Removal of Cadmium in the Cold Purification Step by Semi-batch Process

Pourya Abbasi[1], Keivan Shayesteh[1, *], Vahid Vahidfard[1], Hossein Jangara[1]

[1] *Chemical Engineering Department, University of Mohaghegh Ardabili, Ardabil, Iran*

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ABSTRACT

Since the presence of cadmium ions in the zinc production process causes many problems, removing this impurity is necessary at the cold purification stage. This study considers cadmium removal in the cold purification process of the Dandi zinc smelting plant (Zanjan-Iran). To this end, the cadmium cementation was conducted by using zinc dust in the leached solution of the Dandi zinc smelting plant and the synthetic solution. Furthermore, modeling of the system is performed to predict the behavior of cadmium removal. Therefore, cadmium cementation kinetic was studied at different temperatures, and the reaction rate constants were obtained. Then, the governing differential equation of cadmium removal was solved analytically. Finally, to verify the model, the outcomes obtained from modeling were compared with the experimental data. Results showed that the mathematical modeling outcomes agree with the experimental data.

1. Introduction

Zinc is one of the most commonly used metals in the world, and it is extensively employed in galvanization and the production of various alloys [1]. The most common method of zinc production is the electrolysis of the zinc sulfate solution [2]. Zinc manufacturing plants usually include four sections: leaching, cold and hot purification, electrolysis, and casting [3]. The impurities with a higher chemical potential than the zinc must be purified from the zinc sulfate solution before electrolysis [4]. The purity of the electrolyte solution reduces energy consumption and ensures high-purity zinc production [5, 6]. The main impurities removed from the solution during the cold and hot purification processes are cobalt, nickel, and cadmium [7]. During the hot purification in the Dandi zinc smelting plant, cobalt is usually removed at 80-90 °C by adding potassium permanganate and lime water [8, 9]. On the other hand, during cold purification, nickel and cadmium are removed from the electrolyte solution at 75-80 °C by adding zinc dust [10, 11].

Currently, cementation is one of the most effective and efficient methods to purify the zinc sulfate solution and recycle toxic and precious metals from industrial wastewater [12]. Cementation of the impurities by the zinc dust is the removal or reduction of materials with higher chemical potential than that of the zinc [13]. In previous research, the cementation reaction kinetic is described as a first-order chemical reaction, but some researchers also reported deviation from the first-order kinetic regime [14-18].

Usually, in the leached solution from zinc ore, cadmium has the highest concentration in comparison with other impurities [19]. Since cadmium is nobler than zinc, cadmium deposits in the electrolysis process before or simultaneously with zinc. This issue reduces the purity of the zinc and leads to zinc dissolving in acidic electrolytes. As a result, zinc acts as a catalyst to reduce the hydrogen ions and produce hydrogen gas, significantly decreasing efficiency [20, 21].

In previous studies, cadmium cementation was conducted with zinc dust in different conditions. Aurousseau et al. [22] investigated the kinetics of cadmium cementation by zinc dust in low-level

∗ Corresponding author.; e-mail: k.shayesteh@uma.ac.ir

ultrasonic. Their study showed that ultrasonic amplification in the range of 50 to 120 W is desirable. Also, they observed that the high initial concentration of cadmium causes an increase in the reaction rate, whereas the concentration of zinc ions does not affect the reaction kinetics.

Nosier [23] studied cadmium cementation to remove cadmium from industrial wastewater using zinc dust. They concluded that the removal reaction rate of cadmium increases with increasing agitation speed, temperature, cadmium initial concentration, and residence time. Also, they investigated the activation energy of the reaction and observed that the cadmium cementation was controlled by cadmium molecular diffusion to the zinc dust surface. Yong Ku et al. [24] considered the cadmium cementation by zinc dust in aqueous solution. They found that the amount of cadmium and zinc consumed intensely depended on the solution's pH, and the cementation reaction occurs well in the acidic medium. According to their results, the reaction kinetic regime was reported as the first order. They represented that sodium sulfonate and anionic surfactants significantly increased the cadmium removal reaction rate.

Abaza et al. [25] studied the magnetic field's effect on cadmium's cementation rate by zinc dust. They observed that the cadmium cementation rate increases significantly with the increase in the magnetic field. Halikia et al. [20] stated that the zinc dust dissolution and the deposition of cadmium ions are in direct proportion with the stirrer speed, the cadmium initial concentration, and the concentration of the additive in the solution. Also, they found that increasing the solution pH causes the high dissolution of zinc dust in the solution and decreases the cadmium removal.

Later, Younesi et al. [26] considered the cadmium cementation by zinc dust in a batch reactor. They investigated the effect of cadmium initial concentration on the removal of cadmium at pH ranges from 5.2 to 5.4. They showed that when cadmium concentration in the solution is more significant than 1000 ppm and less than 500 ppm, the ash diffusion and film diffusion models control the process, respectively. They also found that when the cadmium concentration in the solution is between 500 and 1000 ppm, the combination of the ash diffusion and film diffusion models controls the overall rate of cadmium removal. Finally, they concluded that the cadmium cementation with zinc dust is a first-order reaction. Most researchers believe cadmium ion cementation is a first-order reaction, and the diffusion model controls the reaction rate. Although removing cadmium ions by zinc dust is

thermodynamically appropriate, in practice, it rarely approaches the equilibrium condition. Therefore, the information required for the design and operational purposes depends more on the reaction rate than the thermodynamic condition [23, 24, 27, 28].

