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A STUDY ON THE CADMIUM REMOVAL FROM AQUEOUS SOLUTIONS BY ZINC CEMENTATION

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ABSTRACT

The cementation of cadmium ions from aqueous solutions on zinc powder was studied as a function of solution pH and temperature, amount of zinc, concentration of cadmium ion, contact time, and the addition of several organic compounds, including surfactant and chelating agent. Cementation of cadmium was shown to be a feasible treatment process to achieve a high degree of cadmium removal within a fairly reasonable contact time. The deposition of cadmium and zinc consumption are highly dependent on solution pH conditions and is most practically operated at weak acidic conditions of pH 4–5. The reaction rate is approximately first order with respect to both the amount of zinc and the concentration of cadmium ion. Among the surfactants used in this study, only the presence of sodium dodecyl sulfonate, an anionic surfactant, noticeably enhanced the cementation rate of

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cadmium by zinc powder. The presence of ethylene-diaminetetraacetic acid (EDTA) in aqueous solutions inhibited the removal of cadmium by zinc due to the possible formation of Cd-EDTA chelates, which possess higher redox potential than that of free cadmium ions.

Key Words: Cadmium ions; Cementation; Surfactant; Zinc powder

INTRODUCTION

Growing concerns on the removal of cadmium from the aquatic environment has been a subject of increasing importance because of human health and environmental hazards. Currently, the most frequently practised treatment technology for the removal of heavy metals from aqueous solutions is chemical precipitation, which merely relocate the heavy metals from aqueous phase to solid phase, leaving sludge problems to be solved. Therefore, the removal of heavy metals from numerous industrial wastewaters has stimulated vigorous research activities in the development of a number of specialized treatment technologies with the possibility of metal recovery.

The cementation process has been demonstrated to be a feasible alternative because of its relative simplicity, ease of control, and the possible recovery of valuable metals. Several experimental studies have been conducted successfully for the removal of various heavy metals from wastewaters by cementation process (1-5). Theoretically, the removal of heavy metals from aqueous solutions by cementation process is capable of reducing these soluble metal species from aqueous solutions, almost completely into solid form, if the cementation reaction is taken long enough. However, the sufficiency of redox potential and excessive consumption of sacrificing metals limits the application of this process.

In this particular study, the removal of cadmium species from aqueous solution by cementation process using zinc powder was studied. The primary interest is to investigate the effect of operating factors such as solution pH, reaction time, and dosage of zinc on cadmium removal by cementation in the aqueous solutions with the presence of surfactant and chelating agent. The surfactants studied in this research include sodium dodecyl sulfonate (SDS, anionic type), *n*-cetyl-*n*, *n,n*-trimethyl-ammonium bromide (CTAB, cationic type), and octylphenol decaethylene glycol ether (Triton-X100, nonionic type) to explore the additional effect of surfactant with various electrical types on the cadmium cementation rate. The chelating agent used is ethylene-diaminetetraacetic acid (EDTA), which is released frequently from metal plating and



finishing industries. The molecular structures of these organic compounds used in this study are shown in Fig. 1. The temporal behavior of cementation and morphology of cemented deposits are examined, and the determined reaction rates and kinetic parameters obtained from experimental data are expressed and discussed in terms of deactivation kinetics.

THEORETICAL CONSIDERATION

Cementation process is a common practice for metallurgical industries and is a spontaneous redox reaction utilizing a less-expensive and noble sacrificing solid metal for the recovery of another more-expensive and less noble dissolved metal species, originally present in the aqueous solutions. For applications in wastewater treatment, cementation processes are usually employed for the deposition of a more-toxic metal reduced by a less-toxic metal. For instance, the removal of cadmium ions by zinc cementation studied in this research is achieved spontaneously because the redox potentials of zinc and cadmium are 0.76 and 0.315 V, respectively, and can be expressed by the following redox equation:



Like most redox reactions, the redox potential of a spontaneous cementation reaction is determined by the concentrations of reacting species, solution temperature, and the number of electrons transferred, and can be calculated by Nernst equation. However, the interference of some other redox

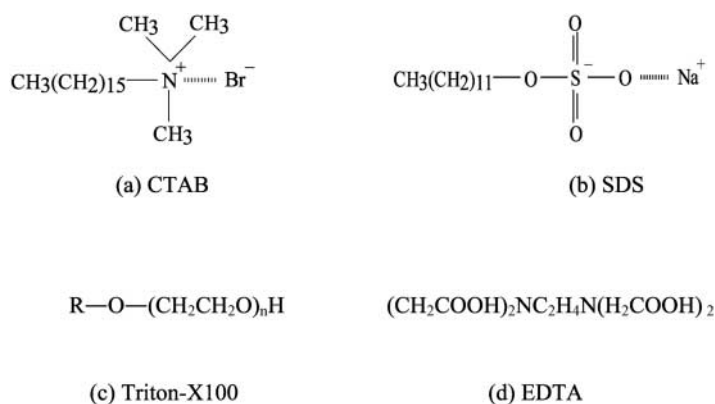


Figure 1. The molecular structures of the organic compounds used in this work.



and complexing reactions may affect the calculated redox potential for a pure cementation system.

