pump. After that, samples were collected from the filtered solutions, and the metal ions were analyzed by atomic absorption. This study employed a first-order reaction model to investigate the kinetics of cadmium cementation. Equation 3 (Ku et al., 2007) represents the first order.

$$\ln(C_0/C_f) = kt \quad (3)$$

Where  $k$  represents the constant of the reaction rate, and  $t$  represents the contact time of the reaction.

The activation energy of cadmium cementation reactions was calculated using Arrhenius, collision, and transfer theories. Equation 4 (Hobley, 2019; Rao et al., 2020) refers to the Arrhenius equation:

$$K = AT^m \exp\left(-\frac{E}{RT}\right) \quad (4)$$

Where  $m$  is represented by 0, 0.5, and 1 for theories of Arrhenius, collision, and transition state theories, respectively. Since the reaction constant depends on the temperature, Equation (5) can be applied and its natural logarithm can be taken, which becomes as follows:

$$\ln\left(\frac{K}{T^m}\right) = \ln A - \frac{E}{RT} \quad (5)$$

Where  $A$  represents the Arrhenius factor,  $T$  represents the temperature of the reaction,  $E$  represents the activation energy of the reaction, and  $R$  represents the constant of the gas.

The experimental procedure used in this study for the cementation of cadmium is illustrated in Figure 1.

## 3. Results

### 3.1 Reaction of Kinetic

The pH of the cementation solution is an important factor that indicates the thermodynamic stability of the cementation process. Therefore, it was necessary to determine the solution's pH. The different values of pH at (1, 2, 3, 4, and 5) were used to determine the optimal pH for this study. Figure 2 shows the  $\text{Cd}^{2+}$  removal efficiency and final pH in sulfate solution using Zn at different temperatures (50, 60, 70, and 80 °C) and different initial pH (1 to 5) for 20 min. From Figure 2, the cadmium removal efficiency is less than 20% at pH 1 and 2. Figure 2 also shows an increase in cadmium removal efficiency with increasing solution pH. The highest cadmium removal efficiencies were 24, 28.4, 36, and 38.2% for temperatures "50, 60, 70, and 80 °C", respectively, at pH 4 and 20 minutes. These results can be explained according to (Shayesteh et al., 2020), by the fact that at acidic pH values, the diffusion coefficient of hydrogen ions is higher than that of cadmium ions, and thus they reach and react easily with the zinc surface. But when the pH rises, i.e. when the medium is basic, the production of hydroxide concentration in the solution occurs, and these hydroxides deposited on the zinc surface form a layer that resists cadmium binding to zinc powder.

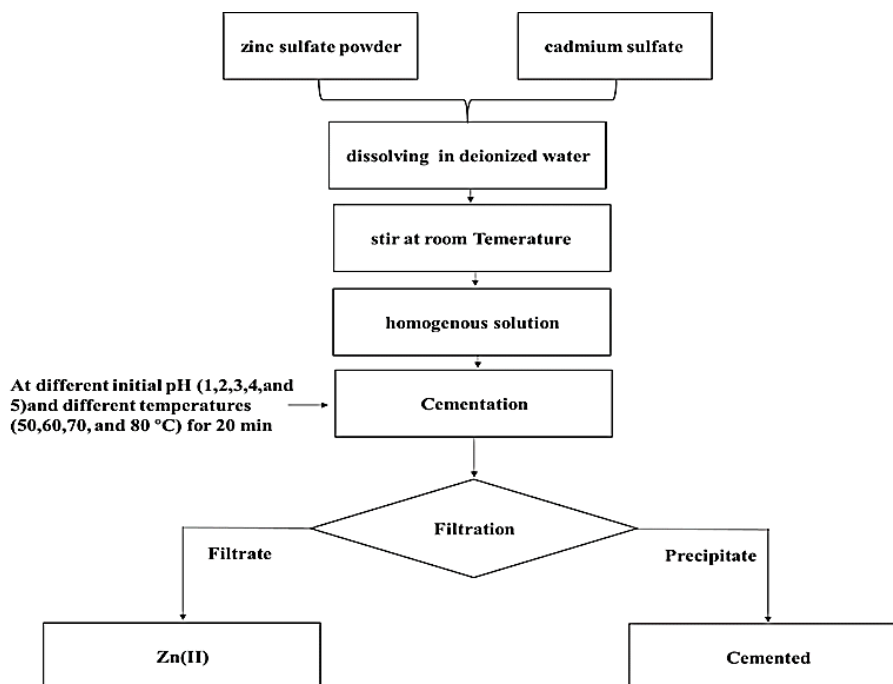
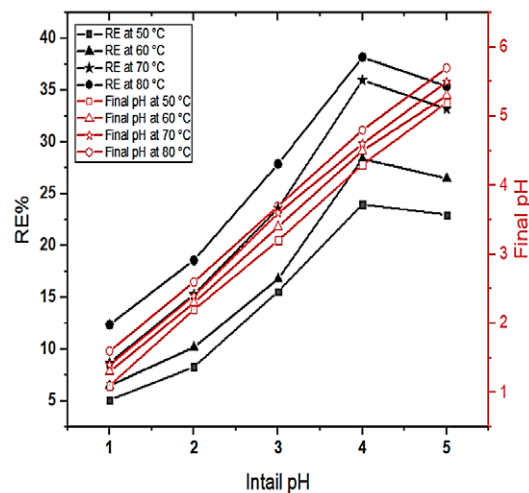