In all previous research, the studies were conducted in batch reactors. Still, the semi-batch method was studied for the first time to optimize time, energy, and higher efficiency in zinc production factories. This study investigates cadmium removal from synthetic and leached zinc sulfate solution. To this end, cadmium cementation's kinetic and activation energy reaction rate was studied at different reaction temperatures. Since mathematical modeling of the process can prevent high operating costs, cadmium cementation modeling was performed to describe the cadmium removal process at the cold purification process of the Dandi zinc smelting plant. Also, the cadmium cementation was conducted in the laboratory and industrial scales. Results showed that the mathematical modeling of the process is in good agreement with experimental and industrial outcomes. So, the results demonstrate that the modeling of the system is reliable and accurate to predict the process.

2. Experimental setup and procedure

Experimental studies are divided into two parts. In the first section, all experiments were carried out for reaction constant calculations in a two-liter batch reactor. 2 g/lit of industrial-grade zinc dust was used. Sulfuric acid and NaOH (Merck, Germany) were used to adjust the pH of the solution. The solution was prepared by creating 600 mg/ml cadmium in a zinc sulfate solution at a concentration of 85 g/lit in the laboratory. One liter of the solution was poured into the reactor, and the pH was adjusted to 4.5. A heater heated the solution (Heidoloph MR 3001 K) to reach the desired temperature. Then, specific amounts of zinc dust were added to the reactor, and the stirrer mixed the solution. After a while and the proper mixing, the mixture was filtered. Afterward, the solution was sampled, and the impurities were measured by an atomic absorption device (AA 240-Varian, Australia).

In the second part of the experiments, the cadmium cementation was performed in a reservoir of 40 m3. The reactor volume was filled with a solution up to 36 m3 to improve the mixing quality. The volumetric flow rate of the inlet to the reactor was 1.2 m3/min. The zinc dust was added when half the reactor volume was filled with leached solution. When the leached solution was fully introduced into the reactor, the zinc dust was added so that its concentration remained at 2.5 g/lit. At this level, 15 min was considered for the reaction. After

filling the reactor, the reaction occurred 45 minutes in the batch condition. Finally, the solution was evacuated with a 0.6 m3/min flow rate. Then, the solution was sampled at different time steps, and the samples were analyzed using the atomic absorption device.

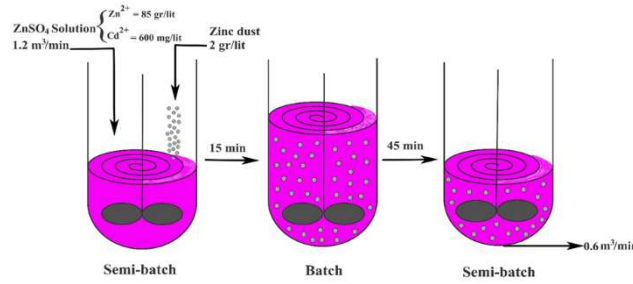Fig. 1 shows the schematic view of the experiment in three stages.

Fig. 1. Schematic view of the experiment.

3.Model development

Mathematical modeling plays a determining role in the study and analysis of the system. Mathematical modeling aims to obtain an equation that shows the effect of different parameters on the system's behavior to optimize time and prevent extra costs. Since the exact analytical solution is accurate and reliable for system analysis, an explicit analytical solution has been provided for the present study.

Suppose the cementation reaction occurs by zinc dust in a reactor. Three stages were considered to remove the cadmium from the solution. Accordingly, modeling was divided into three parts. In the first part, the reactor was filled by a leached solution with 1.2 m3/min. When the first half of the reactor volume was filled, 45 kg of zinc dust was added. Filling the second half of the reactor was 15 minutes, during which the cementation reaction occurred. Afterward, the reactor was worked in the semi-batch condition in the third stage. Hence, the mass balance was written for the semi-batch reactor at an unsteady state for the first part of the model:

$$C_{cd_0} v_0 = (-r_A) \times V(t) + \frac{d(C_{cd_0}V)}{dt} \qquad (1)$$

According to that of the reaction introduced as first order in the previous research [23, 24, 27, 28], the reaction kinetic was considered as follows: $r_{cd} = -kC_{cd}$ (2)

Finally, the governing differential equation of the cadmium from the Eq. 1 is obtained as follows:

$$\frac{1+k\tau}{\tau}C_{cd} - \frac{C_{cd_0}}{\tau} = -\frac{dC_{cd}}{dt} \qquad (3)$$

Solving Eq. 3 with the initial condition $C_{cd}|_{t=0} = C_{cd_0} = 480ppm$ leads to the concentration profile of cadmium:

$$C_{cd} = \frac{C_{cd_0}}{k\tau} + \frac{C_{cd_0}\tau_0}{\tau}\exp(-kt) - \frac{C_{cd_0}}{k\tau}\exp(-kt) \qquad (4)$$

V denotes the volume of the leached solution, τ is the residence time, k is the reaction constant, C_{cd0} is the initial concentration of cadmium, t is the time, and v_0 is the volumetric flow rate. In the second part of the model, by cutting off the reactor inlet, a residence time (60 min) was considered to occur in the reaction. So, the reactor works in batch condition, and the governing differential equation is as follows:

$$\frac{dC_{cd}}{dt} = -kC_{cd} \qquad (5)$$

Integrating and solving Eq. 5 while considering the obtained concentration at the end of the previous part of the model as the initial condition, the concentration of cadmium in the different reaction times was obtained as:

$$C_{cd} = C_{cd_0}\exp(-kt) \qquad (6)$$

Eventually, after the end of the previous stage, draining of the reactor beginsprevious stage's end, the reactor drains. Sixty minutes were allocated to the draining process. Therefore, the volume of the solution decreases over time. Writing the mass balance for this stage leads to the following differential equation:

$$vC_{cd} + \frac{d(C_{cd}V)}{dt} + (-r_{cd})V = 0 \qquad (7)$$

After simplifying Eq. (7), the governing differential equation of the third part of the purification process was obtained as follows:

$$\frac{dC_{cd}}{dt} = -kC_{cd}$$

(8)