Even though there are no foreign species originally present in the aqueous solution, several redox side reactions relevant to the presence of hydrogen ions and dissolved oxygen may still influence the cementation of cadmium by zinc. Figure 2 illustrates the calculated pe–pH relationship for cadmium in the aqueous solution based on the Nernst equation and depicted the cadmium species distribution under various solution conditions.

Free cadmium species possess a higher redox potential for low pH conditions indicating that the cementation of cadmium is more favorable in acidic solutions. Figure 2 also implies a higher dissolution of metallic cadmium in strongly acidic solutions due to the reduction by hydrogen ions. Therefore, the

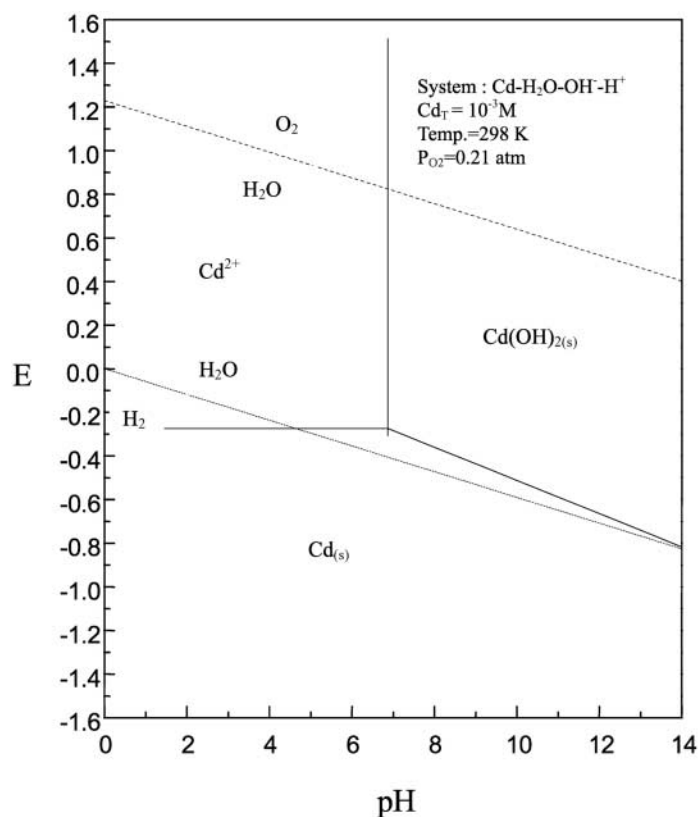


Figure 2. The calculated pe–pH relationship for cadmium in the aqueous solution based on the Nernst equation.



determination of proper solution conditions for practical application must compromise the considerations of various aspects. The calculated pe–pH relationship for zinc in the aqueous solution based on the Nernst equation is shown in Fig. 3. Similar to the species distribution of cadmium, free zinc species possess a higher redox potential for low pH conditions indicating that the reduction ability of zinc powder to cadmium cementation is more disadvantageous in acidic solutions. To avoid the possible effects caused by the presence of dissolved oxygen in aqueous solutions, the experimental work of this research was carried out under a nitrogen atmosphere, and the dissolved oxygen levels were kept under 0.5 mg/L. Thus, the only critical side redox reaction for cadmium cementation by zinc is assumed to be the reaction between zinc and hydrogen ions.

In addition to the redox reactions described above, complexation between the metal species with hydroxyl ions in aqueous solutions may lead to the

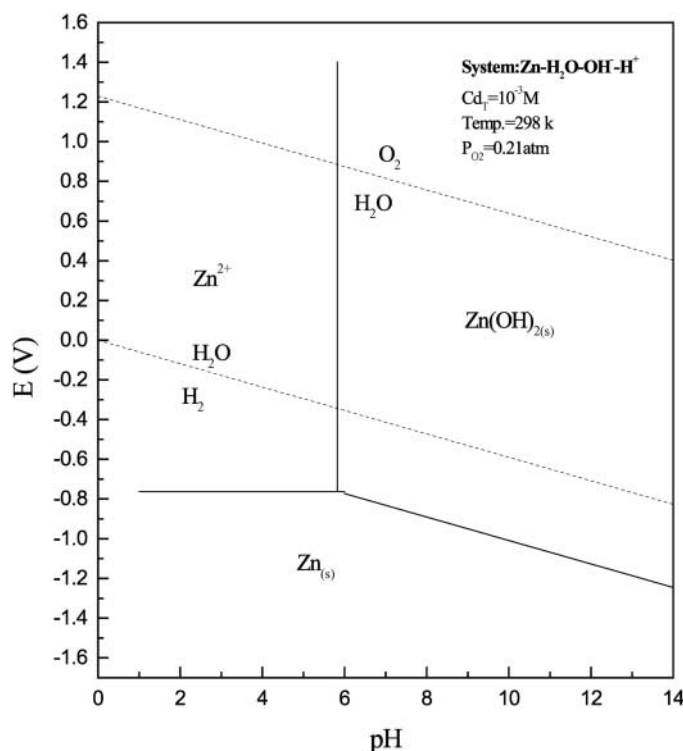


Figure 3. The calculated pe–pH relationship for zinc in the aqueous solution based on the Nernst equation.



formation of various metal–hydroxide complexes. Several research papers (6,7) reported that metal cementation process was impeded for applications in alkaline solutions because of the formation of several metal–hydroxide complexes and precipitates. To minimize the formation of metal–hydroxyl complexes occurring in alkaline solutions, most of the experimental works in this research was carried out in acidic conditions (pH less than 5.0).