Figure 1. Flow chart for cadmium cementation.

The functional groups on the zinc surface remain protonated at low pH due to the high hydrogen concentration, which explains the low removal efficiency of cadmium from sulfate solutions at low pH, as shown in Figure 2. The removal efficiency of cadmium increases with increasing pH. The removal efficiency of cadmium and the final pH increase as the functional groups on the zinc surface become more negatively charged, thereby enhancing cadmium removal efficiency.

First-order reactions are one of the best ways to understand and study reaction order, as shown by plotting  $\ln(C/C_0)$  vs. time, as shown in Figure 3. Figure 3 shows different times for cadmium cementation (20, 40, 60, and 80 min) using the initial cadmium concentration of 500 mg/ml. It shows that the cementation rate of cadmium increases with increased reaction time. This can be explained by relying on (Karlsson et al., 2018), which confirms that in the case of short deposition time, the production of hydrogen ions decreases and the chance of hydroxides formation is high, and thus these hydroxides precipitate on the zinc surface forming a white precipitate that is difficult to spread in the solution and thus remains in the film layer, which leads to difficulty in reducing cadmium ions.

The reaction constant finding from Figure 3, and listed in Table 1, shows from the values of  $R^2$  that the cadmium cement followed a first-order reaction. From Figure 3, as

Figure 2. The removal efficiency (RE%) of  $Cd^{2+}$  and final pH of sulfate solutions at different initial pH for 20 min.

temperature increases, the rate of cadmium cementation increases, and the reaction constant also increases. This result, consistent with Vahidfarid et al. (2020), shows that cadmium ions diffuse faster on zinc surfaces at higher temperatures. This is because increasing the temperature increases the kinetic energy of the solution ions, which increases the diffusion rate of cadmium ions on the zinc surface, thereby increasing cadmium ion reduction.

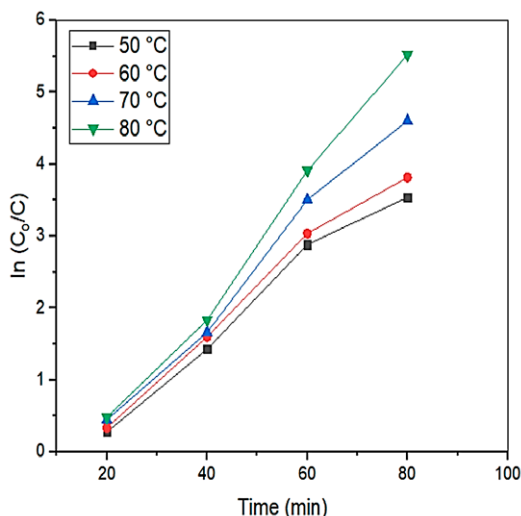


Figure 3. Effect of temperature on the time of the kinetic cadmium cementation: the initial cadmium concentration of 500 mg/ml.

However, when using a temperature higher than 85 °C, the reduction of hydrogen ions is the dominant reaction in the solution, thus reducing the reduction of cadmium ions. The highest reaction constant we obtained was 0.344 at 80 °C for 80 min.

Table 1 clearly shows that temperature affects the reaction rate constants. The modified Arrhenius equation (Equation 3) is considered a good equation for the dependence of the reaction constant on temperature.

Table 1. The kinetic of cementation of cadmium at different temperatures.