Considering the cadmium concentration value at the end of the second stage as the initial value, the solution to Eq. 8 was obtained, giving the cadmium concentration at various times. Therefore, the analytical solution for the last part of the model is as follows:

$$C_{cd}(t) = C_{cd_0}e^{-kt}$$

(9)

4.Results and discussion

4.1.Reaction kinetic

Cadmium cementation was studied at different temperatures. The cadmium reaction rate concerning cadmium concentration is assumed to be a first-order reaction. Therefore, $\ln(C_{cd0}/C_{cd})$ versus time was plotted to obtain the reaction constant for cadmium cementation at different temperatures. The results are presented in Table 1. According to the R^2 values, the first-order reaction assumption is correct. Since the cadmium cementation was considered a first-order reaction, Eq. 10 was used to understand the cadmium removal kinetics:

$$\ln\left(\frac{C_{cd_0}}{C_{cd}}\right) = kt$$

(10)

Fig. 2 shows the variations of $\ln(C_{cd0}/C_{cd})$ over time at various temperatures for the solution containing 480 ppm cadmium and 85 g/lit zinc sulfate. According to Fig. 2, it is evident that with increasing temperature, the rate of cadmium cementation by zinc dust rises. At the beginning of the process, there is no significant change in the concentration at different temperatures. However, increasing the residence time causes the reaction rate to grow, and a high removal percentage of cadmium is achieved. As shown in Fig. 2, the temperature significantly affects the cadmium cementation in the synthetic solution without any impurities. It is clear that as the temperature rises, the slope of the lines increases, and accordingly, the reaction constant increases. The reaction constants for cadmium cementation at different temperatures are given in Table 1. Table 1 shows a significant difference in the reaction constants at low temperatures compared to those at high temperatures. In other words, a high reaction constant is obtained at high temperatures. Therefore, the best temperature for removing cadmium by zinc dust is achieved at 85 °C.
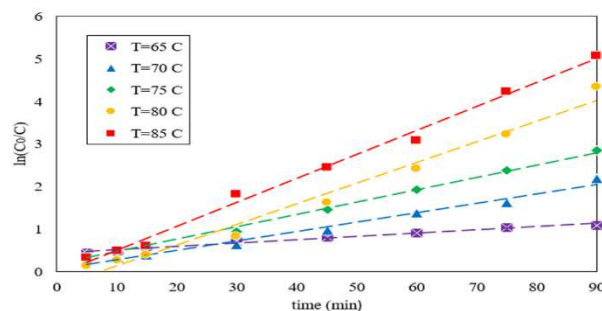

Fig. 2. ln(Ccd0/Ccd) versus time at different temperatures in the synthetic solution.

Table 1. Cadmium reaction is kinetic at different temperatures.

T (C)	k (min⁻¹)	R²
65	0.0078	0.9834
70	0.0219	0.9857
75	0.029	0.9955
80	0.0484	0.9811
85	0.0562	0.9937

In Fig. 3, the reaction kinetics of cadmium removal was investigated in a leached solution of the Dandi zinc smelting plant in the presence of 170 mg/liter of nickel. Most of the research is usually based on synthetic solutions, and few researchers have investigated leached solutions [23, 24, 26-29]. Hence, in this study, both synthetic and leached solutions were studied. The results of the leached solution investigation showed that the effect of temperature on the reaction constant is negligible. At low temperatures, the reaction of cadmium cementation by zinc dust can be done well. In Fig. 3, $\ln(C_{cd0}/C_{cd})$ was plotted against time to obtain the

kinetic cadmium reaction in the leached solution made from zinc ore leaching. In this figure, the slope of the lines at temperatures between 65-85°C do not differ significantly. Consequently, the reaction constants are so close together, and cadmium removal kinetic shows that temperature is not effective on the rate of cadmium cementation in the leached solution of the Dandi smelting plant. The reaction constants obtained in various temperatures are given in Table 2.