The metal species distribution may be influenced by the presence of complexing or chelating agents in aqueous solution because of the formation of different stable metal complexes or chelates. The metal species distribution in aqueous solution can be calculated by establishing mass balances for various reacting species with appropriate equilibrium constants. For instance, the species distribution for cadmium in the aqueous solution in the presence of EDTA at various solution pH is shown in Fig. 4.

The formation of different complexed metal species is reported to influence the electronegativity of metal species and the cementation of metal ions in

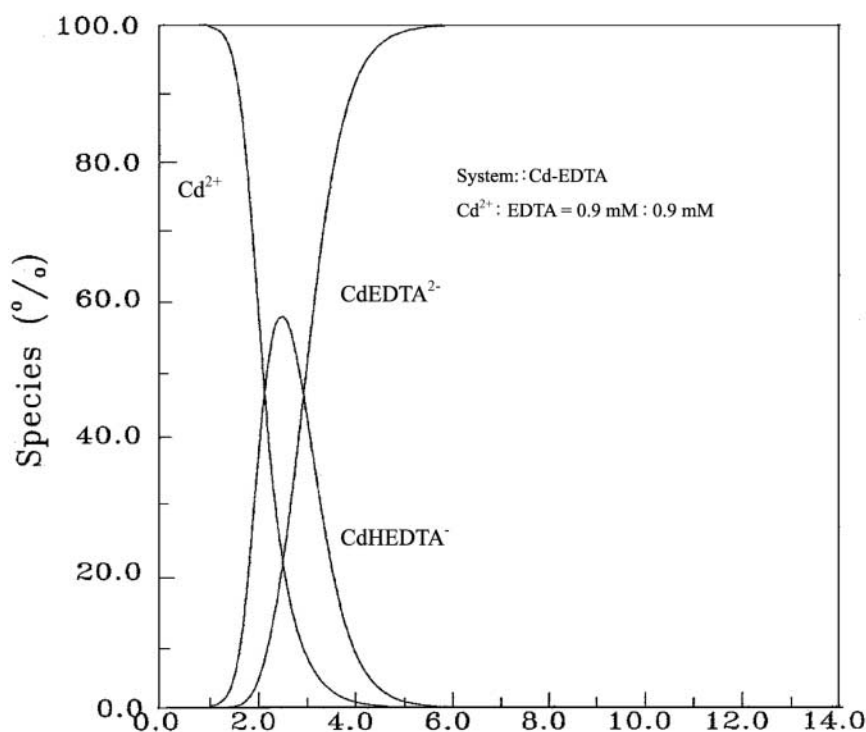


Figure 4. The species distribution for cadmium in the aqueous solution with the presence of EDTA at various solution pH values.

aqueous solutions (8,9) to certain degrees, depending on the solution conditions. The standard redox potential of cadmium–EDTA chelates is calculated to be -0.72 V based on the equation developed previously (10), comparatively higher than the recorded value of -0.3145 V for free cadmium ions by Antelman et al. (11).

Several research works have reported the effect of various organic surfactants on metal cementation. Karavasteva (12–15) investigated the cementation of copper, cadmium, and nickel with zinc particles in the presence of three organic surfactants (nonylphenolpolyethylene glycol, dinaphthylmethane-4,4,prime-disulfonic acid, polyethylene glycol with molecular weight 400) under various experimental conditions. Experimental results indicated that the effect of surfactant on the reaction rate and morphology of cemented metal deposits was highly dependent on the experimental conditions and some controversial findings were also reported about the effects among different systems. The research literature reported by Mahoud (16) on the effect of several surfactants on copper cementation by zinc indicated that the presence of surfactant reduced the mass transfer and cementation rates of copper ion. Owusu (17) found that the addition of an organic surfactant, LIX 622, enhanced the cementation rate of copper by zinc, probably because of the extraction of copper ions by this surfactant.

Cementation is essentially a solid–liquid reaction that the reduces metal deposits nucleated at cathodic sites on the surface of sacrificing metal, sequentially coalescing as cementation proceeds. Contact area between the bulk solution and the residual sacrificing metal is gradually diminished when the reduced metal deposits occupied the metal surface, and cementation stops finally when the contact area is totally engaged or the mass transfer of mobile species is exclusively ceased. The loss of sacrificing metal surface is similar to the deactivation of catalysts by coking mechanism for catalytic reactions. Assuming that cementation only occurs on the surface of sacrificing metal particle, which is spherical and of uniform size, the deactivation kinetics developed previously (6,18) are applied in this study to describe the behavior of cementation. On the basis of necessary assumptions, the deactivation rate equation of a reactant in an isolated deactivation system can be shown as below (6):

$$-dC/dt = kC^{\alpha}A^{\beta} \quad (2)$$

where C is the concentration of reactant (mM); k , reaction rate constant; A , active site on the surface of sacrificing metals (g); α and β , reaction orders.