Temp (°C)	K (min <sup>-1</sup> )	R <sup>2</sup>
50	0.22507	0.98115
60	0.2377	0.987
70	0.28645	0.99
80	0.344	0.994

In this work, the Arrhenius, collision, and transition state theories were taken to evaluate and understand the effect of these different theories on calculating the activation energy of the reaction.

Figures 4, 5, and 6 show plots of Arrhenius, collision, and transition state theories, respectively, showing a plot of the values of  $\ln(k/T^m)$  versus  $(1/T)$ . Figures 3, 4, and 5 and Table 2 show that the activation energies are 13.75297, 12.18379, and 10.94719 kJ/mol for Arrhenius, collision, and transition state theories, respectively. R<sup>2</sup> is 0.94, 0.93, and 0.91613 for Arrhenius, collision, and transition

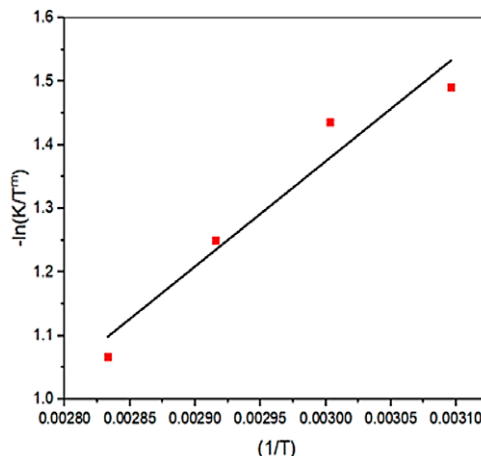


Figure 4. Plot of Arrhenius's theory for cadmium cementation: m=0; the initial cadmium concentration of 500 mg/ml.

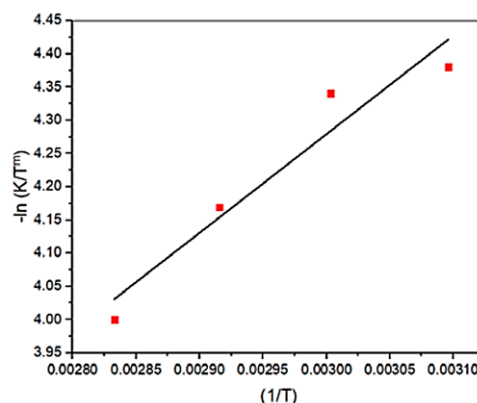


Figure 5. Plot of collision theory for cadmium cementation; m=0.5; the initial cadmium concentration of 500 mg/ml.

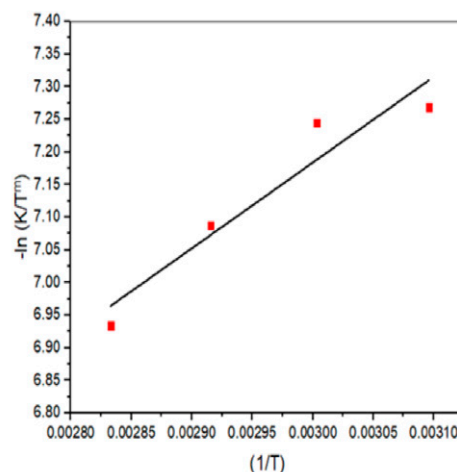


Figure 6. Plot of Transition State theory for cadmium cementation; m=1; the initial cadmium concentration of 500 mg/ml.

state theories, respectively. The results showed that as temperature increased, the activation energy increased, indicating that temperature provided the initial energy for the reaction to begin. These results show that the Arrhenius theory is the most accurate for calculating the activation energy. These results agree with those of Younesi et al. (2006), who reported activation energies of 9.9 and 7.2 kJ/mol for high and low cadmium concentrations in 60 g/L zinc sulfate solutions at 15 min, respectively. This study's activation energy results exceed those of Younesi et al. (2006), probably due to differing experimental conditions. The activation energy results with different values for cadmium cementation in water and industrial wastewater were also reported by Taha & Abd El-Ghani (2004), Nosier (2003), and Ku et al. 2007).

Table 2. Activation energy calculation for synthetic solution ( $\text{Cd}^{2+}$ ) using different theories.

m	E/R	E	R <sup>2</sup>
0	1654.194	13.75297	0.94
0.5	1465.455	12.18379	0.93
1	1316.717	10.94719	0.91

### 3.2 Morphology Study

The mechanism of reaction and adhesion of metal impurity ions on the zinc surface follows the contracted core model, which shows that the reaction first occurs on the outer surface of the zinc powder core, then a reaction occurs between the zinc powder core and the metal impurities over time, forming a layer of metal impurities on the zinc powder core. Over time, these layers become denser, and the zinc powder core becomes smoother (Younesi et al., 2006; Zhang et al., 2013; Pal et al., 2014; Karavasteva, 2014).