Table 2. Cadmium and Nickel reaction kinetic at different temperatures

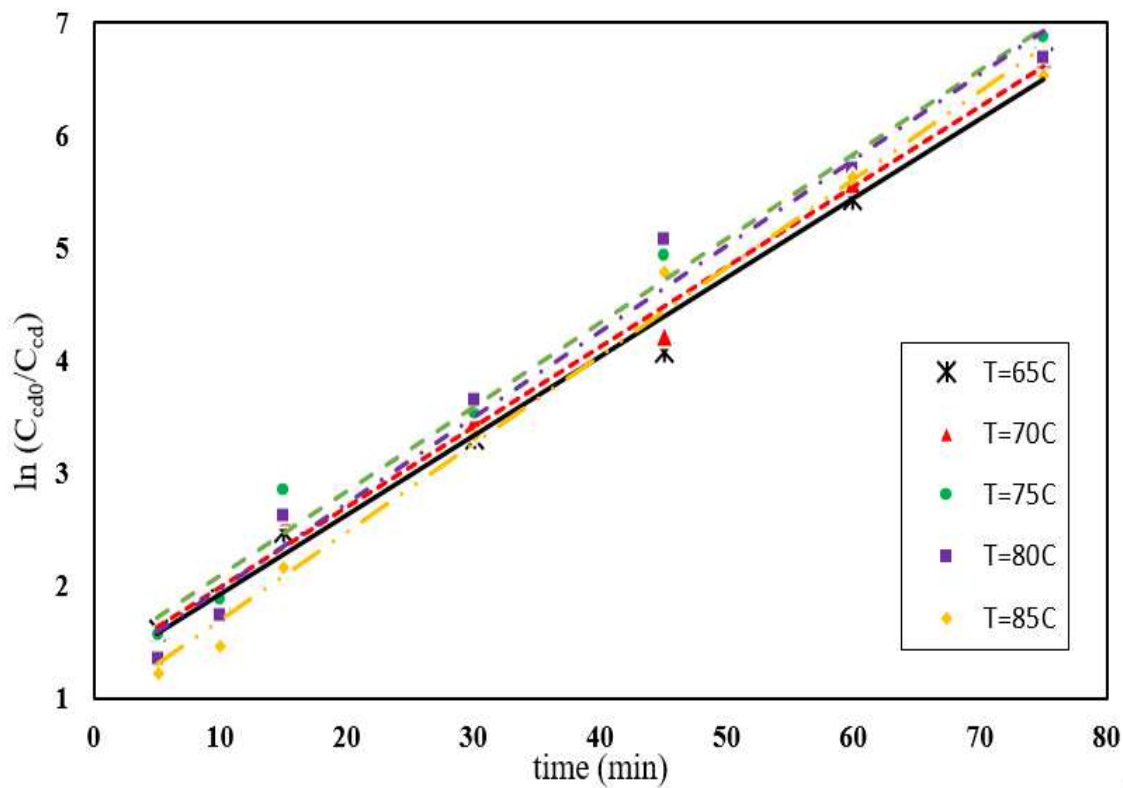| T (C)	k (min^{-1})	R^2
65	0.0703	0.9916
70	0.0711	0.992
75	0.0747	0.9889
80	0.0762	0.9816
85	0.0783	0.9903

Fig 3. $\ln(Ccd_0/Ccd)$ versus time at different temperatures in the leached solution.

4.2.Activation energy

It is clear from Tables 1 and 2 that the reaction rate constants are dependent on the temperatures. According to the modified Arrhenius equation (Eq. 11), the reaction constant dependence on temperature is well represented as follows:

$$k = AT^m \exp(-E/RT)$$ (11)

Applying the natural logarithm in Eq. 11 leads to:

$$\ln\left(\frac{k}{T^m}\right) = \ln(A) - \frac{E}{RT}$$ (12)

Ln (k/Tm) values were plotted against (1/T) to evaluate the effect of different theories on the determination of the activation energy of the reaction, where k denotes the reaction rate constant, and R is the universal gas constant. Also, m equals 0, 0.5, and 1 for Arrhenius, collision, and transition state theories. Fig. 4 shows the activation energy calculation for the synthetic solution, and Fig. 5 illustrates the activation energy for the leached solution using the three theories above. Figs 4 and 5 show that Arrhenius is the best theory for calculating activation energy. The activation energy of the cadmium cementation in the synthetic and the leached solution of the Dandi zinc smelting plant was calculated using Arrhenius, collision, and transition state theories. The results are presented in Tables 3 and 4. The high activation energy of cadmium cementation

in the synthetic solution indicates its high dependence on temperature variations. In other words, temperature provides only the energy needed to start the reaction. But when the leached solution (zinc ore solution) was used, impurities played a catalytic role and caused the activation energy of cadmium cementation dependency on the temperature to decrease significantly [30]. Zheng et al. and Yang et al. [31, 32] found that the cobalt removal from the Cd-Zn and Cd-Co alloys in the presence of cadmium increases the reaction rate of cobalt cementation. Also, the sources confirmed the effect of arsenic, antimony, copper, lead, and tin on the cobalt cementation rate [33-37].

Regarding the previous research, different activation energy values were presented for cadmium cementation. Younessi et al. [26] obtained the activation energy at high and low cadmium concentrations in the 60 g/liter zinc sulfate solution at 15 minutes of 9.9 and 7.2 kJ/mol, respectively. Taha et al. [28], Nosier et al., and Ku et al. [23, 24] Also reported different activation energy values for cadmium cementation in water and industrial wastewater solutions. The results of this study show that the presence of nickel and other impurities in the leached solution is highly effective in the activation energy of cadmium cementation reduction.
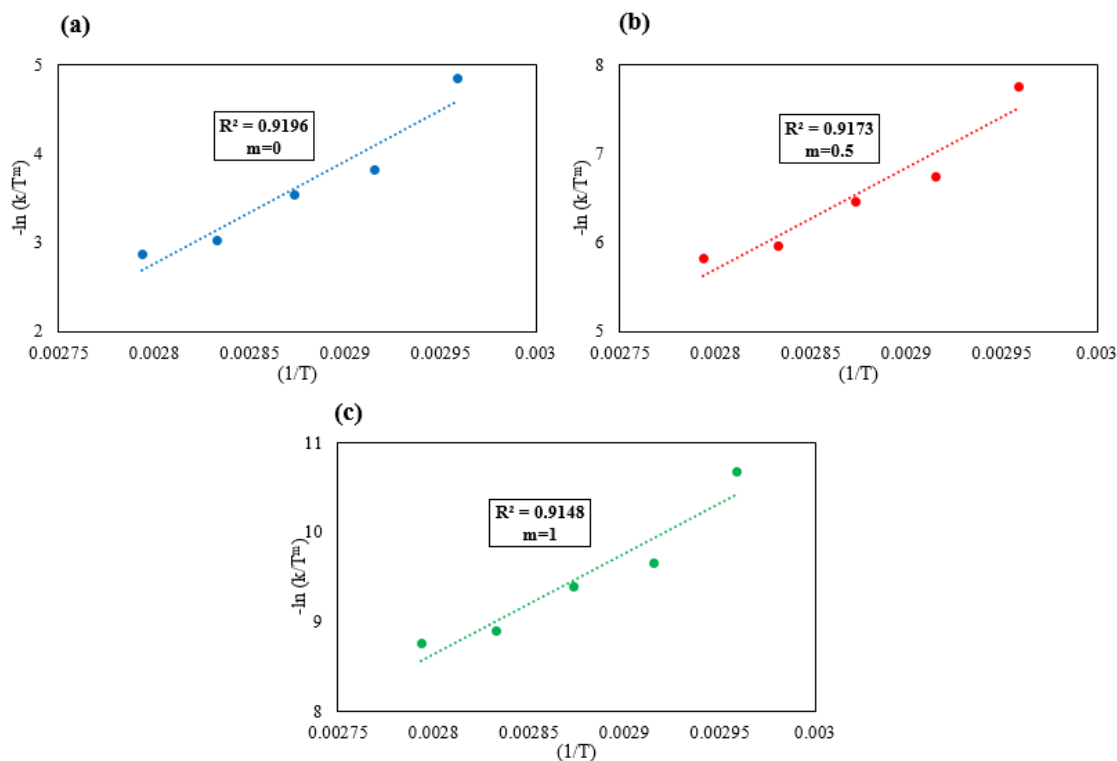