If the sacrificing metal is overdosed, Eq. (2) can be simplified to be:

$$-dC/dt = k'C^{\alpha}, \quad k' = kA^{\beta} \quad (3)$$



Integrating Eq. (3):

$$C^{1-n} - C_0^{1-n} = (n-1)k't, \quad n \neq 1 \quad (4)$$

$$\ln(C/C_0) = -k't, \quad n = 1 \quad (5)$$

where k' is the pseudo-first order reaction rate constant (min^{-1}).

Using the same deactivation kinetics, the value of n was reported to be 1 by previous researchers (9,18) in the Fe/Cu^{2+} and Fe/Pb^{2+} cementation systems.

EXPERIMENTAL PROCEDURE

Zinc powder of greater than 99.99% purity with medium sizes of $45 \mu\text{m}$ was used as the sacrificing metal for cadmium removal in this research. The chemicals used in this study were certified reagent grade chemicals and all experimental solutions were prepared with double distilled water. Standard cadmium sulfate ($3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ with molecular weight 769.5 g mol^{-1}) solutions of 1000 mg/L (from Merck) were diluted to prepare required cadmium solutions with different concentrations. The effect of sulfate ions on the cadmium cementation rate can be neglected in this work (18). The reaction kinetic studies were performed in a 2-L reactor, made entirely of Pyrex glass. The vessel was then filled with cadmium solution of known concentration, purged with nitrogen gas continuously to keep the dissolved oxygen levels below 0.5 mg/L , and monitored by an Orion 820 dissolved oxygen meter, throughout the course of the reaction. Using an automatic pH controller, the solution pH was adjusted and maintained at the desired level during the reaction course by periodically adding 1 N NaOH and 1 N H_2SO_4 solutions. A mixing device with Teflon mixer (equipped with three impellers, the ratio of the impeller length to the diameter of reactor is 0.25, i.e., $5 \text{ cm}/20 \text{ cm}$) was used and the mixing speed was kept at 600 rpm after a series of mixing tests were performed to ensure sufficient mixing to exclude the effect of external mass transfer on the cementation process. Appropriate temperature control was maintained for the reaction by containing the entire apparatus setup in a constant temperature water bath.

At the beginning of the experiment, a predetermined amount of powdered zinc or iron was added to the batch reactor containing cadmium feed solution without/with surfactants or chelating agent. Typical experimental runs lasted $40 \text{ min} - 1 \text{ hr}$. At intermittent reaction time periods, a portion of the reactor solution was transferred and then filtered using a $0.45\text{-}\mu\text{m}$ filter. Total sample volumes were kept below 5% of the total reactor solution volume. The resulting filtrate was diluted (if required) and analyzed for its soluble metals content by the atomic absorption method with a Varian Spectra AA-20 spectrophotometer. If



necessary, the concentrations of surfactant and chelating agent were determined by an OIC 700 total organic carbon (TOC) analyzer. The residual zinc particles were rinsed with acetone, dried, and kept in a nitrogen environment prior to studying the surface morphology by scanning electron microscopy (SEM) using a Cambridge S360 and the composition of the cemented deposits by x-ray diffraction (XRD) using a Philips MP 710.

RESULTS AND DISCUSSION

The cementation of cadmium from aqueous solutions by zinc at various stirring speeds is displayed in Fig. 5. More than 90% of cadmium removals were accomplished by zinc powder for stirring speed greater than 600 rpm. The incomplete cadmium cementation by iron can be realized because the redox potential for Cd/Fe system is very low (standard redox potential is calculated to be 0.043 V). Therefore, only zinc powder was used as the potential metal and the stirring speed in the batch reactor was set at 600 rpm for studying cadmium cementation.

The experimental results of the pH effect on the cadmium cementation by zinc powder shown in Fig. 6 indicate that the residual cadmium concentrations can be lowered substantially by using a sufficient amount of zinc powder throughout the solution pH range studied. For experiments conducted for solution pH greater than 3, more than 99% of cadmium removal could be achieved within 60 min. The removal rate of cadmium by zinc powder from aqueous solution was increased with initial solution pH.

Experimental results summarized in Fig. 6 also revealed that the amounts of zinc consumption were stoichiometric with the amounts of cadmium cementation at weakly acidic conditions. After about 60 min of reaction time, the replacement of cadmium by zinc powder was almost completed. However, the residual concentrations of zinc were found to be markedly greater than the stoichiometric consumption by cadmium cementation afterward, especially for experiments conducted at lower solution pH levels as shown in Fig. 6. The excess consumption of zinc occurred due to the dissolution of zinc metal by hydrogen ions present in the solution. The concentrations of Zn^{2+} ions in the effluents were quite high (60–100 mg/L) and may be recovered with appropriate end treatments to avoid secondary pollutions.

The calculated global removal rates of cadmium based on the experimental results gave very satisfactory correlation, and were found to be approximately first order with the cadmium concentration. The pseudo-first order rate constants calculated based on experimental results for various operating conditions are summarized in Table 1.