In this study, zinc powder served as both the anode and the cathode in the zinc sulfate solution, allowing ions to move easily between them, as shown in Figure 7. The contracted core model assumes that a layer covers the entire surface of the zinc powder, thereby hindering electron transfer between the cathode and the anode and allowing zinc powder to enter the electrolyte and precipitate zinc ions ( $\text{Zn}^{2+}$ ). Based on the above, we conclude that the contracted core model cannot explain the mechanism of cadmium deposition from the zinc sulfate solution. Figure 7 illustrates the mechanism of cadmium ion cementation by zinc powder. In this reaction, mass transfer is the controlling mechanism. Therefore, both steps 1 and 6 occur in the boundary layer. According to

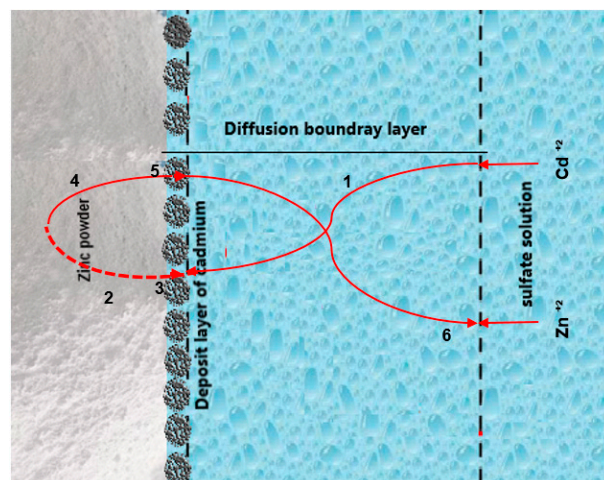


Figure 7. Mechanism of cadmium cementation using zinc anode and cadmium ion cathode.

Figure 7, the steps of the cementation reaction process are as follows:

1. Transfer of cadmium ions ( $\text{Cd}^{2+}$ ) from the sulfate solution to the interface of the metal–solution through the boundary layer.
2. Conducting electrons from the dissolved zinc anode to sites of the cathodic through adhesion between deposits and the electrons transfer.
3. The cadmium deposit dehydration and the formation of a crystalline network of the deposited cadmium atoms by linking the cadmium atoms together undergo the following electrochemical reaction:
 
$$\text{Cd}^{2+} + 2e \rightarrow \text{Cd}_{(s)} \quad (6)$$
4. Diffusion of zinc ions ( $\text{Zn}^{2+}$ ) from sites of the anode and hydration undergoes the following electrochemical reaction:
 
$$\text{Zn}_{(s)} - 2e \rightarrow \text{Zn}^{2+} \quad (7)$$
5.  $\text{Zn}^{2+}$  ions transferring to the solution through the cadmium sediment.
6.  $\text{Zn}^{2+}$  ions transfer to the sulfate solution through the layer of the boundary of the solution.

The scanning electron microscope (SEM) was used to study and demonstrate the morphology of the cadmium deposited at 20 and 80 minutes at 80 °C, as shown in Figure 8. Based on Figure 8, the morphology of the deposited layer is denser at the 80-minute sedimentation time than at the 20-minute sedimentation time. As is known, at the beginning of cadmium deposition, cadmium formation and growth occur in low-angle areas and sites with high

energy. Then it extends to other parts where thin cadmium lamellar forms after 20 min, as shown in Figure 8a. As the cementation of cadmium progresses, i.e., the reaction progresses, the cadmium covers the zinc, producing a thick layer, as shown in Figure 8b. These results are also consistent with (Thamir et al., 2017; Mohammed et al., 2021) that the coating layer is denser with an increase in the resulting layer roughness, which results from the increase in grain size resulting from the increase in deposition rates, which results in the deposition of a thick layer.

To verify the success of the cadmium cementation method, EDS was performed for the particles remaining from the cementation of cadmium, where the examination of EDS results, as shown in Figure 9 and Table 3, showed the presence of cadmium and zinc elements, which indicates that cadmium was deposited on zinc. Therefore, these results indicate the success of the cadmium cementation method from the zinc sulfate solution.

Table 3. EDS results analysis for cadmium deposition.