Fig 4. Activation energy calculation for the synthetic solution: a) collision theory, b) Arrhenius theory, c) transition state theory.

Table 3. Activation energy calculation for synthetic solution (Cd) using different theories.

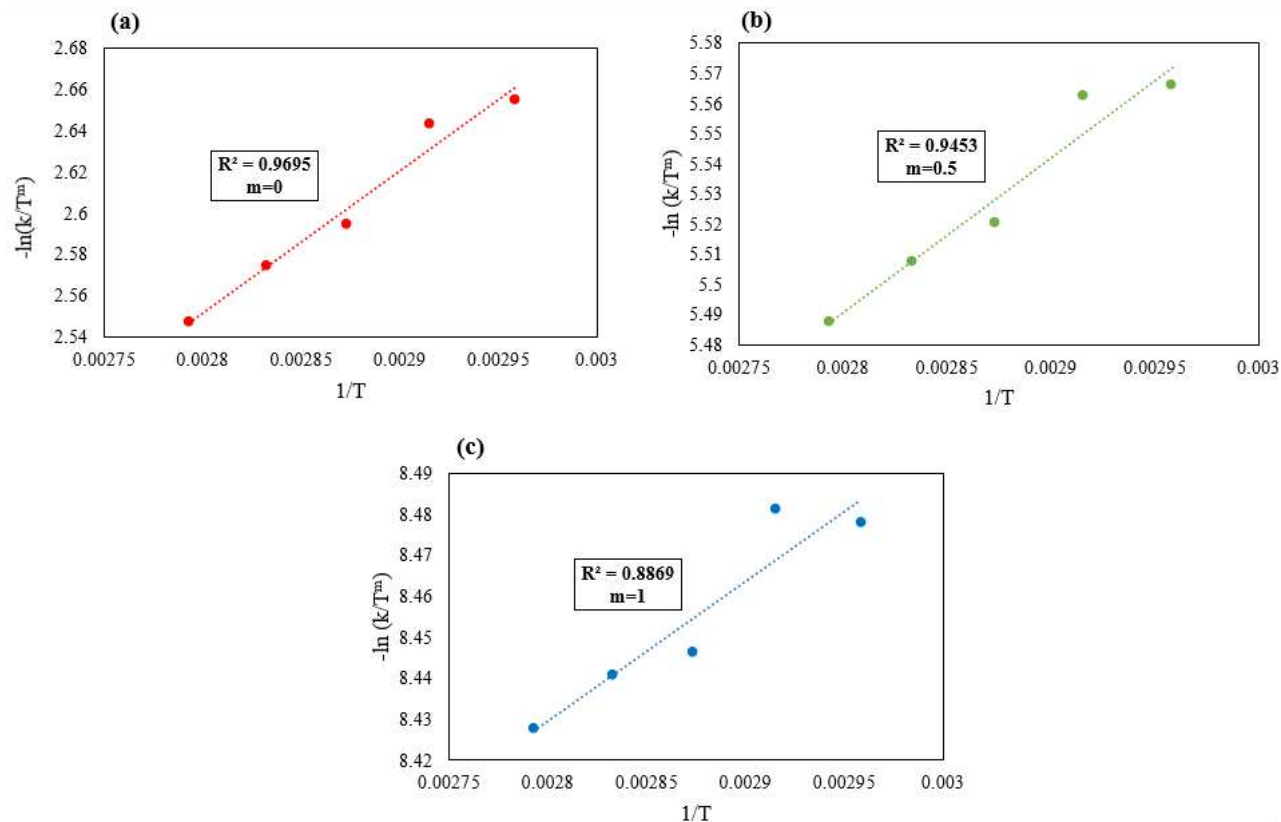| m	E_a/R	E_a(Kj)	R^2
0	11529	95.852	0.9196
0.5	11354	94.397	0.9173
1	11181	92.958	0.9148

(a)

(b)

(c)

Fig 5. Activation energy calculation for the leached solution: a) collision theory, b) Arrhenius theory, c) transition state theory.