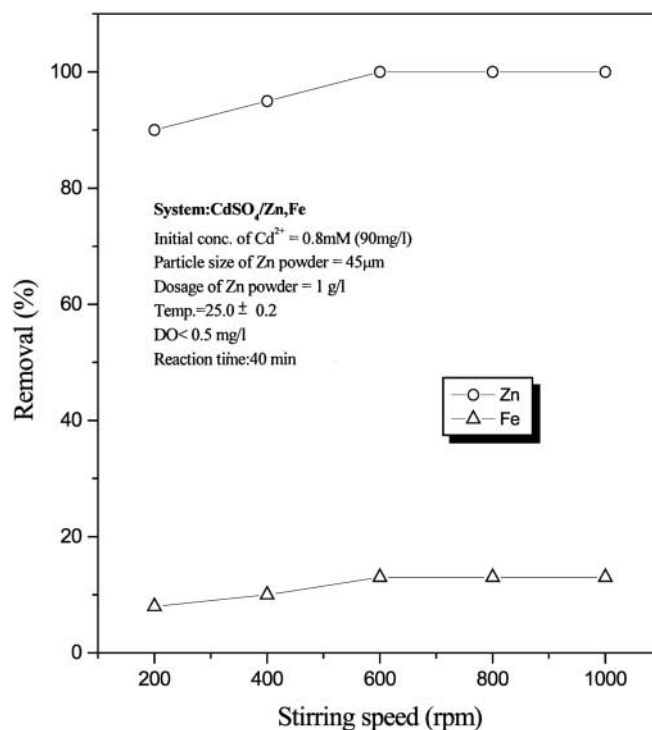


Figure 5. The effect of stirring speed on the removal of cadmium in the Zn/Cd²⁺, Fe/Cd²⁺ cementation systems.

For experiments conducted for initial cadmium concentrations between 25 and 200 mg/L, the maximum removal rate of cadmium ions by zinc was for experiments conducted at initial cadmium concentration of 50 mg/L as shown in Table 2.

Further increases of initial cadmium concentration gradually decreased the removal rate of cadmium. Comparable experimental results were reported by several researchers (3,5,19) for various cementation systems and were contributed to the morphology of cemented deposits. For experiments conducted at low initial metal concentrations, the driving force for cementation is weak but the metal deposits formed on the surface tends to be smooth and uniform, therefore, interferes with the further deposition of metal ions. Coarse and porous deposits are formed for experiments conducted at higher initial metal concentrations and may permit the transfer of metal ions through the deposits. But for experiments conducted at initial metal concentration higher than a certain



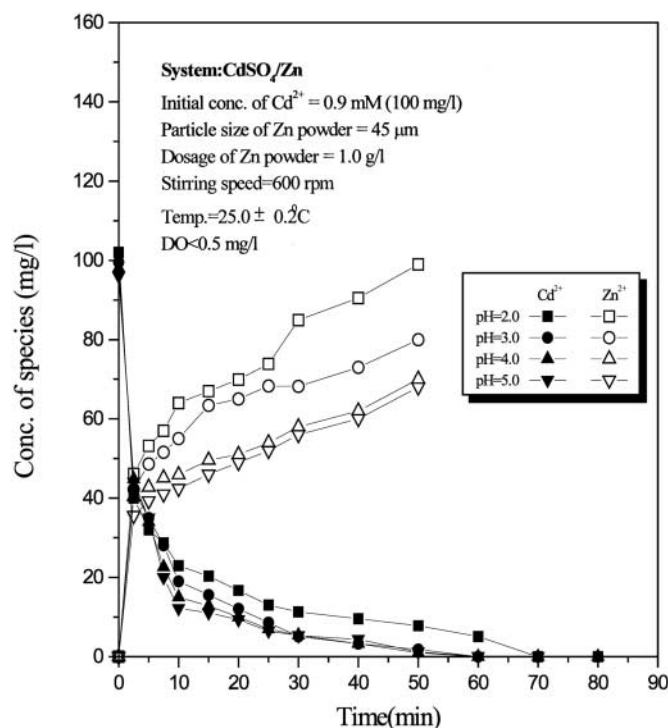


Figure 6. The effect of pH on the removal of cadmium in the Zn/Cd²⁺ cementation system.

value, the transfer of metal ions is gradually interfered by the thick, porous, and amorphous layer of metal deposits.

The experimental results for studying the effect of various surfactants on the cementation of cadmium by zinc was shown in Fig. 7. The presence of SDS, an anionic surfactant, enhanced the removal of cadmium ion from aqueous solutions by zinc cementation. However, the presence of cationic and nonionic surfactants, CTAB and Triton-X1000, respectively, retarded the cementation of cadmium ion.

Comparing Figs. 6 and 8, the consumption of zinc was markedly reduced due to the presence of surfactants when compared to the experimental results conducted at similar operating conditions without any surfactants. Mahoud (16) and Karavasteva (15) reported comparable results and contributed their findings to the formation of a protective film by surfactants on the surface of the sacrificing metal and prevent corrosion by hydrogen ions.