Element	wt%
Zn K	33.9
Cd K	36.5
O K	29.6

## Conclusions

Lately, research into cleaner, more efficient cadmium extraction has focused on improving zinc powder cementation, a cost-effective, proven technique ideal for industrial applications.

In this work, the cadmium cementation method was used. The cementation method is a simple, highly efficient way to cement high-purity metal, and it is also inexpensive compared to other methods. Researchers have also conducted studies on the kinetics of the cementation process to forecast energy requirements.

1. The cadmium removal efficiency was evaluated using different pH levels (1, 2, 3, 4, and 5). The highest removal efficiency was 38.2% at pH 4 for 20 minutes.
2. The cementation rate of cadmium ions increases with the increased reaction time. Also, increasing temperature increases the reaction rate constant and thus improves the efficiency of cadmium cementation; the highest reaction constant obtained was 0.344 at 80 °C for 80 minutes.
3. The provided data on activation energy supported the Arrhenius theories and collision theory, and the

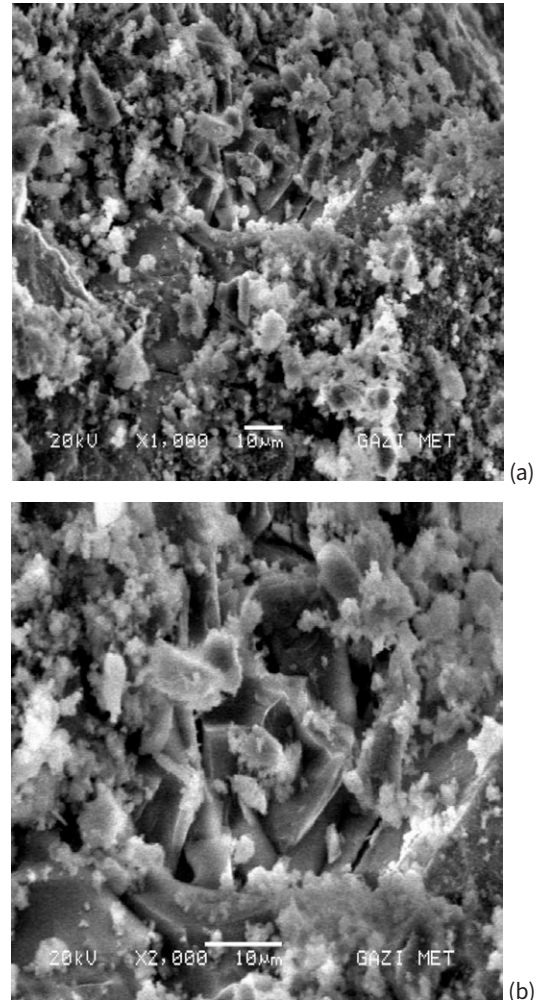


Figure 8. SEM of precipitated cadmium using the initial cadmium concentration of 500 mg/ml at 80 °C (a) 20 min (b) 80 min.

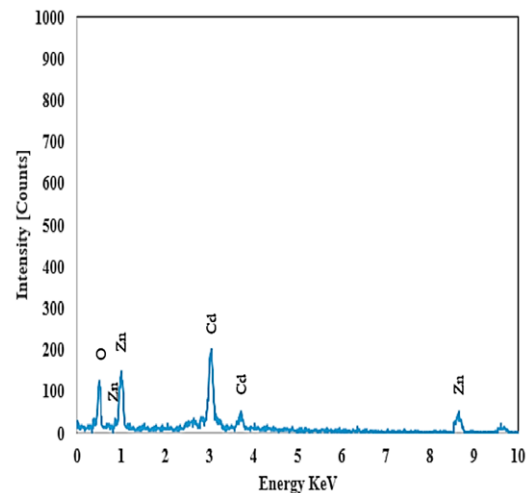


Figure 9. EDS of cementation of cadmium using the initial cadmium concentration of 500 mg/ml at 80 °C for 80 min.

obtained transition state. Arrhenius's theoretical data proved highly suitable.

- The results of the SEM image showed that the cadmium layer deposited was denser and more cohesive at 80 minutes than at 20 minutes.
- The results of the EDS image showed the presence of cadmium and zinc elements, which indicates that cadmium was deposited on zinc.

## Acknowledgments

The authors thank the College of Production Engineering and Metallurgy/University of Technology for providing the necessary support to complete this work.

## Funding

The authors have no funding for this work.

## Conflict of interest

There are no conflicts of interest to be cleared by the authors.

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