Table 4. Activation energy calculation for leached solution (Cd-Ni) using different theories.

m	E_a/R	E_a(Kj)	R^2
0	689.09	5.729	0.9695
0.5	513.91	4.272	0.9453
1	341.3	2.837	0.8869

4.3.Modeling of the system

Since optimizing the effective parameters in the purification process is very important, mathematical modeling can provide a good description of the system and help to study the system accurately. According to Figs 6 and 7, concentration profiles of cadmium cementation in the synthetic and leached solution are illustrated at different temperatures in the three stages of the purification process.

As it is clear from the figures, increasing the temperature significantly affects the cadmium reaction rate in synthetic solutions. Meanwhile, in the leached solution, the effect of temperature variation on the cadmium removal rate is negligible. The low impact of temperature on the removal rate in the leached solution is due to the presence of other impurities in the solution that play the catalyst role. Also, Figs. 6 and 7 show a significant difference between the concentration profiles of cadmium in the synthetic and the leached solution, representing the high removal efficiency of the leached solution compared to the synthetic.
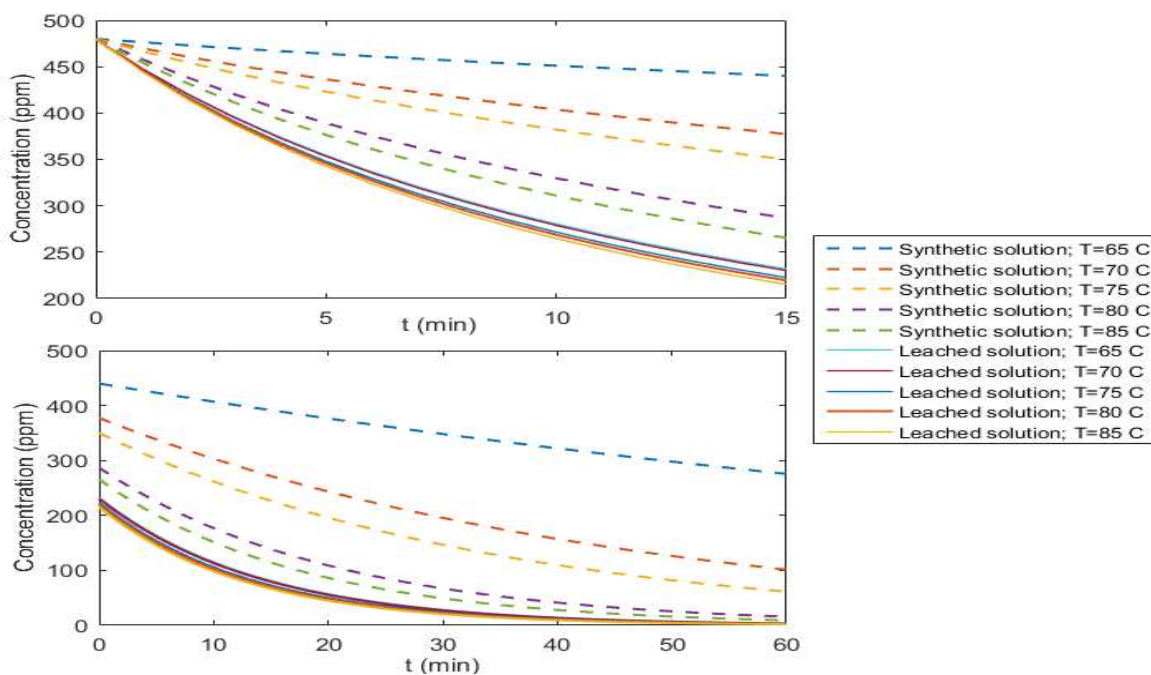
Fig. 6. Cadmium concentration profile at different temperatures for a synthetic and leached solution for a) first stage; b) second stage.
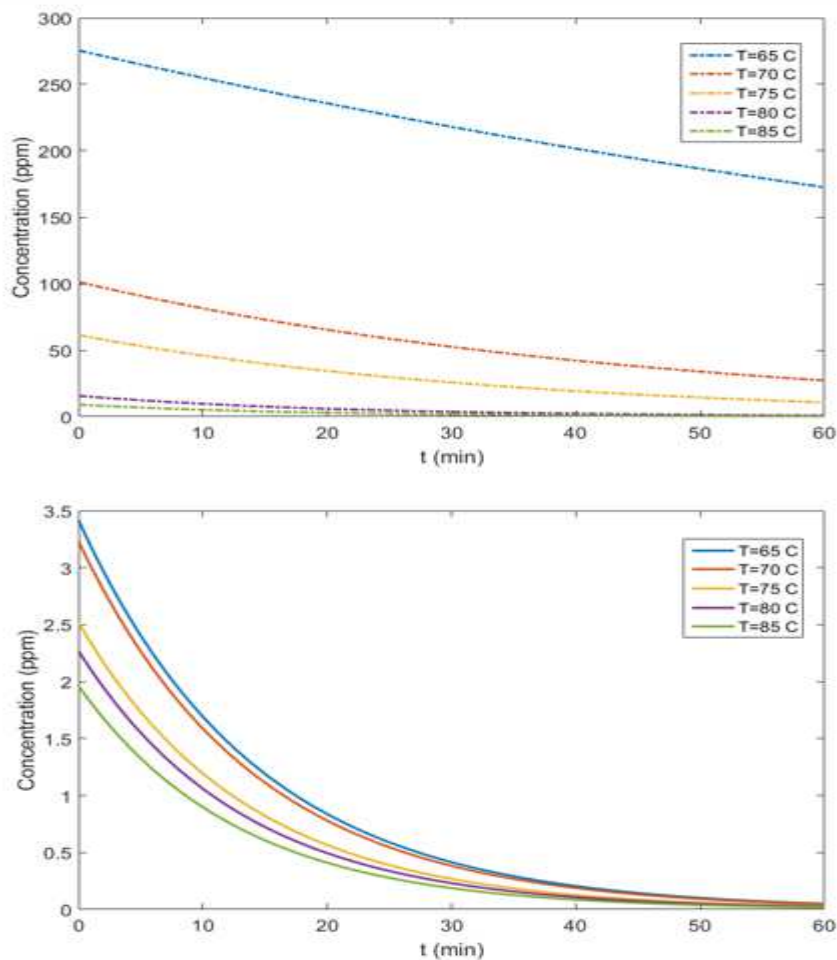