Table 1. The Pseudo-First Order Rate Constants of Cadmium Ions at Various Solution pH Values and Zinc Dosages by Zinc Cementation

Factors	Levels	k' (min ⁻¹)
pH	2.0	0.2768
	3.0	0.3040
	4.0	0.3344
	5.0	0.3568
Zinc dosage (g/L)	0.1	0.0960
	0.25	0.1504
	0.5	0.1800
	1.0	0.3344

At very dilute concentrations, surfactant molecules are dispersed on the aqueous solution interfaces. As the concentration increases, the surfactant molecules gradually gather because the hydrophobic groups on the molecules are attracted to each other. Micelles are formed when the concentration of surfactant exceeds the critical micelle concentration (CMC). For the experiments conducted in the presence of various amounts of SDS between 5 and 160 μM , the specific pseudo-first order rate constant (k' /total surface area of zinc powder) increased with the concentration of SDS up to 20 μM (about 5 mg/L) as shown in Fig. 9.

The presence of higher concentrations of SDS was found to decrease the cementation rate. Because the amount of SDS used in this research is much lower than the CMC (8.2 mM) (20), therefore the effect of SDS concentration

Table 2. The Pseudo-First Order Rate Constants of Cadmium Ions at Various Initial Cadmium Concentrations by Zinc Cementation

Factor	Levels	k' (min ⁻¹)
Initial cadmium concentration (mg/L)	25	0.2912
	50	0.3888
	100	0.3344
	200	0.3168



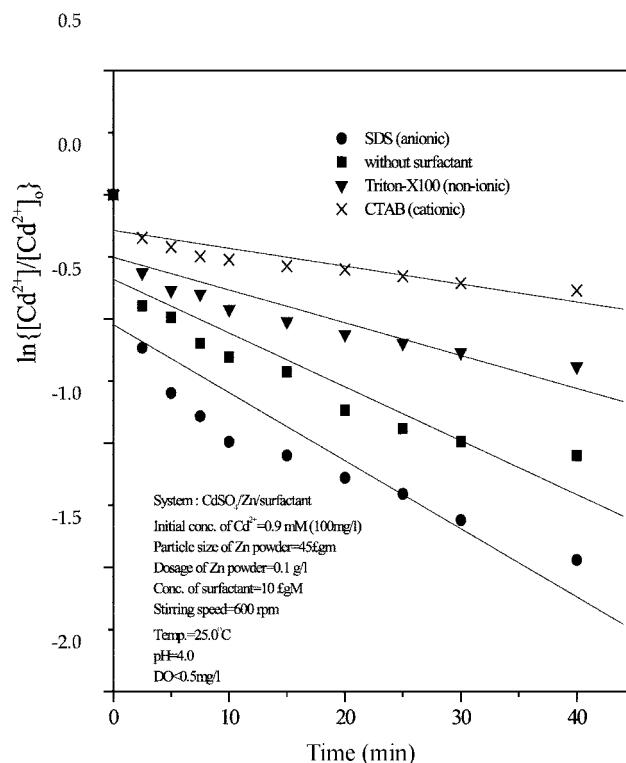


Figure 7. The effect of surfactant on the removal of cadmium in the Zn/Cd²⁺ cementation system.

is contributed to the adsorption behavior on the solid/liquid interface. The surface charge on the zinc surface may be neutralized in the presence of SDS and the active surface is increased because zinc powder is uniformly dispersed in aqueous solution without coalescence. The decrease of cementation rate for experiments conducted at higher concentrations of SDS may be due to the fact that excessive amount of active sites is occupied by SDS molecules.

The effect of solution temperature on the removal of cadmium ions by zinc cementation was studied at 25, 35, 45, and 55°C. Elevated solution temperature slightly increases the cementation reaction rate. An Arrhenius relationship based on pseudo-first order rate constants shown in Fig. 10, indicated that the apparent activation energy for the cementation system is about 4.8 and 2.6 kcal/(g mol) for experiments conducted without and with



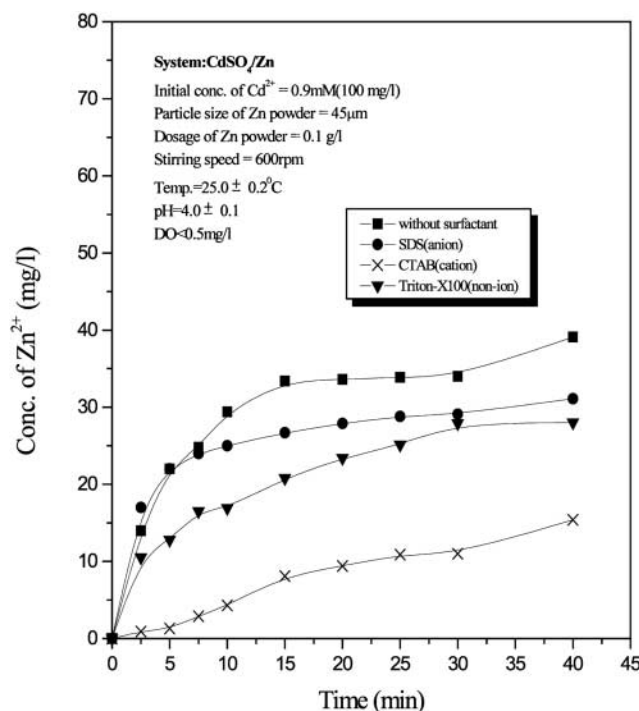


Figure 8. The effect of surfactant on the dissolution of Zn^{2+} in the Zn/Cd^{2+} cementation system.

$10 \mu\text{M}$ SDS surfactant, respectively. Similar experimental results were observed by other researchers (2,5,7,21) that activation energies for various cementation systems were ranging from 5 to 20 kcal/(g mol). The low activation energies imply that diffusion from solution to the surface of sacrificed metal is the rate-controlling step for cementation processes.

Experimental results indicated that the presence of EDTA significantly inhibited the cementation of cadmium by zinc as the depicted by the rate constants in Table 3.

On the basis of the discussion in the previous section, the formation of Cd-EDTA chelates may increase the redox potential and reduce the extent of cadmium cementation. Increased EDTA concentrations gradually increase the electronegativity of cadmium species and XRD analysis was performed to study the composition of cemented deposits under various experimental conditions.



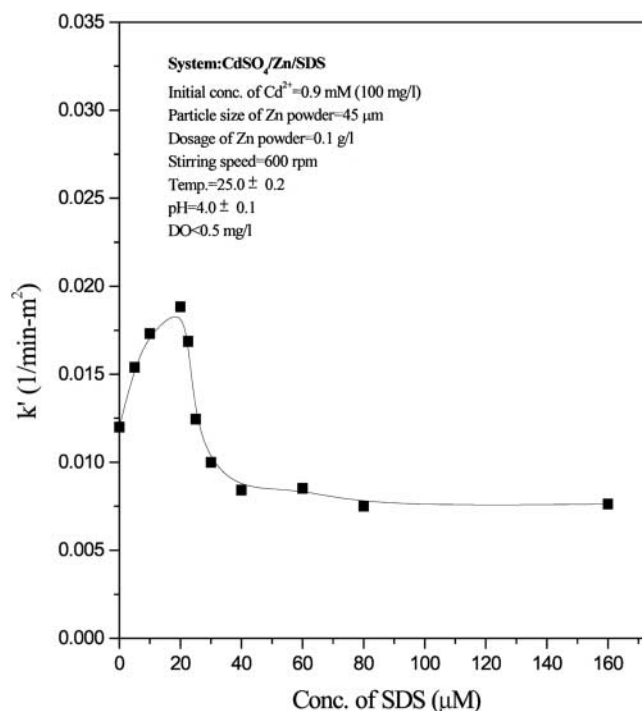


Figure 9. The effect of SDS dosage on the specific pseudo-first order rate constant of cadmium in the Zn/Cd²⁺ cementation.

The results indicate that cadmium deposits on zinc surface are almost in the form of cadmium molecules. No indications of any alloy or metal oxides/hydroxides were found on the surface. The distribution of the reflection intensity in the XRD analysis was found to be significantly different for deposits formed in the presence of different surfactants indicating the different morphology of deposits. Scanning electron microscopy studies of the cementation reaction products obtained with and without surfactants SDS, CTAB, Triton-X100 reveal the different type of deposits, as shown in Fig. 11. During the course of reaction, the iron surface acts as both cathode and anode. The nodular copper deposits indicate that copper cementation preferentially occurs at certain sites on the iron surface. Addition of the three surfactants to the solutions causes aggregation of cadmium deposits. These figures show that the lowest deposit porosity is obtained in the presence of CTAB (Fig. 11d), which is in



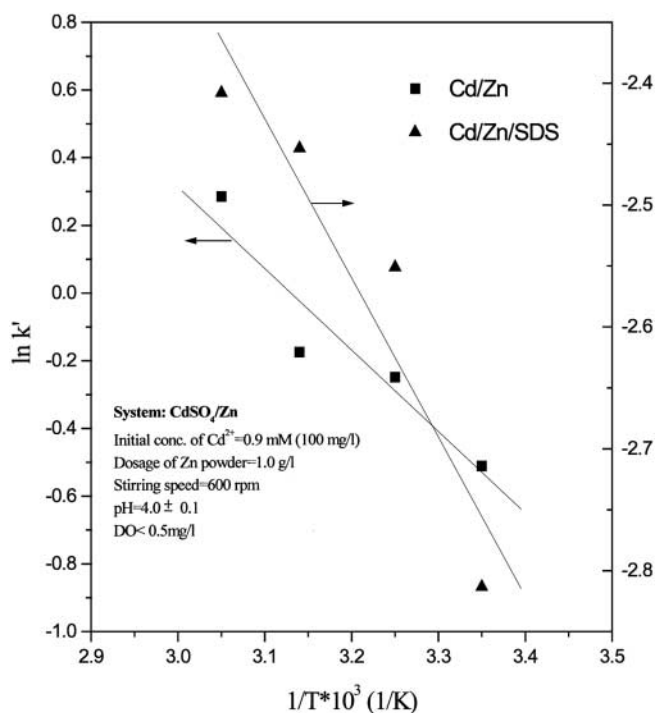


Figure 10. An Arrhenius relationship based on the first-order rate constants.

agreement with the effect of this surfactant on the cadmium cementation rate (Fig. 7). The dendrite formation obtained in the presence of Triton-X100 (Fig. 11e) probably increases hydrogen evolution which is in accordance with the obtained inhibition effect of this surfactant on cadmium cementation (14).