Fig. 7. Cadmium concentration profiles at different temperatures in the third stage of the process: a) synthetic solution; b) leached solution

Fig. 8 illustrates the cadmium concentration profiles obtained from mathematical modeling and experimental data. A comparison of the modeling and experimental outcomes shows a good agreement between them. As it is clear from the figure, in the first stage of the process (semi-batch), the concentration of cadmium ions decreases significantly by increasing the time. At this stage, about 50% of the cadmium ions were removed from the leached solution. This issue causes less cadmium to enter the second stage of the purification process (batch). As shown in Fig. 8b, cadmium concentration decreases significantly due to the high

residence time of the process. When the concentration of cadmium reaches less than 2 ppm, the third part begins, which is the evacuation of the reactor. Fig. 8c shows the concentration profile of cadmium against time at the third stage. According to the figure, increasing the period decreases cadmium concentration and finally approaches zero. However, because the discharge stage takes a lot of time and changes in concentration of cadmium are negligible, to save energy, it is better to ignore this step or consider the less time to the reaction.
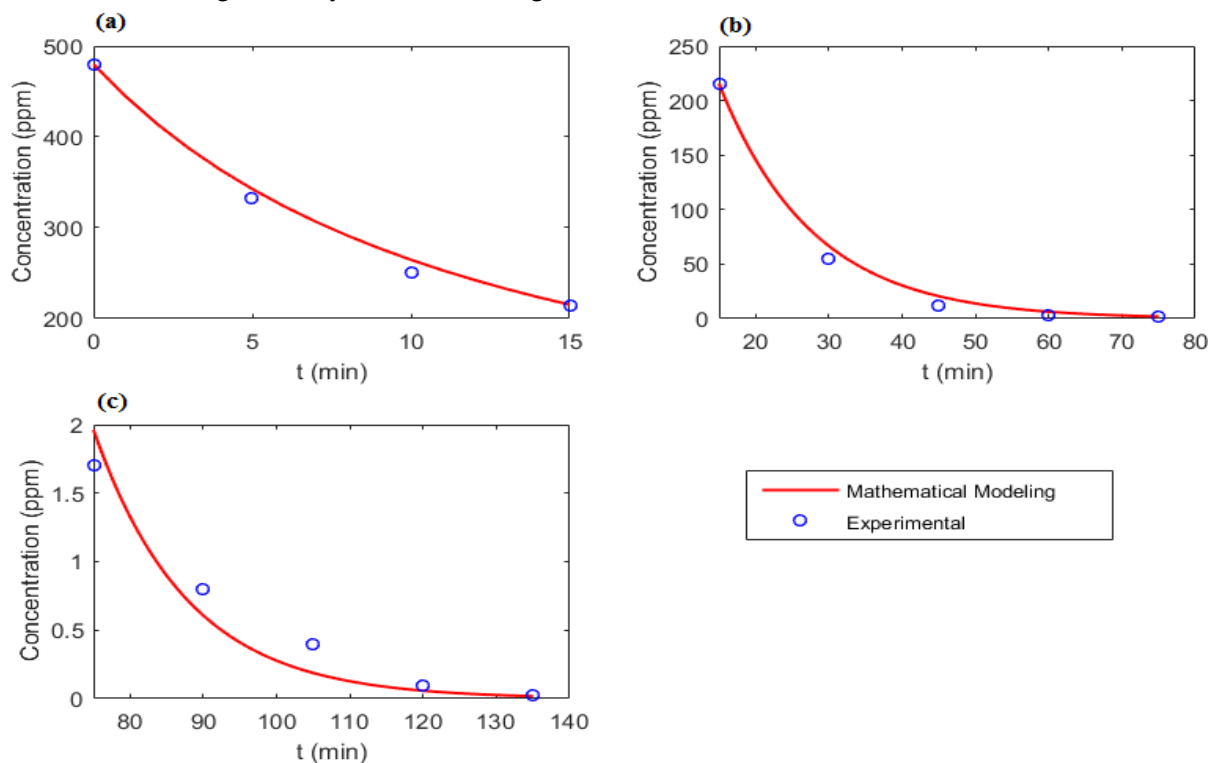

Fig. 8. Comparison of mathematical modeling and experimental outcomes: a) first stage; b) second stage; c) third stage.