Table 3. The Effect of EDTA Concentration on the Pseudo-First Order Rate Constant of Cadmium in the Zn/Cd²⁺ Cementation System

EDTA (mM)	0	0.9	1.8	24.3
k' (min ⁻¹)	0.0592	0.0476	0.0432	0.0092



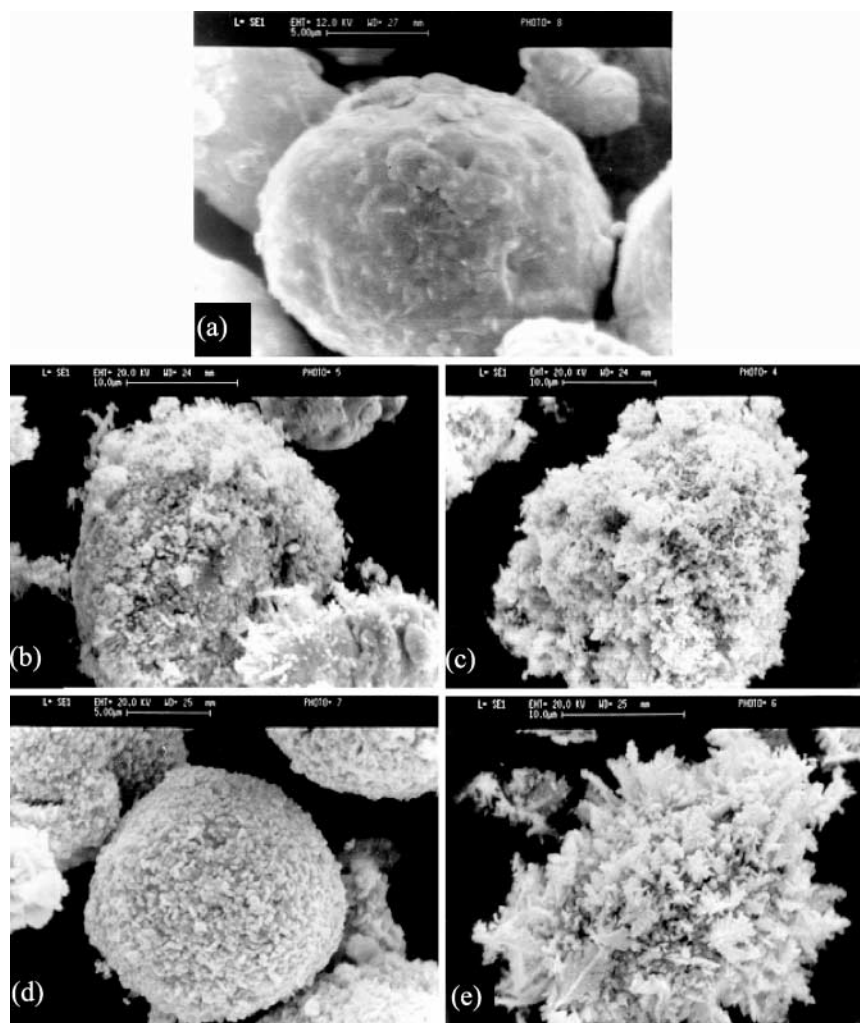


Figure 11. The SEM photograph of the cemented surfaces of zinc: (a) unreacted, (b) reacted without surfactant, (c) reacted with $10\ \mu\text{M}$ SDS, (d) reacted with $10\ \mu\text{M}$ CTAB, (e) reacted with $10\ \mu\text{M}$ Triton-X100.

CONCLUSIONS

Cementation was shown to be a feasible process to achieve a high degree of removal of cadmium for acidic solutions over a broad operational range. Considering the possible inhibition of the cementation rate at neutral and alkaline conditions, and the possible excess zinc consumption at strong acidic conditions, the cementation process is most practically operated at weak acidic conditions of pH 4–5. Apparent reaction kinetics based on a deactivation model give a very satisfactory correlation for the experimental results. The cadmium concentration was found to be of first order to the cementation rate of cadmium. The results obtained with and without surfactants SDS, CTAB, and Triton-X100 indicate their different effects on both the cadmium cementation kinetics, and cadmium deposit structure. The SDS surfactant improves but CTAB and Triton-X100 inhibit the deposition of cadmium. The lowest deposit porosity is observed in the presence of CTAB, which is to be expected from the large decrease in the cadmium cementation rate, when this surfactant is added. The cadmium cementation rate is inhibited in the presence of EDTA in aqueous solutions by zinc because of the possible formation of Cd–EDTA chelates with higher redox potential than that of free cadmium ions. The resulting effluent containing dissolved zinc may be recovered, and should not be discarded without proper treatment to avoid further pollution problems.

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