4.4. Surface morphology survey

Fig. 9a shows the zinc dust before the cadmium cementation. As it is clear, the zinc dust has a smooth and uniform surface before the reaction. Fig. 9b shows the deposition morphology of the cadmium in the synthetic solution. Also, Fig. 9c demonstrates the deposition morphology of the cadmium cementation in the leached solution of the Dandi zinc smelting plant after the reaction. It is clear from Figs 9b and 9c that the surface of the zinc dust is crushed after the cementation process. Accordingly, zinc dust was removed from the surface as zinc ions entered the solution. Cadmium ions have also deposited on the zinc dust as the small particles form. A comparison of Figs 9b and 9c shows that when only cadmium ions are present in the solution,

a lower amount of zinc dust is reacted. Regarding Figs 9b and 9c, the zinc dust layers that are not reacted are quite clear.

According to Fig. 9b, a few impurities are on the surface of zinc dust. Comparing the SEM images shows that cadmium cementation is better on the surface of the zinc dust in the presence of impurities such as nickel, etc. In addition, more amounts of zinc dust were dissolved and recovered in the solution to remove impurities. Figs 9a and 9c shows that the shrinking core model Younesi et al. [23] presented for cadmium cementation needs to be verified.

The cadmium cementation by zinc dust is an electrochemical reaction [38]. Three factors are needed

for this reaction: anode, cathode, and electrolyte [39]. In this work, zinc dust layers act as anode and cathode, and the zinc sulfate solution acts as an electrolyte, which causes the transfer of ions between the anode and the cathode. If the shrinking core model is considered, the surface of the zinc dust is wholly covered, and the transfer of ions between the cathode and the anode is

disrupted. In other words, covering the entire zinc dust surface with ash complicates the electron transfer between zinc and cadmium, and zinc dust cannot enter the solution as zinc ions (Zn2+). Hence, the shrinking core model could not explain the mechanism of cadmium cementation.
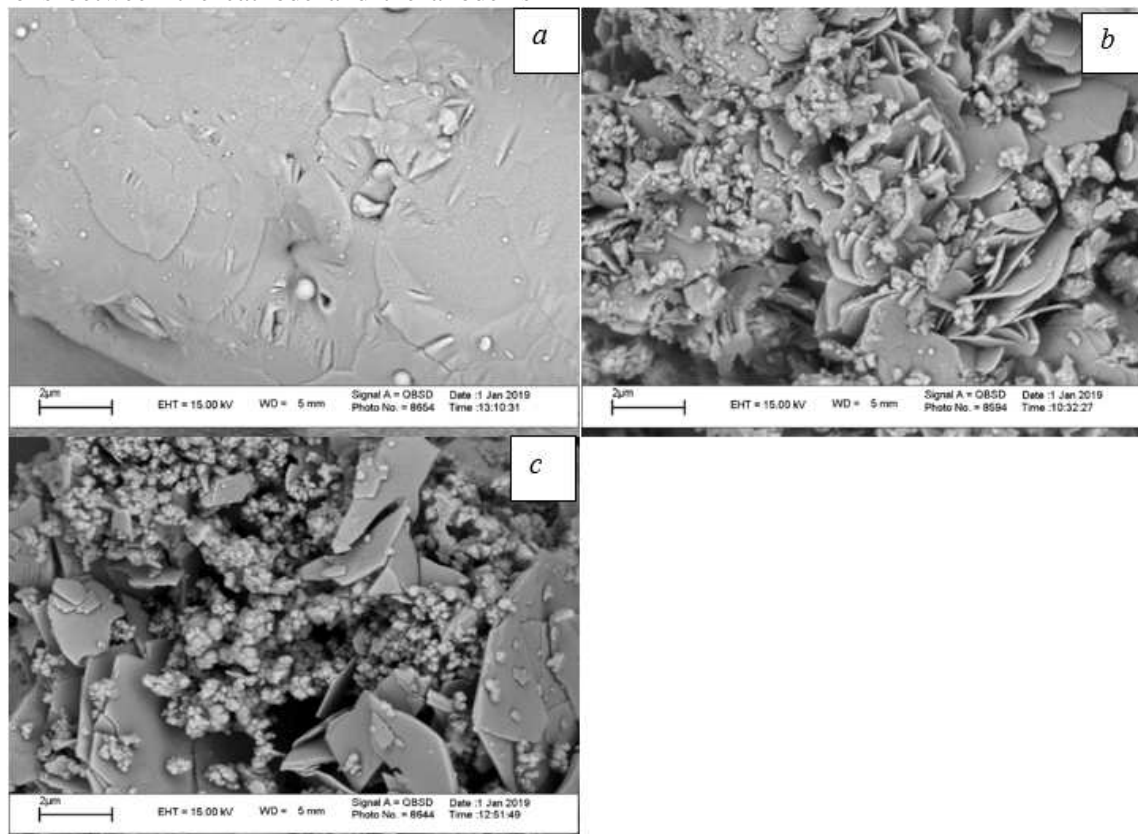

Fig. 9. SEM images of a) zinc dust surface; b) cadmium deposit morphology in the synthetic solution; c) cadmium deposit morphology in the leached solution

5.Conclusion

In this work, the kinetic of cadmium removal reaction by zinc dust in the leached solution of the Dandi melting plant and synthetic zinc sulfate solution with cadmium were studied. The reaction activation energy was calculated for both solutions using different theories such as collision, Arrhenius, and transition state. Finally, the cold purification process was modeled for the cadmium cementation in the reactor, and the governing differential equations were obtained to predict the behavior of cadmium removal at various times. Results showed that the synthetic solution's reaction constant increases with temperature increases, whereas the effect of temperature on the reaction constant for the leached solution is negligible. Activation energy data was fitted with Arrhenius's theory well, and the values for the activation energy for the synthetic and leached solution were obtained as

95.85 and 5.73, respectively. Also, results showed that the mathematical modeling of the system is in good agreement with the experimental data.

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